Rapra Polymer Testing Series

Handbook of Polymer Testing

Editor: Roger Brown

rapra TECHNOLOGY

The Rubber and Plastics Specialists

Handbook of Polymer Testing

Short-Term Mechanical Tests

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Contents

Preface

Knowledge of the properties of materials is essential for several purposes: design, specification, quality control, failure analysis and for understanding the structure and behaviour of new materials. Specific test procedures evolve for each class of materials. These procedures are generally those found best suited to the generic characteristics of the material class and their use helps to provide the most meaningful results and to allow comparison of data from different sources. Plastics are no exception.

When a Handbook of Plastics Test Methods was first published in 1971 for the Plastics Institute (now the Institute of Materials, Minerals and Mining) it was quickly accepted as the standard work on that subject and retained that position through two revisions, both written by Rapra staff.

This new work aims to follow in that tradition of presenting an up-to-date account of plastics testing procedures which is comprehensive in covering all tests in common, and not so common, use. Plastics testing is a very broad subject and to cover it all in one volume inevitably imposes restrictions of both scope and depth. This work is structured in a series of Parts, published separately, which is aimed to provide in total the widest possible scope with full coverage of each subject.

Since 1971 there have been continuous developments in testing methods and a virtual revolution in test instrumentation. Standard methods for virtually all properties have been adopted at national and international level. In recent times there has been considerable advance in aligning national standards with ISO methods and hence the latter have become increasingly important. The majority of standard methods are intended primarily for quality control but also form a basis for use in generating design data and for research. In this work, the standard methods and the requirements for obtaining data for specific purposes are described with discussion of the significance and limitations of test results.

The series is intended as a reference for those directly concerned with testing plastics in whatever capacity, from quality control to research, but will also be of value for students of materials technology and those indirectly involved in testing such as design engineers and technical specification writers.

As always, a book like this is the work of many people, not just the Author. I would like to thank my colleagues from Rapra Technology, Michael Hough for contributing to the chapters on tensile stress-strain and flexural stress-strain and Stephen Hawley for contributing the chapter on impact strength. I also extend my thanks to the Rapra Publishing Team, particularly, Claire Griffiths (Editorial Assistant), Steve Barnfield (Typesetter and Cover Design) and Frances Powers (Commissioning Editor) and to the freelance staff, Sara Hulse of Technical Text (Copyeditor), and John Holmes (Indexer) for all their hard work in getting the book ready for publication.

Roger Brown
September 2002

1 Introduction

1.1 Scope

The term 'short term mechanical tests' is used as a convenience to describe mechanical properties where the effects of long times and cycling are ignored. The term 'static stress strain tests' is used similarly, although quasi-static might be more appropriate. This group of tests includes hardness, tensile, compression, shear, flexing, impact and tear. For the purposes of this volume, it has also been convenient to include density and dimensional measurement, together with the essential matters of test piece preparation and conditioning.

As the title states, the coverage is plastics. Hence, rubbers are excluded but have been dealt with in detail in another volume [1]. Cellular materials and coated fabrics are also omitted as they are distinct classes of materials with specialised procedures. Fibre reinforced plastics, whilst undeniably plastics, can also be considered as a separate material class requiring their own test procedures for many mechanical properties. Here, reinforced materials have not been specifically excluded but, equally, have not been dealt with in depth.

The borderline between rubbers and flexible plastics is somewhat blurred, notably the thermoplastic elastomers. It has been suggested that this group of materials is best considered as rubbers [2] and that line has been followed in this volume. Nevertheless, some tests in the plastics portfolio are applied to these materials and indeed are necessary for the very high hardness elastomers.

The requirements for test apparatus are necessarily discussed but details of commercial sources together with advice on selection can be found in the *Polymer Test Equipment and Services Directory* [3].

1.2 Reasons for Testing

Ives was probably the first to explore a philosophy of the reasons for testing plastics in the first *Handbook of Plastics Test Methods* and the theme has been extended by Brown in later versions and elsewhere – and also copied by others. As he said 'Why test plastics?

For that matter why test anything? Why can we not rely on experience and good workmanship?'

In the case of an established material and application, there would ideally be no reason to continue testing were it not for the unfortunate fact that all men and machines are fallible and liable to vary in performance for a variety of reasons. Hence there is a need to test routinely to detect unacceptable deviations as a quality control measure. Plastics are certainly no exception in this matter and being complex materials require particularly careful control to ensure a consistent product. There is currently a growing trend towards greater demands for quality assurance and increasing consumer protection legislation which is probably resulting in more testing rather than less.

For a new material, a new application or a new product it is clearly prudent, if not absolutely essential, to prove the performance before unleashing the product on an unsuspecting customer. In fact the potential customer will put up very considerable sales resistance if you do not have this evidence and hence there is good reason for the very considerable amount of testing which is carried out to prove fitness for purpose.

The philosophy can be taken further in that at the design stage, physical property data is necessary to correlate with the calculated stresses, the expected environment and so on. Without such data one would be reduced to inspired guesses with its uncertainty of possible failure, or gross over-design with its accompanying wastage.

In the case of plastics these needs are particularly great because of the rapid change within the industry. The plastics in use today are very often not precisely the same as those available 10 years ago, even if the polymer is basically the same, and there are continuing refinements in processing. Also, plastics are being used in more and more new applications, and frequently more critical applications, than before. Thus, in many circumstances there is not much experience upon which to rely and this makes it very difficult to promote the use of plastics in, for instance, structural applications where a guaranteed 50-year performance may be wanted.

Even after all the design, proof of fitness for purpose and quality control, failures and disputes have been known to happen and there arises a fourth need for testing which is to carry out failure analysis to find the reason for the problem. Hopefully, such testing feeds back to aid improved design or quality control procedures.

The fundamental importance of the reason for testing is that to a considerable extent the choice of test method and test conditions depends on the purpose of the test. The requirements for quality control are not the same as for generating design data nor the same as for predicting service performance or for investigating failures. Not having clearly in mind what purpose the test is to serve can lead to an unfortunate choice of method or

conditions. Not appreciating the different needs can also lead to a biased view of the value of a given method – many tests are inadequate for design data but are considered valuable by a quality control person.

As a generalisation, particular attributes can be given to the purpose of testing:

- For quality control, the test should preferably be as simple, rapid and inexpensive as possible. Non-destructive methods and automation may be particularly attractive. The best tests will additionally relate to product performance.

- For predicting product performance the more relevant the test to service conditions the more satisfactory it is likely to be. Extreme speed and cheapness are less likely to be important but there is a need for test routines which are not excessively complex. Non-destructive methods may be acceptable.

- For producing design data, the need is for tests which give material property data in such a form that they can be applied with confidence to a variety of configurations. This implies very considerable understanding of the way material properties vary with geometry, time etc. Extreme speed and cheapness are of relatively minor importance, there is little interest in non-destructive methods. For complex and long running tests, automation may be desirable.

- For investigating failures the first difficulty is to establish what to look for and then the prime need is for a test which discriminates well. There is often little need for absolute accuracy or, in some cases, even relevance to service.

There is of course nothing black and white about attributing these requirements to the purposes of testing but they indicate the emphasis which usually applies in each case. Additionally, there are general requirements attributable to all test methods, such as adequate precision and reproducibility.

It is usual to classify tests by the property to be measured so that this volume covers short term mechanical tests and is subdivided into chapters on tensile stress strain, hardness, etc. A simple, more general classification which can be related to the reasons for testing is to think of Fundamental Properties or Tests, Apparent Properties or Tests and Functional Properties or Tests.

Using strength as an example, the fundamental strength of a material is that measured in such a way that the result can be reduced to a form independent of test conditions. The apparent strength of a material is that obtained by a method which has completely arbitrary conditions and the data cannot be simply related to other conditions. The functional strength is that measured under the mechanical conditions of service, probably on the complete product.

For quality control, fundamental properties are not needed, apparent properties will usually be acceptable although functional properties would certainly be desirable. For predicting service performance the most suitable properties would be functional ones. For design data, fundamental properties are really needed although considerable help can be gained from functional properties and often apparent properties are all that are available. For investigating failures the most useful test will depend on the individual circumstances but it is unlikely that fundamental methods would be necessary.

Using this classification it quickly becomes clear that most standard measures of mechanical properties yield apparent properties and there is a need for fundamental methods, whereas most dimensional methods and many thermal and chemical tests give fundamental properties.

It is interesting to note that the advances in test methods which are generally sought have remained constant over many years - quicker tests, cheaper tests, more reproducible tests, better design data and tests which are more relevant to service performance. Almost by definition this includes contradictions because a more fundamental test is not likely to be also quicker and cheaper. It also explains why there is never one direction in the development of test methods and apparatus. The perceived deficiencies in the existing methods are seen differently according to the particular purpose under consideration and hence development effort is targeted appropriately.

1.3 Source and Condition of Test Pieces

It is a fact that the results one gets depend on where the test piece came from and what has happened to it. This includes how it was moulded or otherwise produced, how old it is and where it has been. As a testing man I am very fond of saying this is not my problem - I can only test what I am given. Whether or not you accept that argument, it is necessary to be aware of the differences which can occur because of the production method, etc.

Ideally, all test pieces would be accompanied by details of their history but this is not often the case. If testing a particular (identified) batch of material or test pieces cut from a specific example of the product, the situation is fairly well defined, assuming nothing untoward has happened to the sample on its way to the laboratory. The problem is most apparent when obtaining material property data, particularly for comparative purposes or for use in a database, when properties can be enhanced or otherwise by careful selection of production conditions (or conditioning).

A somewhat similar uncertainty arises from the actual source of the material tested. This is generally a small quantity which has been taken from a much larger whole. Again the tester frequently does not know whether his sample is representative of the bulk material.

A test result on a plastic material cannot be taken at face value unless there is knowledge of how it was sampled and how the test moulding was produced. The fabrication details should, if known, be quoted with the result.

There are further complications when the actual test pieces used have been produced from the moulding by cutting, sawing, routing, etc. These operations may change the physical properties through excess heating or driving off of moisture which could result in unrepresentative test data. Many properties are influenced by the condition of the surface of the test piece. Notches and scratches act as stress raisers and lower mechanical properties, sometimes seriously. Quite obviously the surface condition is crucial when measuring optical properties. Methods for preparation of test pieces are considered in Chapter 2.

By no means all plastics articles have identical properties along their principal axes, i.e., there are some which are anisotropic. It is easy to envisage this in a fabric reinforced laminate because woven fabrics are usually themselves rather stronger in the warp direction than in the weft, unless a special weave is incorporated. However, materials that are homogeneous with respect to composition can also be anisotropic. An injection moulded bar with the gate at one end is likely to show pronounced orientation of the polymer molecules in the length direction unless special precautions are taken, and the tensile strength, for instance, will be higher along the bar than across it. It is therefore important to state the direction of testing when relevant or, preferably, to examine properties in two, or in some cases three, orthogonal directions.

The story of the problems or pitfalls of which the tester and the user of test results must be wary, continues with the conditioning of the test pieces and the atmosphere in which the testing is carried out. Most plastics are affected by quite small changes in temperature and it is essential that comparisons are only made between results obtained at substantially the same temperature. Many materials and properties are affected by moisture content and it is then necessary both to condition and test under known relative humidity conditions. Quite a short time is needed for a test piece to reach equilibrium temperature with its surroundings but very considerable periods may be needed to reach moisture equilibrium.

Even if carefully conditioned, the age of a material may be important. There are the fairly obvious hazards of degradation by light and the changes which even moderate temperatures will bring about. Some materials crystallise very slowly at room temperature and since polymer properties often differ significantly in the crystalline and amorphous forms, it is necessary to test at equilibrium or when the rate of crystallisation is so slow as to be without significance. Similarly, the process of plasticiser-polymer gelation may proceed slowly, even at room temperature, after the processing cycle, so that plasticised polyvinyl chloride (PVC) for instance, must be examined only after some specified minimum time. Storage, conditioning and test atmospheres are considered in Chapter 3.

1.3 Test Conditions

A parallel to the result depending on the history of the test piece is that for many tests the result will depend on the details of the test piece geometry and the test conditions. Although the tester may not have control over the form of test piece supplied or the test method specified, it is his or her problem to interpret the results considering the conditions used.

It is a basic problem with apparent tests that the result will vary with test piece geometry and test conditions and it may not be easy to extrapolate to different conditions. The prime purpose of standard test methods is for everyone to use exactly the same procedures and hence obtain equivalent results. The same applies to standardised conditioning procedures and test piece preparation.

There are plenty of good reasons for using different test conditions in particular cases – from producing data as a function of temperature to allowing tests on irregular shaped products – but there is no good reason to deviate from the standard procedure unnecessarily or inadvertently.

One suspects that the importance of not changing conditions is not always appreciated. It might be thought that, since most properties are reduced to units of lengths, area or volume to yield the basic data for the material, the precise size of test pieces cannot be of importance - it should all come out in the calculation. The statement of results as per unit thickness, for instance, implies that the property is proportional to thickness but this in fact is likely to be very misleading because the properties of a given material moulded in thin sections and those moulded in very thick sections may be quite different. In a thermosetting material the degree of cure is likely to be less in the thicker section, while with both thermosets and thermoplastics, the extent of locked-in strain is greater in the thicker test piece because of the slower cooling of the centre with respect to the skin. Clearly the measured properties may vary between thick and thin mouldings as a result of effects other than the ratio of the cross-sectional areas.

There are a host of other reasons why size or shape will influence the result and whilst it is not appropriate to try to enumerate them all, a few examples serve as a warning. It is not difficult to envisage that the two dumb-bell shapes shown in **Figure 1.1** would give different results because (b) has undesirable 'stress raisers' at the sharp 'shoulders' which would result in a lower breaking load. This may be an extreme shape but quite a variety of tensile test pieces are in use and they do not all yield identical results. Apart from shape, they vary in actual size and may be produced from different thickness of material.

Water absorption can be largely a surface phenomenon and if the result is to be reported as a percentage weight increase rigorous control of test piece size is necessary so that the surface area is standard and the percentages may be compared.

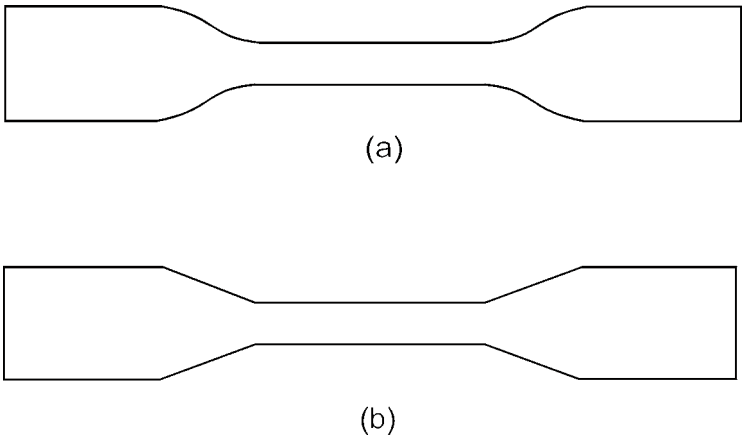

Figure 1.1 Possible shapes for tensile test pieces

The mechanical property of flexural strength is calculated from the force required to break a bar at its mid-point, the breadth and thickness of the bar and the distance between the outer supports (**Figure 1.2**):

$$Bending\ strength = \frac{3FS}{2bt}.$$

Why, then, worry about the precise values of S, *b* and *t* as long as they are measured accurately? In fact the classical bending formula above only holds in the case of ideal three-point bending for certain ratios of *S/t*. Thus, whatever the absolute merits of the property, for comparison of data the test pieces must be essentially identical.

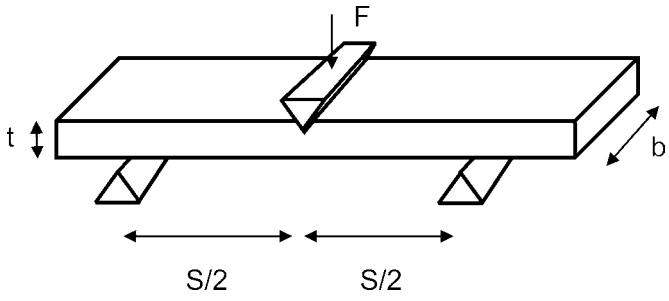

Figure 1.2 General arrangement for flexural strength measurement

Electrical strength is calculated from the breakdown voltage of a flat test piece and its thickness, often in volts per millimetre. This implies that the breakdown voltage is proportional to thickness but **Figure 1.3** shows this is not the case. It is necessary to know the slope of the voltage/thickness curve, i.e., obtain multi-point data, for the results to be relevant to products of different thickness. For comparison purposes the thickness must be carefully controlled and even then the comparison is only valid at that thickness unless the materials exhibit similarly shaped curves.

These are just basic examples of the influence of test piece shape and size on measured properties but they serve to illustrate the extreme care that is necessary to ensure that results are comparable.

Most mechanical tests involve straining the test piece and the apparent stress strain characteristics will be dependent on the speed at which the straining is carried out. In fact changing the speed is equivalent to changing the temperature and quite abrupt changes in behaviour can occur.

Figure 1.3 Electric strength as a function of thickness

1.4 Limitations of Results

The previous sections have made it very clear that a statement such as 'A material has a tensile strength of 10 units' is not an absolute fact. The result is limited by how the material was sampled, how it was fabricated, how the test pieces were formed, what conditioning it received, the dimensions of the test piece and the test conditions such as temperature and strain rate.

On top of this there can be different test methods for the same property, more than one procedure in a given standard method and more than one way of calculating and expressing the result – all conspiring to make comparison of results difficult and emphasising the necessity of quoting all the history and test conditions in the test report.

After all the precautions have been taken - the test pieces prepared correctly, the prescribed method followed to the letter, all the facts have been clearly reported without mixing the units - what is the value of the data obtained? Essentially the figures for strength, modulus, etc., derived from our measurements relate only to conditions which simulate precisely those under which the tests were performed, and strictly only to the particular sample tested. There are two aspects to the value, or the significance, of the results. First, there is significance in the statistical sense: the result is useless unless we know its significance in terms of the extent to which the sample is representative of the material and what reliance can be placed on the result, taking account of experimental error and material variation. The second aspect of significance is the relevance of the result in terms of material or product performance.

No responsible person even remotely connected with testing should be without a working knowledge of statistical principles; anyone ignorant of the basic ideas of this subject runs the risk of undertaking their work inefficiently and being unable to draw the correct conclusions from the results obtained. To understand what the results mean, what is their significance means statistics. The only way to avoid statistics is to bury your head in the sand and for many years this seemed to be a popular activity amongst technologists. For various reasons, including the influence of the quality movement and widespread availability of personal computers, statistical methods are now more widely appreciated and more frequently applied to plastics test results. There are many texts and standards available, including BS 903-2 [4] which specifically covers the related subject of rubber testing, such that it should be unnecessary to include coverage of basic statistical techniques in this work.

The basic fact is that all measurements are subject to variability and the sources of variability are numerous. In testing a sheet of plastic for tensile strength using five test pieces one is likely to get five different strength figures. This spread of results arises from the sheet not being completely homogeneous, differences in test piece preparation, variability due to the

test procedure, machine calibration or operator error. This sheet is only a sample taken from the total population of sheets which could be produced from one batch of material. Hence, testing more than one sheet introduces the variability due to the moulding process. Different batches of nominally the same material will show variability arising from differences in ingredients or the mixing process. Such factors as different operators, different test machines and different laboratories can all contribute different sources of variability. It is only by applying statistical techniques that one can judge the likelihood that different sets of results belong to the same population or are significantly different.

This introduces the important distinction between variability due to the material and variability, or the uncertainty, associated with the measurement. The tester seeks to reduce the variability of his or her results by carefully standardising testing and preparation procedures, calibration of apparatus and training of staff. This is the process of quality control in the laboratory discussed in Section 1.8.

It has already been pointed out, and will be further amplified throughout the book, that the property value obtained may vary according to the test method used. Many of our test methods use quite arbitrary conditions and procedures. The data obtained from this routine type of test, whilst admirable for quality control and perhaps as an indication of service performance if interpreted carefully, will rarely give the designer the values upon which to base his calculations. The more nearly a test approaches the real conditions of service the more relevant or significant it is likely to be in terms of predicting service performance. The more fundamental a result is in terms of it being independent of test piece shape or conditions the more relevant it will be for design purposes.

Frequently, pressures of economics and time prevent test procedures being very close to service conditions. The largest gap between most published property data and performance behaviour is in the time scale: just how long will a given component withstand a certain stress without failing? Trouble-free service over a period of years may be essential, yet tests have lasted but a few seconds or minutes. Accelerated tests can often provide very useful guides and frequently are the only solution to such demands. However, it must always be remembered that to produce this very acceleration, some test variable or variables have had to be intensified, for example the temperature raised, the nature of the environment changed or the frequency of stressing increased. These necessary changes may in themselves induce effects which would never occur at the usual ambient temperature, etc., and thus misleading data may result.

Considering the variation of materials, the uncertainty of the measurement process, the limitations due to the effect of test piece size and shape, the timescale of the test, the influence of accelerating effects and the nearness or otherwise of test conditions to those of service, it is quite clear that one cannot emphasise too forcefully that all measured properties should be most critically assessed to establish their true relevance and applicability.

1.6 Sampling

As discussed previously, the significance of test results depends to a considerable extent on how the physical sample was obtained. Whatever the purpose of testing it is necessary to question whether the samples tested adequately represent the population being investigated. Efficient sampling really comes down to selecting small quantities such that they are truly representative of the much larger whole.

When sampling from a large number of items for quality control purposes, it is usual to use statistical tables to decide the number to be taken which will provide any given level of probability of out-of-specification items being present. Information on such statistical sampling schemes will be found in texts on quality control. The selection of discrete products should preferably be randomised and certainly care must be taken that the sampling procedure is not biased, for example, by sampling at set times which might coincide with a shift change or other external influence. A book of random numbers (a set of tables designed to pick numbers at random without the risk of unconscious bias) is invaluable. In routine quality control there is the added dimension of needing to sample repetitively in time. This means that a good measure of the population mean and variance is obtained eventually but there is need for a long-term sampling plan and a continuous method for assessing the results.

In many cases the testing laboratory is limited by the amount of material available, there may be only one product or batch to be evaluated and its significance in terms of the population is simply not known. It is still necessary to ensure that the test pieces taken represent the sample properly, for example considering the direction of test pieces relative to the axes of the sheet and randomisation of their position in the sheet if the sheet cannot be guaranteed homogeneous and isotropic

The number of test pieces or repeat tests per unit item sampled must be decided. Our current standard methods are not consistent, ranging from one to ten or more and it is usually argued, although open to challenge, that the more variable a test the more repeats should be made. There is no doubt that financial considerations have played a large part in the deliberations, witnessed by certain very variable but time consuming methods calling for one test piece only. There is no doubt that to use one test piece only is rarely satisfactory but testing very large numbers will not yield a proportional increase in precision. There is a trend towards five as the preferred number and this has a lot to recommend it for the more reproducible tests, being just about large enough to make reasonable statistical assessments of variability. An odd number of tests is advantageous if the median is to be extracted. In a continuous quality control scheme the number of test pieces at each point is usually rather less important than the frequency of sampling. For example, it might be better to use one test piece but check five times more often.

1.7 Standards

Standards are the documents which define requirements for products and how they are to be tested. Hence, they are crucially important to a test laboratory. To avoid misunderstanding over terminology it is as well to note that the British Standards Institution (BSI) call all their documents standards and the word specification is reserved for those standards which specify minimum requirements for materials or products. Other types of standard are Methods of Test, Glossaries of Terms and Codes of Practice. It follows that test methods are the building blocks of specifications and a specification may refer to several methods of test. A recent account of standards and standards organisations for polymers is given by Ashworth [5].

Generally, the sources of standards can be placed into three groups:

- International organisations
- National organisations
- Individual companies

The ultimate state of unity would be for all countries to be using the same standards. This would obviously be of great value in smoothing the course of international trade and make it easier for technologists to exchange technical information. It is also a very ambitious concept that the countries of the world can compromise on their national procedures and overcome the very great difficulties of language in a field where language is the most important tool of trade.

In most fields, including plastics, the principal body attempting to achieve the ideal of international agreement is the International Standards Organisation (ISO) which is hence our most important organisation in the standards field. In the electrical field the International Electrotechnical Commission (IEC) performs the same function as ISO.

European countries have particular interest in the more limited scope of European standards. The European Committee for Standardisation (CEN) comprises the national standards bodies of EC and European Free Trade Association (EFTA) countries. CENELEC is the equivalent body in the electrical field. To many people the concept of European standards, or any other national grouping, is an unnecessary complication, it being argued that there is no need for any activity in between ISO and the national bodies. However, the work of CEN assumes great commercial importance because EC countries are bound to adopt them to replace national standards and they can be used to demonstrate compliance with EC Directives.

Although generally each country has one principal standards organisation which provides the official membership of ISO, other organisations can issue standards at national level. It is usual to include government departments in this category. The UK national body is the BSI.

In this English language book, ISO, European, British and ASTM (American Society for Testing and Materials) standards are considered. British standards are now almost always identical to ISO standards. Where European (CEN) standards for plastics exist they have to be accepted by members of the Community. As these CEN standards for test methods are generally identical with the ISO methods, the situation is rapidly being reached where ISO, CEN and British standards are effectively the same thing and are dual or triple numbered, e.g., BS EN ISO xxxx, BS EN xxxx or BS ISO xxxx. Note that the ISO standard may be a year or two older than the dual/triple numbered standard.

There must be literally millions of company standards in existence. Although they have relatively little significance in a national or international sense, they are the basis of many commercial contracts. It would save a great deal of pain and confusion if those writing commercial specifications would wherever possible use published standard test methods, preferably those of ISO. Special tests will often be needed but there is no point in inventing your own procedure for a straightforward test which has been well standardised. Perhaps a lot of the trouble is that in some cases those writing specifications are not well versed in standardisation outside of their own organisation and that many engineers have a poor understanding of plastics and their properties.

Different styles or types of published standard test methods can be recognised. In the simplest case a particular apparatus is specified, one set of mandatory test conditions given and no choice allowed as to the parameters to be reported; this is the form in which the specification writer needs a test method. Many national and international test methods have become rather more complex. This is partially a result of compromise but more importantly because the measurements being described are not intrinsically simple and the method will be required for a number of different purposes and probably for many different end products. The specification user must therefore select the particular conditions which best suit his individual purposes.

We can conveniently distinguish three different circumstances in which a standard method is used: (a) purely for quality control, (b) as a performance requirement, and (c) for development purposes. In the first case the prime consideration is that precisely the same procedure is always used and also that this procedure is relatively simple and rapid. The test conditions may be completely arbitrary but one set of conditions and one set only is required. If the test is intended, apart from a quality control function, to be a measure of the performance of the product then test conditions will be chosen which have some relevance to the product end use. For development work it is highly probable that a series of conditions will be needed in the hope that data of use in designing future products will be realised.

Recently, there has been a proposal that international standard test methods should be restructured to have a number of parts so that the requirements for different purposes are more clearly separated but it is too early to guess if this will happen for plastics methods.

Single, unequivocal procedures are also needed for the input into databases and for comparison of material properties. Fairly recently it was recognised that the number of variations included in many test method standards was not helping in this respect and international standards have been developed to specify the methods and conditions to be used for producing single and multi-point data for plastics [6-10]. There is also a new work item to develop a guide for the acquisition and presentation of design data. It should be noted that references to standards given in this book were correct at the time of writing but standrds are subject to an ongoing revision process and the relevant standards catalogue should be consulted to find the latest edition.

1.8 Quality Control of Testing

In the same way that factory production is expected to be subjected to a quality assurance system so the test laboratory needs its own quality procedures. To keep apparatus, procedures and people in the best condition to produce reliable results requires systems and control. Almost certainly the best way of achieving this in a testing laboratory is to be subjected to the disciplines of a recognised accreditation scheme. The ISO 9000 [11] standards are now commonly applied in companies and the laboratory will be included in that system. However, more rigorous and focused schemes for test and calibration laboratories have been standardised in ISO 45001 [12] which requires procedures for everything from the training of staff and the control of test pieces to, most importantly, calibration of equipment. To maintain the requirements, which are given in deceptively short form in the standards, is both time consuming and difficult but anything less than these standards is not ensuring the highest possible quality in the results – which are the output of the laboratory. In the UK accreditation of laboratories is entrusted to The United Kingdom Accreditation Service (UKAS) with a number of other countries having equivalent bodies. Some of these bodies have mutual recognition agreements.

Whilst all aspects of a laboratory's operation require systematic control, it is the calibration of test equipment which gives rise to most problems and which is also the most expensive. All test equipment and every parameter of each instrument requires formal calibration. For example, it is not good enough to calibrate the force scale of a tensile machine, there are also requirements for speed of traverse, alignment, etc., plus gauges for test piece dimensions.

Calibration is based on the principle of traceability from a primary standard through intermediate standards to the test equipment, with estimates of the uncertainty (which increases at each step in the chain). Wherever possible, bought in calibrations should be carried out by a UKAS (or equivalent in other countries) accredited laboratory. In some cases it is perfectly acceptable for the test laboratory to do its own calibration but then they must maintain appropriate calibration standards and operate in accordance with ISO

10012 Parts 1 and 2 [13, 14]. Definitive guidance on calibration of rubber and plastics test equipment is now available in BS 7825 [15-17]. Part 1 specifies the principles and general requirements, Part 2 gives outlines of calibration procedures for each parameter and Part 3 is in several sections giving detailed schedules for each international standard test method.

Calibration laboratories are required to make uncertainty estimates for all their measurements. Estimates of the uncertainty of test measurements can be made in the same way by considering the uncertainties introduced by each factor involved in the measurement. Producing estimates is not particularly easy and the results tend to be frightening. However, it is to be expected that estimates will be increasingly required of accredited testing laboratories.

Another area which has tended to be overlooked is the validity of manipulations made on the test data. It is probably reasonable to trust a calculator to perform a simple arithmetic operation - although that may not always be the case with the operator. However, increasingly data is being manipulated by a computer to automatically produce the test result, involving quite sophisticated operations. This includes such things as area compensation, modulus calculation and curve fitting. If you carry out these tasks by hand any abnormalities are likely to be apparent but a computer will happily carry on regardless. As they say, rubbish in, rubbish out. It is essential to verify any software used to ascertain that it will produce valid results under all circumstances. A particularly obvious example is to account for offset zero points but others can be quite subtle. A computer will apply a strict formula to deriving figures from a stress-strain curve whereas a human will make judgements based on knowledge and experience. Some standards bodies are now developing specifications to give rules and guidance on software verification.

The object of quality control procedures in the laboratory is to produce correct and reproducible results. Up until the 1980s, although good reproducibility was desired and it was known that some tests were better than others, it was assumed that for most properties the level of agreement between laboratories was reasonable. There was not a wealth of published data to support or contradict this complacent state but the scattered accounts which could be found almost always revealed large discrepancies. One must surmise that that these did not raise great concern because of a general attitude that when there was disagreement the other chap had done something wrong!

When ASTM, followed by ISO and others, started conducting systematic interlaboratory trials to obtain precision data for test methods the true state of affairs became apparent [18]. For many standards the variability was worse than realised and in some cases was so bad as to question whether the tests were worth doing at all or whether specifications based on them could be considered valid. The general advance of the quality movement prompted these investigations and reproducibility has continued to occupy one of the top spots for attention.

The interlaboratory trials result in precision statements in the test method standards. These give measures of the within and between laboratory variability which were obtained under specific conditions. Although it is true that a different set of figures might have been obtained from another trial with a different group of laboratories, they are representative of the variability which can be expected. Interlaboratory comparisons organised by standards committees are conducted with what are considered to be good quality laboratories so that they might be expected to represent an optimistic situation. However, there is some unpublished evidence that a comparison within a closer group, for example all UKAS accredited, produces better results.

There are a number of reasons for excessive scatter of results found between laboratories - wrong calibration, incorrect apparatus, misinterpretation of the standard, deviation from the procedure, operator mistakes, etc. They reduce in the end to either the standard being too lax in its specification and tolerances or somebody is doing something wrong. An interlaboratory comparison tells you the magnitude of the scatter but not which of the possible causes is responsible. That requires further and probably very expensive investigation. There have been various initiatives to investigate the causes of variability and make improvements but financial restrictions have kept the scale of these modest in relation to the size of the problem.

The most powerful tool to minimise the component of variance due to error in the laboratory is the discipline which recognised accreditation schemes bring. They encompass all the likely areas which produce mistakes, documented procedures, training, checking procedures, control of samples, monitoring conditions, formal audits and perhaps above all calibration. The general quality movement has produced pressures to make laboratory accreditation commonplace and as more laboratories reach this status it must be expected that reproducibility will improve. At present it is more prevalent in some countries than others, but international agreements will encourage universal adoption as well as ensuring uniform levels of the accreditation criteria.

1.9 Test Equipment

To be adequate for its purpose test equipment always needs to comply exactly with the standard test method being used, be in good working order and be properly calibrated. However, within these conditions there is scope for a considerable range of sophistication, ease of use, etc.

The greatest change in test laboratories in recent times has been the improvements made to apparatus by the introduction of automation and, in particular, the application of computers to control tests and handle the data produced. These advances in

instrumentation and data handling are primarily noticed as improvements in efficiency or accuracy rather than intrinsically improving the relevance of tests to product performance. However, they can and do influence the test techniques which are used, for example by allowing a difficult procedure to become routine and hence increase its field of application.

Wherever appropriate, comment is made on the form of apparatus now available for any particular test but it is not practical to include a chapter specifically dealing with hardware and software in detail. As mentioned in Section 1.1, apparatus commercially available and guidance on selection is given in a published guide which is also available on CD [3]. It is worthwhile to bear in mind the ways in which instrumentation advances have been advantageous, and also their less desirable aspects.

Automation in particular is first thought of as saving time and hence money. If the test can be left to measure itself and an operator's time is saved, there is a particularly attractive cost benefit. However, automation is also frequently very important in improving accuracy, reproducibility or making a procedure possible. Many aspects of the automation of mechanical testing are covered in an ASTM publication [19].

Some processes are taken for granted, for example no one is on record as having sat up all night adjusting the controls of an ageing oven and to manually maintain a temperature ramp on a Vicat test, although attempted, is the next thing to impossible. Thermal analysis techniques such as differential scanning calorimetry (DSC) only became feasible with developments in instrumentation, tailored dynamic loading cycles needed the introduction of servo-hydraulic machines and many other examples could be cited where the test is not possible without the instrumentation.

Automation frequently aids accuracy and/or reproducibility by being more consistent than humans. Non-contact extensometers ensure no unwanted stresses on the test piece and any automatic extensometer will be less subjective than a technician with a ruler. Digital thermometers, load cell balances and many other apparatus introductions have made measurements easier and less prone to operator error.

Time and cost saving has been most notable in the logging and processing of results where computerisation has amounted to nothing less than a revolution. Around 1970 it was estimated that my own laboratory could spend half its time processing results. That time is probably now only a few percent. It is also significant how views have changed. Then it was widely held that direct links between test machine and computer were only justified in a few cases. Now any major equipment is likely to be operated via the keyboard.

The automation of sample handling has not taken off as some predicted in the sixties when the first automatic systems were developed for tensile machines and hardness and

density apparatus. Robots are rare alongside the test rig and the reason is doubtless to do with volume, as such automation only becomes worthwhile when a very large number of identical tests have to be made.

Advances in instrumentation have not been without their disadvantages. On a pure time saving basis tests would now be remarkably cheap but the cost advantage has been counteracted by the fact that more sophisticated apparatus costs more money and is likely to be outmoded more quickly, leading to much higher capital costs. Although development should make equipment more reliable it can be generalised that more complicated and advanced equipment requires more maintenance by highly skilled and highly paid people. The cost side of the equation has also been added to by rising standards of calibration and laboratory quality control generally.

The calibration of more sophisticated apparatus has also been fated with additional problems arising from the difficulty of directly reaching the actual measured values. The software which so efficiently transforms the data can give rise to concern as to what has happened between the transducer and the final output. As mentioned earlier, the software itself requires verification, which is often not an easy task.

1.10 Product Testing

The test methods discussed in this book almost all relate to tests using test pieces which are formed from the material, cut from sheet or cut from suitably shaped products. Similarly, most product, and almost all quality control, specification requirements relate to tests on the materials. Testing the whole product is generally more difficult and more expensive but, particularly for demonstrating fitness for purpose, there are occasions when it is highly desirable.

There are in fact three possibilities as, in addition to testing the materials or the product, in many cases tests can be made on test pieces cut from the product. This has the advantage that the material properties measured are those that relate to the material as processed in the factory rather than to those on test pieces prepared under laboratory conditions. The only disadvantage is the limitations to obtaining suitable test pieces from many products.

If our knowledge of the properties and behaviour of polymers, and hence our design rules, were such that we could predict the performance of the product accurately from tests on laboratory test pieces then product testing would be rarely needed. Tests which yield fundamental data in a form that is independent of test conditions and test piece geometry are unfortunately rather rare. Hence, the fact that our understanding is by no means perfect means that there will often be need to test the whole product since this is the only way to be sure that it will perform satisfactorily.

It follows that the main reason for testing the product is to establish fitness for purpose, i.e., performance testing. Very often testing will be to prove compliance with a performance specification.

In the case of a new design it can be more expedient to subject prototypes to real service rather than to develop simulation tests. No simulation test will reproduce service perfectly and proving in the field will probably give the greatest confidence. However, there are many cases when this is simply not sensible for time, cost or safety reasons. Nevertheless, it is worth noting that even when accelerated tests are used the exposure of prototypes to natural service does mean that you will know of any unforeseen failures before they happen to products sold.

Product tests can also be used for quality control but very often this is restricted because of the value of the product. Prototypes can also be tested to provide input to an ongoing design programme. Failure analysis sometimes makes use of product tests to demonstrate compliance or not with a product performance requirement.

It is generally very clear when it is desirable to test the whole product. What is usually much more difficult is to weigh up the risks and the information gained against the costs of testing. Where test pieces are being used for quality control or performance of the product it is always preferable to use pieces cut from the product.

It can be extremely difficult and/or expensive to devise tests to simulate performance adequately and justification for the investment will be in proportion to the importance of the product in risk and/or sales terms. Service conditions are almost inevitably complex and include mechanical and environmental stresses over an extended time period. There is clearly much skill involved in designing rigs and test schedules which give maximum information at minimum cost. In practice there is a danger of spending very large amounts and still not getting the simulation accurate enough but, most commonly, the pressure is to under-design the apparatus and curtail the programme to cut costs.

The same principle applies to quality control testing, but here there is a much greater probability that the experience gained from proving the product initially will allow the quality of subsequent production to be reliably judged on the basis of tests on test pieces. Further, it may be adequate to control limited aspects of the service conditions. Sometimes a product test will give more valuable assessment of quality for the same testing cost as needed for test pieces. This would be true, for example, for impact resistance of a bucket (See **Figure 1.4**) because the cost of moulding test pieces would be little different from the value of the bucket and the testing costs would be equal. Impact testing the bucket would actually be cheaper than machining standard impact test pieces from it.

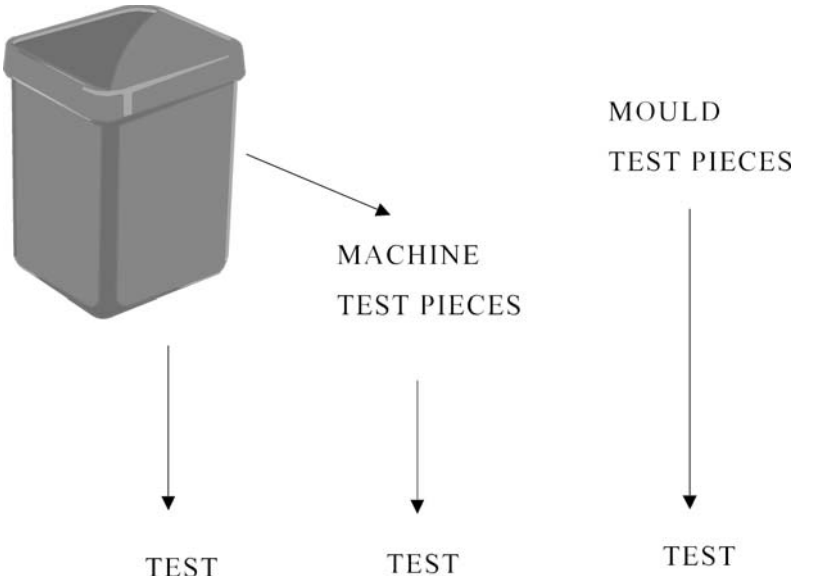

Figure 1.4 Choices for impact testing of a bucket

When the value of the product far outweighs the cost of making test pieces it is again a matter of judging whether control on test pieces gives us sufficient confidence to reject the costly alternative of product tests. It is here that non-destructive tests on the product become especially attractive and, not surprisingly, great effort is made to devise such tests, which give more confidence than the use of test pieces and additionally may even be cheaper to carry out.

There are legions of product tests, about as many as there are products, and the trend is for more product specifications to include performance tests on the product. Almost by definition, product tests are devised to suit a particular part and application. A fair number have been standardised but even then many show their *ad hoc* origins. The range of sophistication goes from the extremely simple to the very complicated.

Generally, one cannot simulate full service conditions in one test and it is necessary to restrict the scope to one bit at a time, or things get excessively complicated. For example, loading that varies with time of use plus abrasion from external sources and environmental ageing in general

The usual starting point is to look at how the object is stressed in service and simulate it - drop a loaded container, put force on a handle, pressurise a pipe, etc. The more

complicated the service stresses the more complicated the simulation will become - a handle may be bent and twisted, which has to be done together, whereas a container could fall on its side or a corner which can be done sequentially.

More often than not this action is repeated in service many times and the test is made to do likewise, i.e., we move to having a fatigue test. The frequency of repeated action can usually be higher than in service, and hence a degree of acceleration is achieved, but care must be taken that the frequency is not such as to cause behaviour which would not occur in practice. It should be remembered that a fatigue test will only compound any inadequacy in the choice of stresses imposed and their amplitude.

In real life the stressing, perhaps repeated, takes place in what might be termed an aggressive atmosphere. This is taken to mean heat, cold, UV light, chemicals, abradants, etc. or some combination.

This is where the test design can get especially complicated. Firstly, it is difficult to define the atmosphere which will vary from day to day or place to place. Secondly we are now into ageing tests and to test in a reasonable time we will want to accelerate the effect. The problem of validating any acceleration process and the extrapolation in time to predict real life performance is notorious and applies to products as well as test pieces. The costs to add environmental effects will escalate and the uncertainty of truly matching service rises steeply.

To help lessen the complications of product testing, a fairly common approach is to test mechanical properties on a relatively simple product test rig but to make degradation studies on the material or materials. It can then be argued that a given degree of deterioration in the mechanical properties will result in a proportional reduction in the product performance. Alternatively, the product can be subjected to an ageing process and subsequently tested for mechanical performance.

The limited range of application of any product test rig often means that very few are built. This, together with the costs and technical difficulties, results in many cases in the validity and reproducibility of the test being inadequately investigated. If tests are included in national or international performance specifications before proper evaluation, problems of interpretation and differences in results are likely to arise.

1.11 Modes of Stressing

Mechanical tests are carried out using a variety of modes of deformation of which tension, compression, flexure and simple shear are the most common. Others include torsion, biaxial tension, bulk (hydrostatic) compression and hybrid configurations as in indentation

hardness or the drape of film. The mode of deformation should in principle be chosen as that most relevant to the intended application but the choice may also be influenced by experimental convenience and the form of test piece available. For example, tensile properties are much more commonly measured than shear because they are relatively easier to perform.

Although in principle stiffness in the different modes of deformation are related, for practical materials and strains the relationships are generally complex. If we assume linear elastic behaviour and small strains moduli can be related via Poisson's ratio:

$$G = \frac{E}{2(1+\nu)} \qquad K = \frac{E}{3(1-2\nu)}$$

where:

G = shear modulus
E = Young's modulus
K = bulk modulus
ν = Poisson's ratio

Measurements of Young's or shear modulus are usually taken at small strains where the stress strain curve is approximately linear.

Moduli measured in different ways by often give different values. For example, Young's modulus from tensile and flexural tests should be equivalent for a homogeneous material but with larger strains and any degree of heterogenity can differ markedly.

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2 Test Piece Preparation

2.1 Introduction

With the exception of tests on whole products, before a test can be carried out test pieces have to be obtained in the form and dimensions specified. Essentially, the two possibilities are directly moulding or cutting from a sheet or product. In addition to specifying dimensions (and number needed) the test method standard may favour one of these possibilities but even if test pieces are normally directly moulded to shape (as is usual for tensile tests) there will always be circumstances when cutting in some form is necessary. This will be the case, for example, when a product but no moulding material is available.

The properties of a material, and hence the test results, are dependent on how the material was formed, not only on whether there was any cutting or machining involved but on the details of mould configuration and moulding conditions. Consequently, exactly how a test piece was formed is essential information for understanding the significance of the results. For results to be comparable it is essential that a consistent procedure is used for the test piece production.

The best way of producing test pieces is open to debate. To investigate the properties of a material as they are in a product clearly needs test pieces to be formed by cutting or machining from that product. This applies not only to mouldings but perhaps particularly to extrusions and sheets of film. To obtain material properties for use in a data sheet or database the preferred approach will be to mould using standardised procedures and conditions. For investigations relating to a product the conditions relevant to production may be of more interest.

International and national standards for moulding test pieces have been existence for many years but have not been followed as frequently as they should. The relatively recent standards for producing single and multi-point comparative data [1-5] quite specifically state that they shall be used together with the conditions given in the appropriate material standard if this exists.

The methods that have been standardised for mixing plastics materials and moulding test pieces are outlined in the following sections but it is recognised that in many cases the expertise for processing the materials will be separate from the testing laboratory.

2.2 Mixing

In most cases where moulding of test pieces is involved, the material arrives in the laboratory in the form of granules or powder which can be directly moulded without any mixing. Certain materials to be compression moulded require the production of a preform by milling and this is recognised in, for example, ISO 293 [6] (see section 2.3).

Where compounding, blending or mixing of ingredients is necessary before moulding this would be carried out using procedures appropriate to the material in question but there are no general standard procedures. Where liquid resins have to be mixed, for example in casting methyl methacrylate or using a polyester resin to produce a laminated test sheet, it is usual to refer to instructions from the manufacturer of the resin.

There are standard procedures for producing polyvinyl chloride (PVC) pastes in ISO 4612 [7], using a planetary type mixer, and in ISO 11468 [8] by the dissolver method. Harrison and co-workers [9] investigated the particular problems of producing test pieces from plasticised PVC, finding that milling temperature had the biggest single influence on tensile strength.

2.3 Moulding

When considering moulding of test pieces or test sheets the first consideration is whether to produce by injection or compression. The orientation produced in an injection moulded test piece may yield very different results from those from a relatively non-oriented compression moulding process. There have been many studies which illustrate this point and also of the effect of other parameters such as temperature and pressure. Crawford and co-workers [10] made a comprehensive study of the effect of injection moulding parameters and their effect on mechanical properties. The magnitude of effects will be dependent on the particular conditions and material. As a general rule, the method selected will be that most appropriate to the end use of the material.

There are several international standards which give general requirements for moulding thermoplastic and thermoset materials [6, 11-16] These can be used in conjunction with standards for particular polymers. The information on producing test pieces is generally contained in Part 2 of these standards titled '*Preparation Of Test Specimens and Determination of Properties*'. Those that exist currently are given in **Table 2.1**.

It is common for the test piece moulded to conform to ISO 3167 [39] which specifies multi-purpose test pieces. Two test pieces are detailed but type A is preferred. Both are tensile dumbbell shapes which are multipurpose because, in addition to the use for tensile properties, various other tests can be carried out by machining the appropriate shape from the moulding.

Table 2.1 Standards for particular plastics		
ISO	**Material**	**Reference**
1163	Unplasticised PVC	[17]
1622	Polystyrene	[18]
1872	Polyethylene	[19]
1873	Polypropylene	[20]
1874	Polyamide	[21]
2580	Acrylonitrile/butadiene/styrene	[22]
2897	Impact resistant polystyrene	[23]
2898	Plasticised PVC	[24]
4613	Ethylene/vinyl acetate	[25]
4894	Styrene/acrylonitrile	[26]
6402	Impact resistant acrylonitrile/styrene	[27]
7391	Polycarbonate	[28]
7792	Thermoplastic polyester	[29]
8257	Poly(methyl methacrylate)	[30]
8986	Polybutene	[31]
9988	Polyoxymethylene	[32]
10366	Methyl methacrylate/acrylonitrile/butadiene/styrene	[33]
12086	Fluoropolymers	[34]
13000	Polytetrafluorethylene semi-finished products	[35]
14910	Thermoplastic polyester/ester and polyether/ester	[36]
15103	Polyphenylene ether	[37]
15526	Polyketone	[38]

ISO 293 [6] covers compression moulding of thermoplastic materials and gives fairly basic requirements for the press, moulds and procedure. Both frame type and positive moulds are allowed and there are four choices of cooling rate. Consequently, working to this standard alone could produce considerable variation in properties and reliance is placed on more specific instructions being given in the material specification. The ASTM equivalent is D4703 [40] and there is also a method specifically for compression moulding polyethylene test sheets and test pieces which is similar [41]. In ASTM D4703, details of conditions for various materials are given in an appendix.

Injection moulding of thermoplastics is covered by three parts of ISO 294 [11-13], Part 1 deals with general principles and moulding of multipurpose and bar specimens, Part 2 with small tensile bars and Part 3 with small plates.

In Part 1, the general principles are largely a comprehensive set of definitions and a diagrammatic representation of the injection moulding cycle. The point is made that ISO moulds, as specified in the standard, are strongly recommended for the acquisition of data intended to be comparable. Part 1 includes the ISO A and B moulds, A being the multipurpose test pieces of ISO 3167 [39] and B for bars. The moulds are not tightly specified but recommendations are quite comprehensive. There are also briefer descriptions of a single cavity mould and a family mould which can include dumbbells, bars and disks.

Reciprocating screw type injection machines are specified with tolerances for times and temperatures and recommendations for screw diameter and locking force. The procedure for moulding is outlined and there are requirements for measuring mould and melt temperatures.

Part 2 specifies a four cavity mould, the ISO C mould, for small bars which are the type 4 test piece of ISO 8256 [42] for tensile impact. Part 3 specifies two twin cavity moulds for disks, ISO D_1 and D_2 moulds, which differ in the thickness of the test pieces produced. The disks are intended for use in a variety of tests and some recommended applications are given in an appendix.

The ASTM methods, which are not identical, are given in D3641 [43]. Somewhat curiously, there were also separate ASTM methods that were identical to ISO 294 Parts 1 and 3 but these seem to have disappeared.

A modular system of injection moulds for test pieces has been described by de Jong [44]. During the course of the revision of ISO 294 [11-13] an interlaboratory comparison was carried out to obtain precision data and this has been reported [45]. Most interestingly, it concludes that for styrene materials at least, the reproducibility of different moulds, used in accordance with the ISO standard, is no worse than the reproducibility of impact and tensile testing procedures.

A further part of ISO 294 for producing test pieces for determining anisotropy of the properties of thermoplastic mouldings is at a committee draft stage. This is simply using a two cavity mould to produce plates from which tensile test pieces can be machined.

Compression moulding of thermoset materials is covered by ISO 295 [14] which is applicable to phenolics, aminoplastics, melamine phenolics, epoxides and unsaturated polyesters. A positive type mould is specified, the example given being the multi-purpose test piece of ISO 3167 [39]. Moulding conditions are specified for each plastic type, including pre-treatment and temperature.

The ASTM equivalent D5224 [46] is similar but not technically equivalent to the ISO. There is also an ASTM method D1896 [47], for which there is no ISO equivalent, for transfer moulding. This specifies a five test piece mould.

ISO 10724 [48, 49] deals with injection moulding of thermosets in two parts, Part 1 is general principles and multi-purpose test pieces and Part 2 small plates.

The style and content of ISO 10724 are similar to that of ISO 294. Part 1 specifies the same ISO type A mould, but there are no alternatives. Part 2 covers the ISO $D_1$and D_2 moulds.

The ASTM method is D3419 [50] and specifies the same five cavity mould as D1896 but is, as usual for ASTM standards, not identical.

ASTM D4204 [51] is concerned with preparing plastic film specimens for a round robin study and covers the taking and distributing samples rather than moulding.

Fibre reinforced plastics test pieces are prepared by such methods as hand lay-up, moulding of a prepreg and spiral winding. ISO 1268 [52-55] has currently 4 parts. Part 2 describes contact and spray up moulding, Part 3 wet compression moulding, Part 5 filament winding and Part 7 resin transfer moulding. Presumably there are other parts to follow. An ASTM method [56] covers the production of ring test pieces.

Modelling of injection moulding of fibre reinforced materials has been investigated by Guenther and co-workers [57] who concluded that drastic changes in properties can result from changing moulding parameters. Dickson and co-workers [58] have discussed vacuum bagging techniques for producing thin walled tubes and Tudgey [59] has described an improved method for production of carbon fibre reinforced plastic test bars.

The general conclusion is that there is no shortage of standards covering the moulding of plastics materials but their use will not always ensure comparable results nor will they cover all the situations that might be needed in practice. The moulds for producing test pieces are described in the standards cited previously but test pieces are defined in a range of test method standards. There is currently a proposal to create a more easily maintained system whereby all the test pieces are defined in one standard and all the moulds in another, which would mean combination of some of the moulding standards.

2.4 Stamping from Sheet or Film

This operation is only applicable to flexible sheet materials and film but for such materials is the most convenient way of producing test pieces. Despite seemingly being a very simple operation, considerable care has to be taken over the condition of cutters to

ensure that as far as possible the cut edges are free from defects which would effect measured properties.

A study made many years ago [60] with four laboratories using four different methods (die, manual razor, rotary and shear) to cut specimens from very thin films of polyethylene, polyester and polycarbonate illustrated the very large variability which can occur. Yield strength data showed the least scatter, 10% or less covering all four techniques and laboratories. However, much greater divergences were found with ultimate tensile strength (30%) and elongation at break (up to approximately 75%), with the laboratories being relatively consistent and the techniques accounting for the majority if not all the variation. Die-cut specimens were clearly the worst in this exercise which probably demonstrates how difficult it is to keep dies in really good condition.

In this work simple rectangular specimens were examined, many more complex shapes, such as dumbbells, could not be conveniently prepared other than with a die. For thicker sheets of flexible material dies or a rotary cutter are the only possibilities even for straight cuts.

It is essential that dies are very sharp and free from nicks or unevenness in the cutting edge which would produce flaws in the test piece. Even with the sharpest cutter there is a tendency for the cut edges of the test piece to be concave and 4 mm is the absolute maximum thickness as the dishing effect becomes more severe as the thickness increases. Dies for stamping can be of two types, fixed blade and changeable blade. Changeable blade type cutters make use of sharpened strips of steel rather like long single-edged razor blades. These have the obvious advantage of being very sharp when new and are simply replaced when blunt. They are commonly used for simple shapes such as parallel sided strips but, although very successful dumbbell cutters can be made in this manner, such dies do not appear to be commercially available. Razor blades or similar are best for producing a slit as in trouser tear test pieces.

ISO 2818 - preparation of test specimens by machining [61], has just one paragraph on stamping from a sheet, stating the need for sharp dies and the use of a slightly yielding backing material. The standard for rubber, ISO 4661 [62], has rather more information.

A suitable design for the cutting edge profile of a fixed edge blade type is given in ISO 4661 and the standard also points out the necessity for the die to be suitably rigid and the desirability of some form of test piece ejection system. If there is no automatic ejection system some care has to be taken not to damage the cutting edge of the die or the test piece whilst prodding with whatever sharp object has come to hand.

ISO 4661 does not give any details of the press which should be used with the dies for stamping operations and the particular design of press is probably not important as long as it operates smoothly and vertically to the test piece surface. In practice, quite a variety of

presses are to be found and, although the choice is largely a matter of personal preference, there are several points which can be considered. Automatic sample ejection has been mentioned, but this is not very easy to combine with rapid interchange of die shapes. Some toggle action presses require rather more force to operate than is convenient for routine use. Recoil types can be operated very rapidly but are found by some people to be difficult to use. For general use there is a lot to be said for the screw action type operated by a large hand-wheel. Motorised presses are only worthwhile if the volume of work is very large.

Rotary cutters can be used to produce discs or rings from thin sheet and are necessary for sheet above about 4 mm thick to prevent distortion. Generally, such cutters are used on vertical drilling machines and may consist of either annular or part annular blades. A number of designs have been tried including the incorporation of a second blade simultaneously cutting a large diameter disc. No particular design is referenced in ISO 4661 nor is any recommendation given as to suitable speeds of rotation.

The cutting of flexible materials is generally made much easier if a lubricant is applied to either the material or the cutting blade. A lubricant which has no effect on the plastic must be used and a weak solution of detergent in water is usually suitable. It is not normally necessary to lubricate for stamping operations but it is often essential when using a rotating cutter.

To keep fixed blade cutters in good condition means frequent sharpening. It cannot be over-emphasised that many low results and cases of poor reproducibility are caused by blunt or chipped cutting dies. People take them for granted but they need hours of attention and sharpening is a very skilled job. This can be done by the manufacturer or by workshop personnel, but only rarely is the necessary facility and expertise available in the laboratory. A technique suitable for the laboratory has been described by Ennor [63] which uses shaped stones in a vertical drilling machine and this procedure is reproduced in ISO 4661. Experience at Rapra has shown that drilling machines generally revolve too slowly and better results may be obtained using the high speed router of a plastics test specimen machining apparatus.

It should be noted that the procedure of using cylindrical stones with the die mounted on a tilted base is inaccurate on the curved parts of the die.

2.5 Machining

The term machining is used rather broadly here to cover cutting shapes from sheet or products and reduction of thickness. Rigid materials other than very thin film cannot be cut or stamped with a blade. ISO 2818 mentions milling, planing, sawing, broaches and

abradants although it is not clear what differentiates planing from milling. Some advice is given on the tools for these operations and there is a table with recommended conditions such as speeds and tool geometry.

It is usual to rough out rectangular test pieces using a saw or cutting disk and for some tests where the edge finish is not important no further preparation is necessary. Circular disks are similarly cut with a tubular saw. Hardened steel, tungsten carbide or diamond tipped blades are all used depending on material and blade life which can be accepted. Care must always be taken that, particularly due to blunt blades, heat build up is not sufficient to cause degradation.

More generally, the final shape is formed by milling or planing, although a stack of disks can be turned on a lathe. Standard machine tools may be used provided they can be operated at a high enough speed and be fitted with suitable cutters. Tungsten carbide or diamond tipped milling cutters give the longest life but high-grade steel cutters may be used with many materials and will give an acceptable service life. Tool rotation speeds vary according to the material machined and are best found by trial and error, but generally lie within the range from 8000 to 30000 rpm. In this context, is doubtful whether the conditions given in ISO 2818 are comprehensive or the best in all cases. Recent investigations into machining fibre reinforced materials have been made by Puw and Hocheng [64] and Hocheng and co-workers [65]

The production of curved test pieces, e.g., dumb-bells, require some form of copying milling machine or router. Many variations have been used but all essentially consist of a motor-driven cutting tool against which the blank is machined, guided by a pin following a template of the required shape.

When machining some plastics, a coolant is necessary if a satisfactory surface finish is to be obtained. A jet of compressed air is suitable in many cases, but if a liquid coolant is used care must be taken to ensure that it does not affect the plastics material being machined.

ISO 2818 does not offer any recommendation about reducing the thickness of a test piece blank or sheet but it could be implied that this could be achieved by sawing followed by milling.

The technique of grinding (or buffing) plastics is not so widely practised as for rubber where it is quite commonly used for reducing the thickness of rubber test pieces. If grinding is used for plastics it is important to use the right grade of abrasive wheel. Open grit wheels give the best results but, in the absence of specific information, the advice of the abrasive wheel manufacturer should be sought both as to the correct abrasion grade and the optimum speed of rotation of the wheel. Generally, the dangers of heat build up restrict the use of this approach other than to use fine abrasives for finishing. Use of abrasives to finish notches cut in test pieces is not permitted by ISO 2818.

When reducing the thickness of sheet to produce test pieces of the specified dimensions the specification for the material under test, or the test method, should be consulted because some require that one face of the manufactured sheet should be left intact, while others specify that both surfaces shall be machined uniformly to give the required thickness.

A particular case of machining rigid plastics is the production of notches in impact test pieces. These can be cut on conventional workshop milling or shaping machines or, for certain shaped notches, produced by sawing and drilling. This latter process is likely to be variable. It is obviously more convenient for the technologist to produce notches in the laboratory and manual apparatus for standard notches is available, generally based on a broach. Very sharp notches are usually produced by tapping or sliding a razor blade into the machined root of a relatively blunt notch. Kazakov [66] has described devices for notching single edge notch (SEN) test pieces and pipe sections.

As impact strength can be very sensitive to notch geometry it is essential that notches are accurately and reproducibly cut. ISO 2818 specifically excludes the use of abrasives on notches.

For cutting test pieces from bulky flexible products the practices used for rubbers are applicable [67]. Rough shapes are obtained in an arbitrary fashion using various knives. The test piece is then obtained by buffing to remove relatively small quantities, such as surface patterns, or by slitting using machines designed for the leather industry

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3 Conditioning

3.1 Introduction

Plastics are affected by variations in ambient conditions such that their properties are dependent on the conditions at the time of testing and perhaps also on the conditions between production and test. The process of conditioning test pieces is aimed at providing a reference point for the measurement of properties. The reference conditions chosen are to some extent always arbitrary but are generally selected to correspond with what may be considered 'normal'. Because it is obviously important for results to be comparable, reference conditions have been standardised. What is considered normal, or what is thought to be the most appropriate conditions, can vary depending on the viewpoint or local conditions, so even standards are not completely definitive.

The conditioning process can be divided into:

- What happens between the process that formed the material and it being prepared for test
- Bringing test pieces to equilibrium with standard conditions, and
- The conditions during test

The term conditioning is generally used for the second of these with the first being referred to as storage and the third as test conditions.

The actions of conditioning can involve temperature, humidity and mechanical (and sometimes electrical) stressing. The attention paid to these agents is generally in the order given, with temperature virtually always standardised and controlled, humidity often controlled and mechanical conditioning more often than not ignored. In fact, there are circumstances where the mechanical condition could have more effect on the result than the variations in temperature likely to result without control.

3.2 Storage

The problem with storage from the tester's point of view is that he or she usually has no control over it. In rubber testing, an attempt is made in ISO 471 [8] to define standard storage conditions which is paraphrased next:

The minimum time between forming the material and testing shall be 16 hours. Longer may be necessary for whole products or test pieces cut from products and you are expected to find the time in the specification or relevant test method.

For non-product tests the maximum time shall be 4 weeks and comparative tests should be after the same time interval. For product tests the time should not exceed 3 months or at least no longer than 2 months after receipt

In plastics testing ISO 291[1] admits defeat and says nothing.

The common sense is that you aim to avoid exposing the material to any degradative influences such as high temperatures or UV light and avoid extremes of humidity. It is important that time is allowed before testing for transient changes to stabilise but thereafter the storage period is preferably as short as possible. Any changes occur most rapidly immediately after forming and 16 hours must be the bare minimum with 2 or 3 days being preferred for most materials. The case of plasticised polyvinyl chloride (PVC) is often quoted when 7 days is needed to reach something approaching equilibrium and for hardness testing 7 ± 0.2 days is specified. Even with ideal storage conditions it has to be appreciated that properties will slowly change with time and strictly comparable results may need equal storage periods. The changes in properties brought about by physical ageing or post moulding crystallisation can be exploited by choosing the time delay before testing (and/or changing the rate of cooling from the mould) to maximise the property of interest.

3.3 Conditioning

The most common standard atmosphere nowadays is 23 °C and 50% relative humidity (RH). Previously, 20 °C and 65% RH was generally used but nobody seems very sure why the change was made, other than we have got used to warmer rooms. However, the 23/50 conditions do not reign supreme. For all polymer materials 27 °C and 65% RH is allowed for use in tropical countries and for coated fabrics 20/65 is still common. Calibration laboratories generally operate at 20 °C so that even in such a basic thing as standard temperature we cannot all agree.

For plastics the latest edition of ISO 291 has two standard atmospheres, 23/50 and 27/65, with 23/50 being preferred. These conditions are qualified by noting that they apply to normal altitudes with atmospheric pressure between 86 and 106 kPa and air velocity equal to or greater than 1 m/s. What happens under other conditions and the significance of this note is not immediately apparent. ISO 291 also allows ambient atmospheres which may be control of temperature only (23 °C or 27 °C) or control of neither

temperature nor humidity. The implication is that control of temperature and humidity is normally required for plastics. For many materials and properties it is debatable whether humidity needs tight control (for rubbers humidity is rarely controlled).

ISO 291 has two classes of tolerance, ± 1 °C and ± 5% humidity or ± 2 °C and ± 10% humidity. Although normally used in these pairs, it is possible to have other combinations. Traditionally, the normal tolerances were ± 2 °C and ± 5% RH. If tighter tolerances were needed ± 1 °C and ± 2% RH were specified. The latest ISO 291 recognises the fact that ± 2% RH is effectively impossible to attain and ± 5% RH is often debatable. It is easy to glibly claim that tight tolerances are being used but the practice is that with spatial variation and measurement uncertainties control is less good than it appears. For so called ambient temperature, ISO 291 claims that air temperature varies between 18 and 28 °C. Clearly, the range could be much less or higher than this. This revision of ISO 291 can be applauded for at last being realistic about humidity control but in other ways it is drafted in a most clumsy manner.

The question in practice is how important are temperature and humidity variations and nobody likes to be too specific. The effect will be dependent on both material and property. Test methods will specify the atmosphere to be used, commonly 23 ± 2 °C and 50 ± 10% RH, but exceeding the specified tolerances may make negligible difference for some materials whereas for others tighter tolerances may be desirable. For most materials, changes in property over the range 20 to 25 °C are unlikely to be significant, the largest exception being if the material's glass transition (T_g) is around ambient. Similarly, variations in ambient humidity will have negligible effect in many cases, but here there are notable exceptions in plastics that are very moisture sensitive. To be on the safe side the general rule is to follow the standard, but there are probably more discrepancies from other testing factors than from conditioning atmosphere control. The tighter tolerance on temperature is rarely justified and the ± 2% tolerance on humidity had become a joke.

Conditioning has to be carried out for sufficient time for the material to reach equilibrium. The plastics standard requires not less than 4 hours for temperature alone and not less than 88 hours for temperature and humidity. The truth probably depends mostly on what conditions the material is being conditioned from. To reach temperature equilibrium will rarely require as much as 4 hours but if the temperature change is large it can be envisaged that transient effects may take at least this time to disappear. If the humidity in storage was very different to that at conditioning then even 88 hours may be insufficient and far too short for some plastics. This is recognised in a note in ISO 291 and an annex gives alternative procedures which could be used. This annex mentions the possibilities of conditioning at an elevated temperature to remove moisture or conditioning until no weight change indicates equilibrium has been reached.

Special conditions are specified for polyamides which are particularly sensitive to moisture content. An accelerated procedure is given in ISO 1110 [2] involving exposure at 70 °C and 62% RH. The question is whether with such moisture sensitive materials tests should be made dry, with maximum water absorption or somewhere in the middle. The two logical answers would appear to be the conditions which give the answer you prefer or at all three. The order of magnitude of the effect of moisture level is well illustrated by Sichina and Bizet [3].

The normal procedure is to condition the material or product after any test piece preparation, i.e., immediately prior to testing. It is, however, necessary to check the particular test method as there can be special requirements for time intervals after machining and exposure to liquids.

ISO 291 is reproduced as BS EN ISO 291. A restricted form of ISO 291 has been published as a European [4] and a British standard for glass reinforced materials [5] which now needs revising. The equivalent ASTM standard is ASTM D618 [6] which designates 23/50 as the standard laboratory atmosphere but in the current version has retained the old tolerances on humidity and specifies only 40 hours for test pieces less than 7 mm thick. It also lists five other conditioning procedures including drying in an oven, at high humidity and in water. It therefore covers the ground of the appendix to ISO 291 and ISO 1110 but does not include conditions at 20/65, 27/65 or 70/62.

3.4 Heat Treatment

The normal standard conditioning procedures assume that testing takes place essentially as received, the conditioning being to bring the material to equilibrium with a reference point which represents ambient conditions. However, when a material was moulded it may have been cooled slowly or very quickly and this will affect its properties, or it may have changed over time. There exists, therefore, the possibility of re-heating the material with the intent of modifying its properties. Although not commonly applied in routine testing, it is well understood that amorphous plastics materials can be 'refreshed' by heating to above T_g and re-cooled or annealed at just below T_g.

3.5 Mechanical Conditioning

This is a feature generally associated with particular rubber tests, it being known that elastomers containing fillers have their stress-strain behaviour modified when they are deformed. Repeated deformation under the same constraints leads to an equilibrium stress-strain curve being produced. Given sufficient time the filler - rubber structure can

re-build and there is a return to the original stress-strain behaviour. Mechanical conditioning is very rarely carried out on plastics and it is generally assumed to be unnecessary. However, Donoghue and co-workers [7] have looked at the effects of mechanical conditioning on glass fibre reinforced epoxy resins. In dynamic tests, mechanical conditioning takes place automatically.

3.5 Test Conditions

Most tests are, quite logically, carried out under the same conditions as used for conditioning. It is, however, highly debatable whether in most cases control of humidity is necessary. The time from removal from the conditioning atmosphere to completing the test is usually short in comparison to time needed for any change in moisture content. The practical importance of this is that many laboratories do not really need the expense of the whole room being humidity controlled, whatever the standard may say. ISO 291 says somewhat ambiguously that specimens shall be tested in the same atmosphere or at the same temperature in which they have been conditioned.

When tests are made at elevated or subnormal temperatures, there is effectively a second conditioning period required to bring the material into equilibrium with the test temperature. Humidity is then conveniently forgotten. At elevated temperatures the time of conditioning should be as short as possible to limit any degradation. This time will depend on several factors, the major ones being temperature difference between ambient and the conditioning temperature, the dimensions of the test specimen, the surface heat transfer coefficient and the thermal diffusivity of the material. Fortunately, for normal conditioning purposes, these factors do not need to be known with great precision. Tables of calculated minimum times were published by Brown and Hands and can be found reproduced as Appendix A. ISO 291 [1] gives no guidance and ASTM D618 [6] is not much more helpful, stating no less than is necessary to reach equilibrium subject to 5 hours maximum.

However, one must be clear whether the test is looking to measure the effect of temperature alone, or whether structural changes in the molecular morphology are also to be examined. This is generally a low temperature concern. Prolonged elevated temperature conditioning is normally associated with heat ageing effects. At low temperatures, polymers may crystallise or undergo other reversible structural changes and these may take one or two orders of magnitude longer to reach equilibrium than the temperature. When undertaking such tests, therefore, the details of the test method or specification should be carefully followed or quite different results may arise.

ISO 291 does not give a list of preferred test temperatures but there is a list in ASTM D618, in the rubber equivalent, ISO 471 [8], and in a general standard, ISO 3205 [9].

They do not totally agree on temperatures and the tolerances tend to be tighter in the ASTM and the rubber standard, perhaps reflecting the temperature sensitivity of polymers. The rubber international standard can be sensibly applied to plastics.

It is a not uncommon practice where there is no temperature control of the test apparatus to condition the test piece in a cabinet and test it as fast as possible after removal. Quite clearly, this is at best a dodgy process but can be adequate for a very rapid test such as impact with a thick section test piece.

3.6 Apparatus for Conditioning

3.6.1 Air-Conditioned Rooms

Since test methods for polymers almost invariably require the test to be performed under reasonably tightly controlled temperatures, it is necessary for the testing laboratory to have appropriate air-conditioning if the precise requirements of the standard are to be met throughout the working day. Reliable automatic control is necessary to enable the tolerances to be held overnight and over weekends when laboratory staff are not present. Specialists in heating systems are usually consulted when considering the installation of air-conditioning as the design needs to give as uniform a temperature throughout the working area as possible, especially taking into account the heat load from equipment. It is important for them to fully appreciate that the tolerances are to be maintained at all times and not most of the time - requirements for the testing laboratory are much more stringent than those for the average office environment. Provided the laboratory does not have too many windows and outside doors, the provision of good temperature control need not be an excessively expensive option. To help maintain tolerances even inside doors should be kept closed as much as possible.

For plastics testing it is advantageous to include humidity control as well but this is invariably a more technically demanding requirement, which in turn carries a significant cost penalty. Fortunately, it is not essential in many circumstances as test pieces can be conditioned in a cabinet and transferred quickly to the test machine. Where a temperature and humidity controlled room is to be provided, it is useful to have the controlled room situated within another room and with the minimum of windows and doors. Specialist advice to design the system is usually essential.

3.6.2 Enclosures

Humidity and temperature controlled cabinets are commonly used for conditioning and also for moisture ageing tests. There are two types of humidity controlled cabinet in

general use: salt-tray cabinets and moisture-injection cabinets. The simpler type are the salt-tray cabinets in which saturated salts or standard solutions provide the appropriate humidity in the test space at a given controlled temperature. The design of these cabinets is critical if the humidity in particular is to be kept within the required tolerances throughout the working space. ISO 483 [9] contains useful information on the use of such cabinets and although specifically written for plastics it is equally applicable to all polymeric materials. To attain conditions of 23 °C and 50% RH, a glycerol solution of refractive index 1.444 is proposed. The tolerance on the refractive index is very tight, being ± 0.005 for a variation of ± 5% relative humidity and ± 0.002 for a RH variation of ± 2%. Martin [12] demonstrated that the expected humidity in these cabinets is not reached if the test pieces absorb water at a significant rate.

The more sophisticated injection humidity cabinet permits a wide variation in temperature and humidity to be created with a few simple settings of the controls. The humidity is measured by a suitable moisture sensor, such as a wet and dry bulb hygrometer or capacitive sensor, and this is used to control the injection of moisture into the chamber. Through the use of suitable control circuits it is also possible to cause such a chamber to cycle in temperature and/or humidity so that varying ambient conditions over wide extremes may be simulated for assessing such effects on the environmental resistance of polymer products.

3.6.3 Hygrometers

Dew point hygrometers are usually used as reference standards for measuring relative humidity, but for normal use in equipment and enclosures, capacitance/impedance instruments or wet and dry bulb thermometers are generally found. For the latter, platinum resistance thermometers are preferred as they are very stable and robust. Even so, for these to operate accurately there should be an air flow over them of at least 3 m/s. BS 4833 [13] provides hygrometric tables for use with wet and dry bulb thermometers and ISO 4677 [14, 15] specifies the measurement of RH using aspirated or whirling psychrometers (more commonly called hygrometers). Hair or paper hygrometers may prove of use in some instances because of the size and relatively low cost, but it must be understood that they are generally very inaccurate and should never be used where precise humidity readings are needed or where long-term stability is required.

It is now recognised that ± 2% is about at the limit of what the best calibration laboratories can achieve for the uncertainty of humidity sensor readings. On that basis one would have to keep the humidity of the laboratory absolutely unchanging in order to ensure compliance with a ± 2% humidity tolerance. Even ± 5% RH is not easy to achieve, particularly in a room, and this the reason why the latest version of ISO 291 has relaxed the conditioning and testing tolerances.

3.6.4 Thermometers

There are many types of temperature measuring instrument available and, while the electronic versions are very widely used, the ordinary mercury-in-glass, or alcohol-in-glass thermometers are still prevalent. There is more that can go wrong with a mercury-in-glass thermometer than is often appreciated, so there is a need to have them calibrated frequently and the mercury thread should be examined for continuity before use. For accurate work the thermometer must be immersed to the correct depth. Alcohol in glass thermometers may have to be used for certain low temperature tests, since mercury freezes at about –39 °C. Alcohol thermometers are not as accurate as the mercury thermometers and need even more careful checking for calibration. There are a number of standards for mercury in glass thermometers including BS 593 [16], ISO 653 [17], ISO 654 [18], ISO 655 [19] and ISO 656 [20].

The sensing elements for electronic thermometers vary, with the most widely used being thermocouples or platinum resistance thermometers. The latter are more stable and linear, but are less robust than the former and the temperature range they can cover is not as great since changing the metal combinations in the thermocouple enables very wide temperature ranges to be achieved. As noted for the liquid-in-glass thermometers, frequent calibration is still a requirement. The various parts of BS 1041 [21] give guidance into the selection and use of thermometers of various types and there is also an ASTM manual on the use of thermocouples [22].

3.6.5 Apparatus for Elevated and Sub-Ambient Temperature

Generally conditioning just prior to testing at elevated or sub-ambient temperatures takes place in a test chamber that is attached to the test machine itself and is an integral part of the machine. The '*Rapra Guide to Test Equipment*' [23] offers comment on the types of enclosure available and any particular requirement for a test will be indicated in the relevant section of this book. For mechanical tests the chamber is normally an air oven with injection of liquid nitrogen for sub-ambient use. ISO 3383 [24] covers both elevated and sub-ambient conditions for rubbers, giving fairly elementary information on the types of chamber construction and heat transfer media. Within ASTM there is a standard covering sub ambient conditioning for rubbers, D3847 [25].

However, there are times when conditioning has to be carried out in a separate chamber and then the sample removed and tested as quickly as possible. An example of the latter is the low temperature pendulum impact testing of plastic bars, where having the whole instrument in the cold chamber would risk the freezing up of the pendulum bearings thereby influencing the outcome of the test.

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Appendix A

Tables of Thermal Equilibrium Times

Table 3.1 - Cylinders								
Diameter (mm)	Height (mm)	Temperature (°C)	Time to 1 °C off equilibrium (min)					
			Rubber		Crystalline plastic		Amorphous plastic	
			in air	in oil	in air	in oil	in air	in oil
64	38	–50	130	75	135	60	130	80
		0	95	60	100	45	95	65
		50	105	65	115	50	105	70
		100	130	80	140	60	130	85
		150	145	85	155	65	145	90
		200	155	90	165	70	155	95
		250	160	90	170	75	160	100
40	30	–50	75	35	85	30	75	40
		0	55	30	60	25	55	35
		50	60	30	70	25	60	35
		100	75	35	85	30	75	45
		150	85	40	95	35	85	45
		200	90	45	100	35	90	50
		250	95	45	105	40	90	50
37	10.2	–50	35	10	40	10	35	10
		0	25	10	30	10	25	10
		50	30	10	35	10	25	10
		100	35	10	40	10	35	10
		150	40	10	45	10	35	10
		200	40	10	50	10	40	15
		250	45	10	50	10	40	15

Table 3.1 - Cylinders *continued*								
Diameter (mm)	**Height (mm)**	**Temperature (°C)**	**Time to 1 °C off equilibrium (min)**					
			Rubber		**Crystalline plastic**		**Amorphous plastic**	
			in air	**in oil**	**in air**	**in oil**	**in air**	**in oil**
32	16.5	–50	45	15	50	15	45	20
		0	35	15	40	10	30	15
		50	35	15	45	15	35	15
		100	45	20	55	15	45	20
		150	50	20	60	15	50	20
		200	55	20	65	20	50	20
		250	55	20	65	20	55	25
29	25	–50	50	20	60	20	50	25
		0	40	15	45	15	40	20
		50	45	20	50	15	40	20
		100	55	25	60	20	50	25
		150	60	25	70	20	55	25
		200	65	25	70	25	60	30
		250	65	25	75	25	65	30
28.7	12.7	–50	35	10	40	10	35	15
		0	25	10	30	10	25	10
		50	30	10	35	10	30	10
		100	35	15	45	10	35	15
		150	40	15	50	10	40	15
		200	45	15	50	15	40	15
		250	45	15	55	15	40	15
25	20	–50	40	15	50	15	40	20
		0	30	15	35	10	30	15
		50	35	15	40	10	35	15
		100	45	15	50	15	40	20
		150	45	20	55	15	45	20
		200	50	20	60	15	50	20
		250	50	20	60	15	50	20

Table 3.1 - Cylinders *continued*								
Diameter (mm)	**Height (mm)**	**Temperature (°C)**	**Time to 1 °C off equilibrium (min)**					
			Rubber		**Crystalline plastic**		**Amorphous plastic**	
			in air	**in oil**	**in air**	**in oil**	**in air**	**in oil**
25	8	–50	25	5	30	5	25	10
		0	20	5	20	5	20	5
		50	20	5	25	5	20	5
		100	25	5	30	5	25	10
		150	30	10	35	5	25	10
		200	30	10	35	10	30	10
		250	30	10	35	10	30	10
25	6.3	–50	20	5	25	5	20	5
		0	15	5	20	5	15	5
		50	20	5	20	5	15	5
		100	20	5	25	5	20	5
		150	25	5	30	5	20	5
		200	25	5	30	5	25	5
		250	25	5	30	5	25	5
13	12.6	–50	20	5	25	5	20	5
		0	15	5	20	5	15	5
		50	20	5	20	5	15	5
		100	20	5	30	5	20	10
		150	25	10	30	5	25	10
		200	25	10	30	5	25	10
		250	25	10	35	5	25	10
13	6.3	–50	15	5	20	5	15	5
		0	10	5	15	5	10	5
		50	15	5	15	5	15	5
		100	15	5	20	5	15	5
		150	20	5	20	5	15	5
		200	20	5	25	5	20	5
		250	20	5	25	5	20	5

Table 3.1 - Cylinders *continued*								
Diameter (mm)	**Height (mm)**	**Temperature (°C)**	**Time to 1 °C off equilibrium (min)**					
			Rubber		**Crystalline plastic**		**Amorphous plastic**	
			in air	**in oil**	**in air**	**in oil**	**in air**	**in oil**
9.5	9.5	–50	15	5	5	5	15	5
		0	10	5	5	5	10	5
		50	15	5	5	5	10	5
		100	15	5	5	5	15	5
		150	20	5	5	5	15	5
		200	20	5	5	5	15	5
		250	20	5		5	20	5

Table 3.2 - Flat sheets							
Thickness (mm)	**Temperature (°C)**	**Time to 1 °C off equilibrium (min)**					
		Rubber		**Crystalline plastic**		**Amorphous plastic**	
		in air	**in oil**	**in air**	**in oil**	**in air**	**in oil**
25	–50	135	90	115	80	145	100
	0	95	75	80	65	105	85
	50	110	80	90	70	120	90
	100	140	90	115	80	150	100
	150	155	95	130	85	165	105
	200	160	100	135	85	180	110
	250	170	105	140	90	185	115
15	–50	70	35	60	30	80	40
	0	50	30	40	25	55	30
	50	60	30	45	30	65	35
	100	75	35	60	30	80	40
	150	80	40	65	35	90	40
	200	85	40	70	35	95	40
	250	90	40	75	35	100	45
10	–50	45	15	35	15	50	20
	0	30	15	25	15	35	15
	50	35	15	30	15	40	15
	100	45	20	40	15	50	20
	150	50	20	40	15	55	20
	200	55	20	45	15	60	20
	250	55	20	45	20	60	20
8	–50	35	10	30	10	40	15
	0	25	10	20	10	30	10
	50	30	10	25	10	30	10
	100	35	10	30	10	40	15
	150	40	10	35	10	45	15
	200	40	15	35	10	45	15
	250	45	15	35	15	50	15

Table 3.2 - Flat sheets *continued*							
Thickness (mm)	**Temperature (°C)**	**Time to 1 °C off equilibrium (min)**					
		Rubber		**Crystalline plastic**		**Amorphous plastic**	
		in air	**in oil**	**in air**	**in oil**	**in air**	**in oil**
5	–50	20	5	20	5	25	5
	0	15	5	15	5	20	5
	50	20	5	15	5	20	5
	100	20	5	20	5	25	5
	150	25	5	20	5	25	5
	200	25	5	20	5	30	5
	250	25	10	20	10	30	5
3	–50	15	5	10	5	15	5
	0	10	5	10	5	10	5
	50	10	5	10	5	15	5
	100	15	5	10	5	15	5
	150	15	5	15	5	15	5
	200	15	5	15	5	20	5
	250	15	5	15	5	20	5
2	–50	10	5	10	5	10	5
	0	10	5	5	5	10	5
	50	10	5	5	5	10	5
	100	10	5	10	5	10	5
	150	10	5	10	5	10	5
	200	10	5	10	5	15	5
	250	10	5	10	5	15	5
1	–50	5	5	5	5	5	5
	0	5	5	5	5	5	5
	50	5	5	5	5	5	5
	100	5	5	5	5	5	5
	150	5	5	5	5	5	5
	200	5	5	5	5	5	5
	250	5	5	5	5	10	5

Table 3.2 - Flat sheets *continued*							
Thickness (mm)	**Temperature (°C)**	**Time to 1 °C off equilibrium (min)**					
		Rubber		**Crystalline plastic**		**Amorphous plastic**	
		in air	**in oil**	**in air**	**in oil**	**in air**	**in oil**
0.2	–50	5	5	5	5	5	5
	0	5	5	5	5	5	5
	50	5	5	5	5	5	5
	100	5	5	5	5	5	5
	150	5	5	5	5	5	5
	200	5	5	5	5	5	5
	250	5	5	5	5	5	5

Table 3.3 - Flat strips								
Width (mm)	**Thickness (mm)**	**Temperature (°C)**	**Time to 1 °C off equilibrium (min)**					
			Rubber		**Crystalline plastic**		**Amorphous plastic**	
25.4	12.7	–50	**45**	**15**	**50**	**10**	**40**	**15**
		0	30	10	35	10	30	10
		50	35	10	40	10	35	15
		100	45	15	55	10	40	15
		150	50	15	60	15	45	15
		200	50	15	60	15	50	15
		250	55	15	65	15	50	20
25.4	10.0	–50	35	10	45	10	35	10
		0	25	10	30	5	25	10
		50	30	10	35	10	30	10
		100	35	10	45	10	35	10
		150	40	10	50	10	40	10
		200	40	10	50	10	40	15
		250	45	10	55	10	40	15
25.4	9.5	–50	35	10	40	10	30	10
		0	25	10	30	5	25	10
		50	30	10	35	10	25	10
		100	35	10	40	10	35	10
		150	40	10	45	10	35	10
		200	40	10	50	10	40	10
		250	40	10	50	10	40	10
25.4	6.5	–50	25	5	30	5	25	5
		0	20	5	20	5	15	5
		50	20	5	25	5	20	5
		100	25	5	30	5	25	5
		150	30	5	35	5	25	5
		200	30	5	35	5	25	5
		250	30	5	40	5	30	10

Table 3.3 - Flat strips *continued*								
Width (mm)	**Thickness (mm)**	**Temperature (°C)**	**Time to 1 °C off equilibrium (min)**					
			Rubber		**Crystalline plastic**		**Amorphous plastic**	
25.4	5.0	–50	**20**	**5**	**25**	**5**	**20**	**5**
		0	15	5	20	5	15	5
		50	15	5	20	5	15	5
		100	20	5	25	5	20	5
		150	20	5	30	5	20	5
		200	25	5	30	5	20	5
		250	25	5	30	5	25	5
25.4	3.0	–50	15	5	15	5	10	5
		0	10	5	10	5	10	5
		50	10	5	15	5	10	5
		100	15	5	15	5	10	5
		150	15	5	20	5	15	5
		200	15	5	20	5	15	5
		250	15	5	20	5	15	5
25.4	2.0	–50	10	5	10	5	10	5
		0	10	5	10	5	5	5
		50	10	5	10	5	10	5
		100	10	5	10	5	10	5
		150	10	5	15	5	10	5
		200	10	5	15	5	10	5
		250	10	5	15	5	10	5
25.4	1.0	–50	5	5	5	5	5	5
		0	5	5	5	5	5	5
		50	5	5	5	5	5	5
		100	5	5	5	5	5	5
		150	5	5	10	5	5	5
		200	5	5	10	5	5	5
		250	5	5	10	5	5	5

Table 3.3 - Flat strips *continued*								
Width (mm)	**Thickness (mm)**	**Temperature (°C)**	**Time to 1 °C off equilibrium (min)**					
			Rubber		**Crystalline plastic**		**Amorphous plastic**	
15.0	15.0	–50	**35**	**10**	**45**	**10**	**35**	**15**
		0	30	10	35	10	25	10
		50	30	10	35	10	30	10
		100	40	10	45	10	35	15
		150	40	15	50	10	40	15
		200	45	15	55	15	40	15
		250	45	15	55	15	45	15
12.7	12.7	–50	30	10	35	10	30	10
		0	25	10	25	5	20	10
		50	25	10	30	10	25	10
		100	30	10	40	10	30	10
		150	35	10	40	10	35	10
		200	35	10	45	10	35	10
		250	40	10	45	10	35	10
12.7	10.0	–50	25	10	35	5	25	10
		0	20	5	25	5	20	5
		50	20	5	25	5	20	5
		100	30	10	35	5	25	10
		150	30	10	35	10	30	10
		200	30	10	40	10	30	10
		250	35	10	40	10	30	10
12.7	9.5	–50	25	10	30	5	25	10
		0	20	5	25	5	20	5
		50	20	5	25	5	20	5
		100	25	10	35	5	25	10
		150	30	10	35	10	30	10
		200	30	10	40	10	30	10
		250	35	10	40	10	30	10

Table 3.3 - Flat strips *continued*								
Width (mm)	**Thickness (mm)**	**Temperature (°C)**	**Time to 1 °C off equilibrium (min)**					
			Rubber		**Crystalline plastic**		**Amorphous plastic**	
12.7	6.5	–50	**20**	**5**	**25**	**5**	**20**	**5**
		0	15	5	20	5	15	5
		50	15	5	20	5	15	5
		100	20	5	25	5	20	5
		150	25	5	30	5	20	5
		200	25	5	30	5	25	5
		250	25	5	30	5	25	5
12.7	5.0	–50	15	5	20	5	15	5
		0	15	5	15	5	10	5
		50	15	5	15	5	15	5
		100	20	5	20	5	15	5
		150	20	5	25	5	20	5
		200	20	5	25	5	20	5
		250	20	5	25	5	20	5
12.7	3.2	–50	10	5	15	5	10	5
		0	10	5	10	5	10	5
		50	10	5	15	5	10	5
		100	10	5	15	5	10	5
		150	15	5	15	5	10	5
		200	15	5	20	5	15	5
		250	15	5	20	5	15	5
12.7	3.0	–50	10	5	15	5	10	5
		0	10	5	10	5	10	5
		50	10	5	10	5	10	5
		100	10	5	15	5	10	5
		150	15	5	15	5	10	5
		200	15	5	15	5	15	5
		250	15	5	20	5	15	5

Table 3.3 - Flat strips *continued*								
Width (mm)	**Thickness (mm)**	**Temperature (°C)**	**Time to 1 °C off equilibrium (min)**					
			Rubber		**Crystalline plastic**		**Amorphous plastic**	
12.7	2.0	–50	**10**	**5**	**10**	**5**	**10**	**5**
		0	5	5	10	5	5	5
		50	10	5	10	5	5	5
		100	10	5	10	5	10	5
		150	10	5	10	5	10	5
		200	10	5	15	5	10	5
		250	10	5	15	5	10	5
12.7	1.0	–50	5	5	5	5	5	5
		0	5	5	5	5	5	5
		50	5	5	5	5	5	5
		100	5	5	5	5	5	5
		150	5	5	10	5	5	5
		200	5	5	10	5	5	5
		250	5	5	10	5	5	5
6.35	12.7	–50	20	5	25	5	20	5
		0	15	5	20	5	15	5
		50	15	5	20	5	15	5
		100	20	5	25	5	20	5
		150	25	5	30	5	20	5
		200	25	5	30	5	20	5
		250	25	5	30	5	25	5
6.35	10.0	–50	20	5	25	5	15	5
		0	15	5	15	5	15	5
		50	15	5	20	5	15	5
		100	20	5	25	5	20	5
		150	20	5	25	5	20	5
		200	20	5	25	5	20	5
		250	25	5	30	5	20	5

Table 3.3 - Flat strips *continued*								
Width (mm)	**Thickness (mm)**	**Temperature (°C)**	**Time to 1 °C off equilibrium (min)**					
			Rubber		**Crystalline plastic**		**Amorphous plastic**	
6.35	6.5	–50	**15**	**5**	**20**	**5**	**15**	**5**
		0	10	5	15	5	10	5
		50	15	5	15	5	10	5
		100	15	5	20	5	15	5
		150	15	5	20	5	15	5
		200	20	5	25	5	15	5
		250	20	5	25	5	20	5
6.35	5.0	–50	15	5	15	5	15	5
		0	10	5	15	5	10	5
		50	10	5	15	5	10	5
		100	15	5	15	5	15	5
		150	15	5	20	5	15	5
		200	15	5	20	5	15	5
		250	15	5	20	5	15	5
6.35	3.0	–50	10	5	15	5	10	5
		0	10	5	10	5	10	5
		50	10	5	10	5	10	5
		100	10	5	15	5	10	5
		150	10	5	15	5	10	5
		200	10	5	15	5	10	5
		250	10	5	15	5	10	5
6.35	2.0	–50	10	5	10	5	10	5
		0	5	5	10	5	5	5
		50	5	5	10	5	5	5
		100	10	5	10	5	10	5
		150	10	5	10	5	10	5
		200	10	5	10	5	10	5
		250	10	5	10	5	10	5

Table 3.3 - Flat strips *continued*								
Width (mm)	**Thickness (mm)**	**Temperature (°C)**	**Time to 1 °C off equilibrium (min)**					
			Rubber		**Crystalline plastic**		**Amorphous plastic**	
6.35	1.52	–50	**5**	**5**	**10**	**5**	**5**	**5**
		0	5	5	5	5	5	5
		50	5	5	5	5	5	5
		100	5	5	10	5	5	5
		150	10	5	10	5	5	5
		200	10	5	10	5	10	5
		250	10	5	10	5	10	5
6.35	1.0	–50	5	5	5	5	5	5
		0	5	5	5	5	5	5
		50	5	5	5	5	5	5
		100	5	5	5	5	5	5
		150	5	5	5	5	5	5
		200	5	5	10	5	5	5
		250	5	5	10	5	5	5
4.0	12.7	–50	15	5	20	5	15	5
		0	10	5	15	5	10	5
		50	10	5	15	5	10	5
		100	15	5	20	5	15	5
		150	15	5	20	5	15	5
		200	15	5	20	5	15	5
		250	20	5	20	5	15	5
4.0	10.0	–50	15	5	15	5	15	5
		0	10	5	15	5	10	5
		50	10	5	15	5	10	5
		100	15	5	15	5	15	5
		150	15	5	20	5	15	5
		200	15	5	20	5	15	5
		250	15	5	20	5	15	5

Table 3.3 - Flat strips *continued*								
Width (mm)	**Thickness (mm)**	**Temperature (°C)**	**Time to 1 °C off equilibrium (min)**					
			Rubber		**Crystalline plastic**		**Amorphous plastic**	
4.0	6.5	–50	**10**	**5**	**15**	**5**	**10**	**5**
		0	10	5	10	5	10	5
		50	10	5	10	5	10	5
		100	10	5	15	5	10	5
		150	15	5	15	5	15	5
		200	15	5	20	5	15	5
		250	15	5	20	5	15	5
4.0	5.0	–50	10	5	15	5	10	5
		0	10	5	10	5	10	5
		50	10	5	10	5	10	5
		100	10	5	15	5	10	5
		150	10	5	15	5	10	5
		200	15	5	15	5	10	5
		250	15	5	15	5	10	5
4.0	3.0	–50	10	5	10	5	10	5
		0	5	5	10	5	5	5
		50	10	5	10	5	5	5
		100	10	5	10	5	10	5
		150	10	5	10	5	10	5
		200	10	5	15	5	10	5
		250	10	5	15	5	10	5
4.0	2.0	–50	5	5	10	5	5	5
		0	5	5	5	5	5	5
		50	5	5	10	5	5	5
		100	10	5	10	5	5	5
		150	10	5	10	5	10	5
		200	10	5	10	5	10	5
		250	10	5	10	5	10	5

Table 3.3 - Flat strips *continued*								
Width (mm)	**Thickness (mm)**	**Temperature (°C)**	**Time to 1 °C off equilibrium (min)**					
			Rubber		**Crystalline plastic**		**Amorphous plastic**	
4.0	1.0	–50	**5**	**5**	**5**	**5**	**5**	**5**
		0	*5*	*5*	*5*	*5*	*5*	*5*
		50	*5*	*5*	*5*	*5*	*5*	*5*
		100	*5*	*5*	*5*	*5*	*5*	*5*
		150	*5*	*5*	*5*	*5*	*5*	*5*
		200	*5*	*5*	*5*	*5*	*5*	*5*
		250	*5*	*5*	*5*	*5*	*5*	*5*

4 Mass, Density and Dimensions

4.1 Introduction

The obvious connection between mass, density and dimensions, is that density can be derived from a knowledge of dimensions and mass. However, they have been grouped together largely for convenience and also because they are measurements which are used as an essential part of other physical tests. For example density being used to calculate volume loss in an abrasion test, mass being an intrinsic factor in water absorption tests and there are very few tests which do not in some way involve the measurement of dimensions.

Mass, dimensions and density are also important factors in the costing of products. In quality control, checking dimensional accuracy of components is one of the most basic quality control procedures whilst mass is an essential consideration to control quantity of ingredients, etc. Density can also be a useful control measure to monitor variation in materials.

Another common feature of these measurements is that they are probably the most frequently used. Measurements that are made every day have a habit of being taken for granted and this can certainly happen to the measurement of dimensions, resulting in unnecessary errors. Taking the example of the determination of tensile strength, any error in the measurement of the cross section directly results in an equivalent percentage error in the strength measurement. Hence, it is sensible to devote considerable attention to the seemingly simple matter of measuring the width and thickness.

Mass and dimensions are well understood so there is no need for definitions to be given here. However, it should be noted that a mass is often used to produce a force in test methods and the term weight tends to be used indiscriminately. Using SI units, there should not be any cause for confusion.

Density is mass per unit volume (at a defined temperature). Relative density is mass (of a substance) compared with the mass of an equal volume of a reference substance (usually water) and being a ratio is dimensionless. Relative density is the property most often measured but in the usual units (mg/m^3) it is normally adequate to take the density of water as 1. Furthermore, the determination is often made by observation of gravitational

forces but for convenience the forces are expressed in mass units. Relative density used to be commonly known as specific gravity but this term is now deprecated and should not be used. Apparent density is the term used when the density of, for example, a powder is measured from mass and dimensions which includes the voids between particles.

4.2 Measurement of Mass

Mass is measured by weighing the test piece or object in question using an appropriate balance or scales. As the magnitude and the accuracy needed varies, the weighing instrument has to be selected accordingly. Accuracies required are often written in terms such as accurate to 1 mg whereas balances may be quoted as reading to 1 mg. The two are not the same and the standards are not always clear.

4.3 Measurement of Density

The classical ways of measuring density of solids involve either the displacement of a liquid or measuring mass and dimensions. The latter is only sensible on very uniform, regularly shaped objects and some form of displacement method is normally used. There are a number of possible variations from simply weighing in air and water to the use of a so-called density column.

Methods for plastics are given in ISO 1183 [1, 2]. There is an immersion method that suggests a test piece weighing between 1 and 5 g which can be of any shape as long as the surfaces are smooth and there are no crevices to trap air. The test piece is weighed in air and then in water using a balance accurate to 1 mg. The best way of suspending the test piece is by means of a very fine filament, the weight of which can be included in the zero adjustment of the balance and its volume in water can be ignored. However, if smaller than standard test pieces are used the effect of the filament could be significant. A top pan balance is not suitable. Removing air bubbles with a fine wire is mentioned but not the procedure of wetting the test piece with a liquid such as methylated spirit before weighing in water, which is likely to be more effective. The water then needs to be changed relatively frequently because of contamination by the alcohol.

The conditioning and temperature control required by the standard is very badly expressed. Conditioning is supposed to take place during the test but there is no mention of the tolerance on temperature required for the weighing in air, let alone instructions for achieving equilibrium. The weighing in a liquid is supposed to be carried out with a ± 0.1 °C tolerance on the temperature but it is not too clear how this is achieved. In theory the method is accurate to something like 0.2% (or better if buoyancy corrections

are made) but in practice the density is often quoted to 0.01 mg/m^3 for tests carried out in the usual laboratory atmosphere

If the plastic is less dense than water a less dense liquid of known density could be substituted but it is more usual to attach a sinker to the test piece. The sinker can conveniently be a small piece of lead, but using an item like a paper clip to suspend the test piece leads to complications as it will only be partly submerged. The weight of the sinker in water must be measured and it is a common error among new technicians to make this weighing in air.

A pycnometer (or density bottle) method is given for use with powders or when it is necessary to cut the sample into small pieces to avoid trapped air, as might happen with narrow bore tubing. The sample is weighed in a density bottle both with and without the remaining space filled with water. Before weighing the immersed test sample a vacuum is applied to remove trapped air. The bottle is also weighed without test sample both empty and filled with water. This is a more tedious procedure than the immersion method and is generally only used as a last resort.

A third method utilises two miscible liquids of different densities one having a lower density and one a higher density than the test material. The test piece is introduced into a glass cylinder containing a quantity of the first liquid and then the second liquid buretted in until (after stirring) the test piece 'floats' in equilibrium with the mixture. The density can be deduced from the relative quantities of the two liquids. A cruder version of this approach was previously specified which simply used a series of liquids and a pair of liquids found for which the test piece just sinks in one and just floats in the other.

The fourth ISO 1183 method uses a density column which works on the principle that two miscible liquids of different densities can be run into a container such that a uniform density gradient from the bottom to the top of the container results. A diagrammatic representation of the apparatus is given in **Figure 4.1**. The container is normally a glass tube of not less than 40 mm diameter in a thermostatted jacket. This column can then be calibrated by floats of known density which will come to rest at the depth in the column where their densities equals that of the immediate surrounding liquid. Small test pieces are then introduced into the column in the same manner and allowed to come to rest, their height in the column measured and their density deduced from a calibration graph. With care a column will last several months and the range of density in a single column would not normally be greater than 0.2 mg/m but could be as little as 0.02 mg/m. This means that very fine discrimination is possible but very close control of temperature is necessary. Ten minutes is suggested as the minimum time to allow test pieces to come to equilibrium but a large number of samples can be tested at one time and only a very small sample is required.

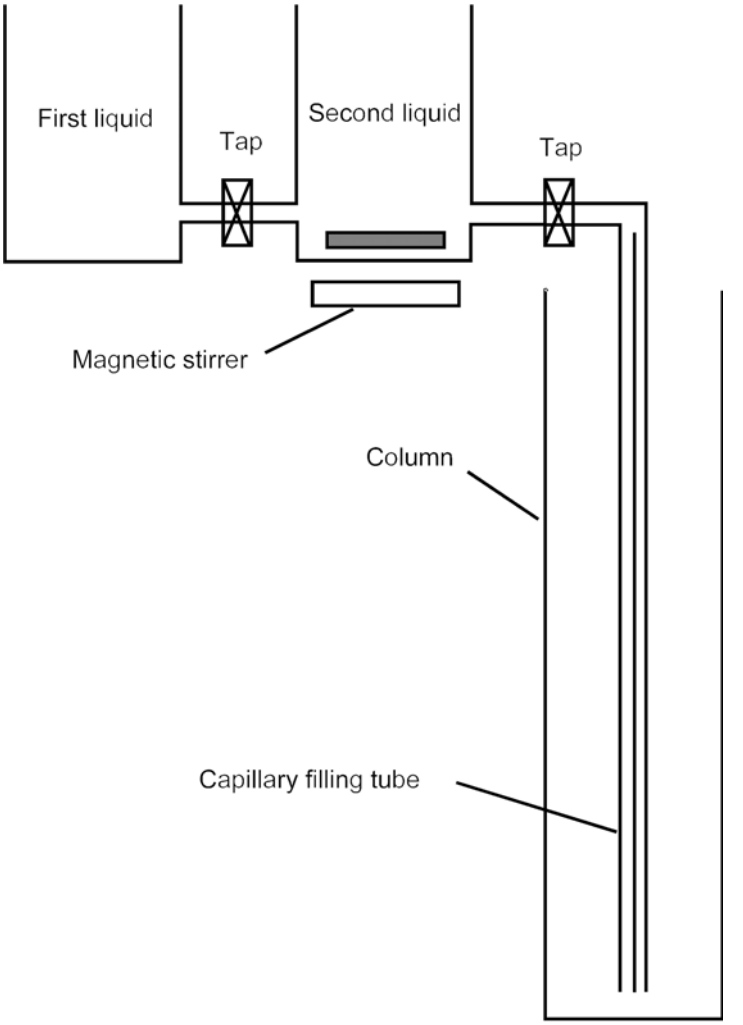

Figure 4.1 Preparation of a density column

A revision of this standard has been progressing very slowly. The existing drafts have split the methods so that the immersion, pycnometer and titration methods form Part 1 and the density column forms Part 2. Additionally a Part 3 was drafted covering an ultrasonic method and a Part 4 using a gas pycnometer. Apparently, the ultrasonic method has been dropped (but see ASTM below) so that the gas pycnometer method becomes Part 3 and has been published. In this procedure the volume change or pressure change on introduction of the test sample into the pycnometer is measured.

The British standards, BS 2782 Methods 620 A-D [3] are identical to ISO 1183. There has, however, been some confusion as 620 has been partially withdrawn due to presuming that the ISO revisions were about to be published. ASTM D792 [4] covers the displacement method and has two procedures, one for displacement of water and one for other liquids. It is not clear why it has been split in this way, particularly as method A is simple immersion and method B uses a pycnometer. The density gradient method is given in ASTM D1505 [5] and is very similar to the ISO procedure. There is also an ASTM method for density of polyethylene by means of ultrasound, ASTM D4883 [6]. This works on the principle of measuring sound velocity in the plastic which correlates with its density. The apparatus requires calibrating with reference materials but is claimed to give accuracies of 0.08% or better. The use of the method would mostly be in quality control and it is questionable whether it should have been standardised. It essentially describes the use of a commercial instrument with no apparatus details, not even the frequency used.

For routine quality control tests on rubbers, semi-automated forms of the immersion method are often used and referred to as specific gravity balances. These could be used for some plastics but generally it is more appropriate to use the density column procedure when a large number of measurements are needed.

Another approach to obtaining density is to measure the attenuation of gamma radiation as it passes through the material. If the thickness is kept constant then the attenuation is proportional to density [7].

For thin films it is often more convenient, and more useful, to measure mass per unit area rather than density. A test piece of given dimensions and uniform shape is simply cut and weighed.

The measurement of apparent or bulk density of powders is covered in ISO 60 [8] and ISO 61 [9]. The first is a procedure for powders which can be poured from a funnel. A funnel of the form shown in **Figure 4.2** is mounted vertically with its lower end 20-30 mm above the top of a measuring cylinder of 100 cm^3 capacity and internal diameter 40-50 mm. With the lower orifice closed, 110-120 cm^3 of well mixed powder is poured into the funnel and then the powder is allowed to flow into the measuring cylinder, assisted if necessary by being loosened with a rod. When the cylinder is full, a straight bladed knife is drawn across the top of the cylinder to remove excess and then the contents are weighed.

For materials that cannot be poured from a funnel a cylinder of 1000 cm^3 capacity and internal diameter 90 ± 2 mm is used. A plunger of slightly smaller diameter and total mass 2300 g fits into the cylinder. Sixty grams of the powder is dropped, little by little, into the cylinder so that it is evenly distributed and has a level surface. The plunger is lowered onto the powder and rest there for 1 minute before the height of the powder is measured. From the height of powder, the diameter of the cylinder and the weight of the powder, the apparent density can be calculated.

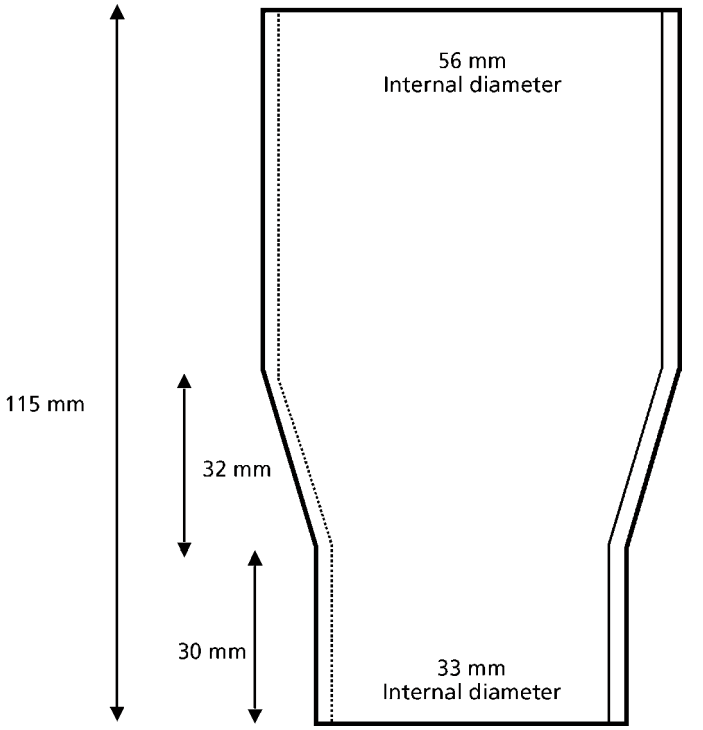

Figure 4.2 Form of funnel for apparent density

The British methods, BS 2782 Methods 621 A and B [10, 11] are identical to ISO 60 and 61. ASTM D1895 [12] has three procedures. Method A is similar to ISO 60 but the funnel geometry is not identical and slightly different results can be expected. An empirical relationship between method A and ISO 60 is given in an appendix but appears to have been derived from a very early version of the ISO standard. Method B uses the same principle but with a larger funnel. Method C is very similar to ISO 61.

ISO 1068 [13] measures the compacted bulk density of PVC resins with a cylinder method which uses a shaker to tamp down the material under a piston. BS 2782 Method 621D [14] is identical.

The bulk factor of a moulding is defined as the ratio of the volume of a given mass of moulding material to its volume in moulded form - the ratio of the density of the moulding to its apparent density before moulding. ISO 171 [15], BS 2782 Method 621 C [16] and the procedure in ASTM D1895 [12] all require determination of apparent powder density and moulded density by the appropriate methods discussed previously.

4.4 Measurement of Dimensions

4.4.1 General

There is hardly a test that does not involve the measurement of the dimensions of a test piece and in most cases the measurement of at least one dimension is critical to the final result. In one of the most common cases, the measurements of thickness and width of a tensile dumbbell is used directly in the calculation of stress. Any error or uncertainty in these measurements is translated directly into an error or uncertainty in the test result.

Probably because most dimensional measurements seem easy, they tend to be taken for granted and perhaps too little attention paid to their accuracy and significance. For many years, measurement of dimensions in standards was dealt with in each individual test method which led to a considerable degree of inconsistency in apparatus and procedure specified. A general ISO standard for dimensions of rubber test pieces was first produced in 1978 but work has only started very recently on a method for plastics.

Dimensions of test pieces can conveniently be classified as large and small where large means that a tape or rule is a suitable measuring device and small is in the realm of dial gauges, micrometers and callipers. Distinction can also be made between contact methods (such as a dial gauge) and non-contact methods (such as a travelling microscope). For test piece measurement, contact methods are most common but non-contact methods are specified for particular circumstances. Additionally, there are the fields of thermal expansion, extensometry, dispersion and surface roughness plus non-linear measures such as angle.

4.4.2 'Standard' Laboratory Procedures

Methods for rubbers are specified in ISO 4648 [17] which is worth considering here because it is very applicable to soft plastics and also illustrates some of the factors to be considered. The standard has four procedures: dimensions less than 30 mm, 30-100 mm, over 100 mm and non-contact. Over 100 mm a tape or rule is used with an error of not more than 1 mm. For 30-100 mm, callipers are specified with an error of not more than 1% and to be applied such that no straining of the test piece takes place. For under 30 mm a dial gauge or equivalent is used with an error of not more than 1% or 0.01 mm whichever is the smallest. The non-contact procedure, using a travelling microscope, projection microscope, etc., is intended for special shapes such as O-rings.

The use of a tape for larger dimensions is straightforward in principle. Callipers for a soft deformable material is not ideal and requires considerable care. Non-contact methods

would be preferable and should yield identical results but may not always be justified on grounds of time and cost. Callipers are not suitable for thin sections which is one reason why the width of dumbbells is taken from the die dimension.

The use of a dial gauge (or equivalent) on soft materials requires consideration of the foot pressure and in ISO 4648 [17] the standard procedure is to apply 22 ± 5 kPa through a circular foot which is smaller than the test piece. For very soft materials below 35 IHRD 10 ± 2 kPa is used. These pressures are somewhat arbitrary but ensure consistency and are a compromise between sensibly defining the surface level and producing excessive strain. Quite clearly, the results will not be identical with non-contact methods.

With rigid plastics the problem is different in that the pressure on a dial gauge is of relatively little consequence and callipers can be used with no fear of straining the test piece. However, errors arise if the test piece or the lower surface is not perfectly flat and a dial gauge is used, because the force may be insufficient to close any gap. Consequently, it is preferable to use a micrometer for measurements of small dimensions of rigid materials. Measurement by dial gauge of a) a soft material and b) a rigid material illustrating possible deviations from the true thickness is shown in **Figure 4.3**.

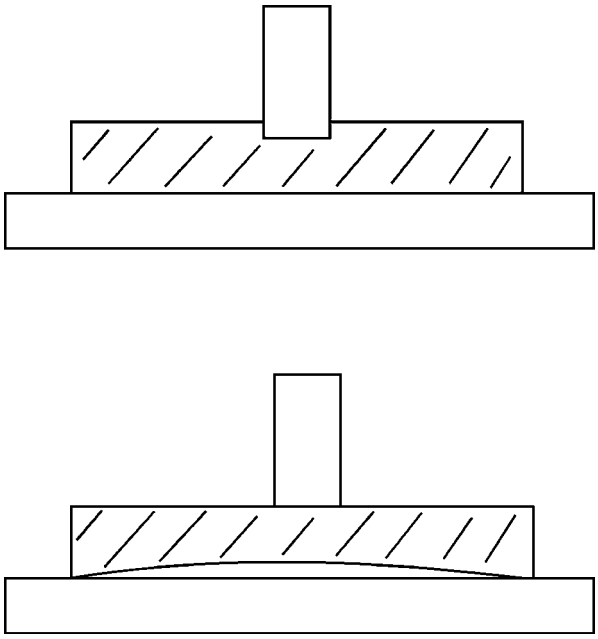

Figure 4.3 Possible errors in thickness measurement

This problem can be overcome by the use of a pair of domed contact members instead of flat surfaces. This used to be specified for measuring rubber compression set buttons which tended to be concave. The set measurement is now made with lubricated buttons which avoids concavity but in any instance where it could occur domed feet would be preferable.

The first ISO draft for a plastics standard gave an ASTM method [18] which is ludicrously detailed and restrictive in its description of micrometers and dial gauges. The draft which appeared later is much improved but is riddled with errors and is not very comprehensive. It ignores larger dimensions and dismisses non-contact devices as being required only to give the same results as contact methods. Micrometers and sliding (meaning vernier) callipers are specified with accuracies of 0.05 mm for dimensions below 10 mm and 0.1 for dimensions above 10 mm. Curiously, micrometers have a foot of 6.4 mm and a force of 5-15 N specified but callipers do not. What happens if a test piece is less than 6.4 mm wide is not mentioned. This draft was clearly thrown together in a hurry and hopefully it will be improved including recognition of softer materials before progressing. The authors could have benefited from looking at the rubber standard.

There are standards for dimensions of thin films, which are concerned with the product rather than for measuring test pieces. ISO 4591 [19] deals with gravimetric thickness, ISO 4592 [20] with length and width and ISO 4593 [21] with thickness by mechanical scanning. The British standards [22-24] are identical.

The gravimetric thickness of plastics film is often used, which is obtained from measurement of lateral dimensions and density. This obviates problems of the sensitivity of instruments needed for direct measurement of thickness. In ISO 4591 a square or circle of material of area 100 cm^2 is cut from the film or sheet and weighed and the density determined so that the thickness can be calculated. In ISO 4582 procedures are given for measuring the length of a roll to 0.1 m and the width to 1 mm for widths over 100 mm or to 0.1 mm for widths less than 100 mm. The length measurement is made by laying the material on a flat surface 10 m long marked in metres and in 0.1 m for the last metre. The width is measured with a graduated scale and for narrow widths the somewhat dubious process of using a magnifying glass and graticule to estimate tenths of a millimetre is used.

For direct measurement of thickness a gauge with an accuracy of 1 μm is needed for the thinnest film rising to 3 μm for film over 250 μm. Clearly, an ordinary dial gauge is not good enough. The foot force specified in ISO 4593 is only 0.5-1 N but thin film can be expected to lie flat.

Whilst reference has been made to traditional dial gauges and callipers, in principle any form of electrical or other transducer could be used as long as the appropriate foot loading can be achieved.

For dimensions of plastic (and rubber) test pieces there has been considerable debate as to how many readings should be taken and what form of average should be used. The general standards are intended to specify the instruments and procedures to be used and the test method standards should specify what and where measurements are to be taken. Nevertheless, the draft ISO standard for plastics specifies a minimum of three readings and takes the arithmetic mean. ISO 4648 [17] again specifies at least three readings but takes the median. It can be argued that for strength measurement the minimum width and thickness is more appropriate. It should also be noted that plastics test method standards will continue to have their own procedure until a general method has been published long enough for revisions of test method standards to refer to it. Even in rubber methods there is not yet complete consistency.

No attempt will be made here to consider all the separate measurement clauses to be found in current test method standards. Until the ISO standards for the measurement of dimensions has become established long enough for all test methods to have been revised and reference it, each test method will have its own procedure and there will not be universal agreement on detail. The essentials are to distinguish between a non-contact measurement and one applying a specified pressure, in the latter case to use the correct standard pressure, and to measure within the accuracy limits specified.

The accuracy of measuring instruments is specified in many cases as 1% which presumably means ± 1%. If this was literally the case then a measurement of area could potentially have an error of about ± 2%, taking account of length and width. The uncertainty of the measuring instrument is not the complete story as there are also uncertainties associated with the operator and conditions. The point is that dimensional measurements can contribute significantly to uncertainty of a measurement and this is often overlooked.

Contact methods are used for most test piece dimension measurements because dial gauges and digital micrometers, etc., are both relatively inexpensive and fairly quick and easy to operate. There are, however, some dimensions which really require a non-contact method. These include the diameter of flexible O-rings, thickness of coatings, dimensional stability, dumbbell cutter profiles, impact notches and tear nicks.

A travelling microscope is fine in principle but tedious to operate. For measurement of changes of lateral dimensions between marked points on a surface in dimensional stability tests it can be the only viable method. The accuracy is such that the thickness of marked lines is the limiting factor

A projection microscope is a very useful instrument and can be used to measure the diameter of O-rings and other sections. It is also widely used to check dumbbell cutter profiles, impact notch profiles and the length of nicks in tear test specimens. The usual

procedure is to draw the profile, complete with tolerances at the given magnification on transparent film. Even for relatively large dimensions it is sometimes possible to use a projection microscope with a suitable jig to measure changes in dimension.

4.4.3 Other Procedures

There are inevitably a number of special circumstances connected with plastics where an unusual type of dimensional measurement such as the thickness of very thin coatings and crack length in fracture or fatigue tests. Very often a great deal of dimensional information can be found by means of microscopy – which is a subject in its own right and can only be touched on here. For example, one would expect to use a microscope to study the geometry of fibres or thin film, and much failure analysis involves detailed optical examination. A microscope fitted with a graticule is used to measure thickness of coatings or multi-layer films after cutting a section. Very thin coatings can be potted in epoxy resin and microtomed.

Crack growth can be monitored with a microscope but is not particularly convenient. Ink can be injected to aid identifying the position of the crack front. Various other approaches have been used, in particular measurement of change in electrical resistance [25-28] and high speed photography [29-30]. In some tests the crack opening displacement can be measured with a displacement transducer [30] or strain gauges can be used [30].

Video extensometers cannot be readily used for absolute dimensions but are excellent for measuring change. Hence, a bonus if you have one for tensile measurements, is their considerable potential for such things as thermal expansion and Poisson's ratio.

The measurement of moulded in strains is by no means restricted to dimensional methods, and solvent cracking and optical techniques are probably the most widely used methods. Classical dimensional methods are generally based on measuring the deflections that occur when the stress state is modified. The best known procedure is layer removal [31] and with pipes stress is relaxed by cutting sections [32]. It would be advantageous to measure relaxation with strain gauges but there are considerable difficulties in adhering gauges to plastics. Newer adhesives and strain gauges have now made this viable [33]. Deliberately introduced surface strains have often been measured by observing the distortion of a grid printed onto the part or test piece. This procedure can be automated by using video camera and image processing [34]. Strains can also be measured by attaching a grating and observing Moire fringes [35].

The various on-line methods to monitor production are outside the scope of this chapter, although inspection is a form of testing. In this context, dimensional measurements are

those most often made and, apart from gauges, micrometers and so on, there are various optical, electrical, nuclear and other methods which may have advantages in continuous production circumstances. Descriptions of the use of such techniques are extensively covered in journals and in manufacturer's literature.

4.4.4 Surface Roughness

It is not often necessary to measure the surface roughness of plastics test pieces or products and no standard methods exist. Where surface roughness measurements are needed, methods established for metals are generally used, most commonly mechanical profiling.

One area where the surface finish is of great importance is in optical measurements since light transmission and reflectance characteristics are very dependent on it. However, properties such as gloss and haze are measured rather than the surface roughness. Another area is friction where the roughness of a surface may be measured to aid in the interpretation of the friction results. The surface finish of metals is sometimes of importance to plastics testing, for example on mould surfaces and on the bore of melt flow dies.

ISO standards for surface finish include ISO 4287 [36]. The main British standard is BS 1134 [37, 38]. ISO 4287 is a terminology standard which used to be reproduced as BS 6741. Definitions are given in BS 1134 although ISO 4287 is referenced as giving further definitions. BS 1134 also takes into account the older ISO 468, which has now been withdrawn. There are other ISO standards in the Geometrical Product Specification series. BS1134 is divided into two parts, the first concerning the method and instrumentation; the second forms a general explanation.

Surface texture can be considered as having three components, roughness, waviness and form, the essential distinction being the spacing of the texture. Roughness refers to closely spaced texture, waviness to wider spaced texture and form is the underlying shape. Clearly, a surface can have roughness superimposed on waviness which is superimposed on the form. A particularly good introductory guide to surface texture is written by Dagnall [39].

There are apparently over 1000 different parameters used to characterise surface finish [40] but mercifully only a few are very widely encountered. The parameter most often used to grade the roughness of a surface is Ra (once known as CLA), the mean deviation of the surface profile above and below the centre line. Rz, which is a measure of the peak to valley height, is the next most widely found. Details of many others can be found in the standards.

Parameters such as Wa refer to waviness. Long-term waviness can be important from an appearance point of view in plastics mouldings, for example components for cars [41].

4.4.5 Extensometry

The measurement of extension (or other mode of deformation) is an essential part of most mechanical property tests. The magnitude of deformation and the precision required depends on the mode of stressing, the material and parameter being measured. Consequently, somewhat different apparatus is required for tensile and compression tests or for plasticised poly(vinyl chloride) (PVC) and a fibre reinforced composite. In some cases the apparatus is essentially a general purpose measuring instrument or transducer, in others a purpose designed extensometer may be necessary. The apparatus requirements, including range and precision, are usually specified in the test methods standard and will be discussed in the relevant sections in later chapters.

4.4.6 Dimensional Stability

A broad definition of dimensional stability would include thermal expansion, shrinkage, softening point and the effect of liquids, which is a mix of thermal properties and environmental effects. All of these are more properly dealt with in depth in other books in this series. For example, volume swelling under effect of liquids and softening point and thermal expansion under effect of temperature.

For change in volume (swelling), or any other physical property, standard methods for exposure to liquids would generally be used, such as ISO 175 [42]. Standard methods for water absorption are generally concerned with mass uptake rather than effect on dimensions. However, the appropriate dimensions of a suitable sized test piece can be measured before and after any exposure treatment by any of the methods mentioned in this chapter. This includes in particular non-uniform dimensional change such as warping of a sheet. Some product tests have been standardised to measure such change particularly where the construction is not homogeneous or there can be one-sided exposure to the environment.

There are no international standard methods for thermal expansion of plastics although an ASTM method exists [43] based on a using a dial gauge to detect change in length of a relatively thick bar. Linear expansion is now usually measured with thermomechanical analysers which are capable of detecting very small changes. A method is being standardised in ISO but is taking a long time to reach publication. The classical method for volume expansion is to use a mercury in glass dilatometer but the procedure is both tedious and difficult.

Although mould shrinkage of plastics, i.e., the reduction in size of cooled moulded articles compared to the mould dimensions, is principally a matter of thermal expansion, it is usual to make a direct measure of shrinkage by measuring a ‘standard’ moulded test bar.

The result is obviously dependent on the processing conditions and most measurements are made to in-house procedures but ISO 2577 [44] exists for thermosetting materials and ISO 294 Part 4 [45] for thermoplastics. Adverse comments on the latter standard at draft stages were that it is essentially single point and shrinkage data is really needed over a range of processing conditions.

Shrinkage of plastics due to relaxation of stresses through heating is what many people think of as dimensional stability. A general method, ISO 8328, was withdrawn in 1996 but there are several others for particular polymers or products, for example ISO 11501 [46] for film or sheet and ISO 3521 [47] for polyester or epoxide casting resins. Post moulding shrinkage is also covered in ISO 2577 and ISO 294-4. The same principle as in liquid exposure applies in respect of none uniform shrinkage.

4.4.7 Dispersion

The dispersion of compounding ingredients can have a large effect on appearance or physical properties and it is often necessary to check on the efficiency of mixing. In plastics processing, trials may be made on laboratory scale processing equipment and there are various non-standardised procedures in use. One approach is to extrude a quantity of material through a screen pack and after removal to count the number of carbon black agglomerates on the screens under a microscope. Alternatively, the increase in pressure due to the build up of agglomerates clogging the screens can be measured. Poor dispersion will be seen by surface defects on the extrusion.

The direct estimation of degree of dispersion is effectively a dimensional measurement using microscopy techniques and is just one example of the value of microscopy for fault diagnosis in polymer products. Carbon black dispersion in rubbers is particularly critical and many methods can be traced to the work of Medalia and Walker [48]. Methods have long been standardised in ASTM D2663 [49] where a torn, cut or microtomed surface is examined and either compared to a set of standard photographs or the number of agglomerates are counted. Examination of a torn or cut surface is relatively quick and convenient for quality control, whereas counting agglomerates is clearly very time consuming and also requires microtomed sections which can be viewed by transmitted light. The ISO methods for rubbers [50] which compare to standard photographs are essentially similar but introduce the use of a split field microscope to simultaneously view the sample and the standards. This is shown schematically in **Figure 4.4**. The standards can be transparencies or images in computer memory. It is proposed to extend this standard to include higher magnifications and an agglomerate counting method using image processing software. The split field technique was suggested by Persson [51], and Richmond [52] describes a computer imaging method.

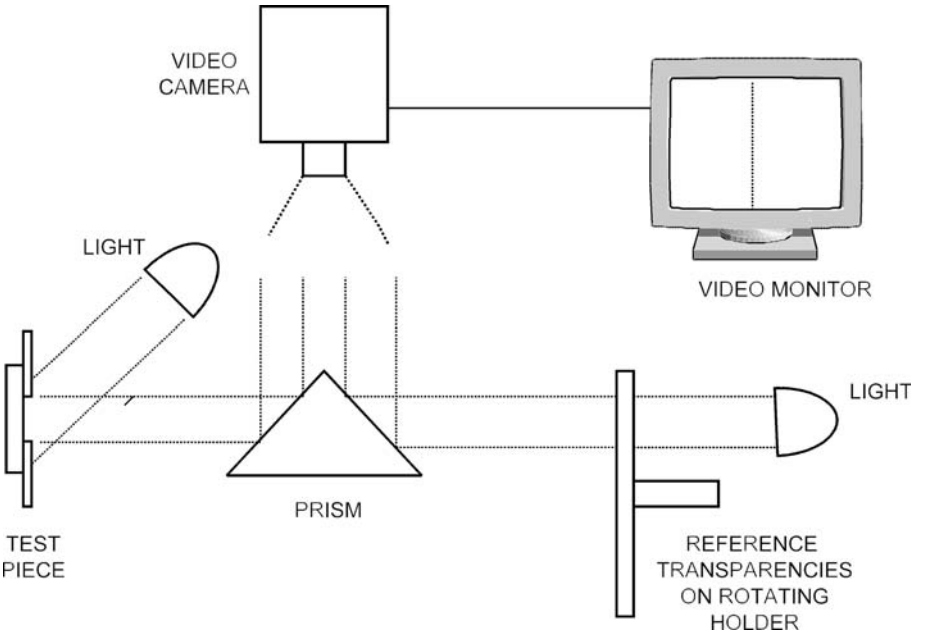

Figure 4.4 Split field dispersion method

Standardised methods for plastics are variations on the rubber procedures which generally use transmitted light and comparisons are made with standard photographs. The use of split field and image analysis techniques have not yet been introduced. With thermoplastics, test pieces can be prepared either by microtoming or by pressing a sample on a hotplate, although the standards are not consistent in allowing both. ISO 13949 [53] and ISO 11420 [54] are for dispersion of pigments and carbon black, respectively, in polyolefin pipes and fittings. There are British methods for polyethylene materials generally [55] and an ASTM standard which widens the scope to plastics [56] was recently discontinued.

Other approaches to dispersion measurement include electrical resistivity which is sensitive to carbon black dispersion and measuring surface roughness. A roughness method is included in ASTM D2663 but these techniques do not seem to have been applied to plastics. Cembrola [57] has compared microscope, stylus and resistivity methods and concludes that no one method is universally the best. A very comprehensive review of characterising dispersions from every aspect has been given by Hess [58].

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5 Hardness

5.1 Introduction

The term hardness is usually taken to mean a measure of modulus derived from the resistance of the material to indentation but has also been applied to scratch resistance and resilience. The mode of deformation under an indentor is a mixture of tension, shear and compression, and hardness is by no means a fundamental property. The result depends on the indentor geometry and degree of indentation as well as the time of indentation after which the measurement is made.

Hardness tests are attractive because of their apparent simplicity and in various forms methods have been devised for most types of material. Although hardness is almost inevitably included in properties of rubbers and very commonly applied to metals, it has been used rather less often for plastics. This is probably due to it not having been seen to have the same significance for characterising plastics. However, more recently microhardness measurements have increasingly been found useful to monitor changes due to environmental influences.

The indenting force can be applied in three ways:

a) application of a constant force, the resultant indentation being measured

b) measurement of the force required to produce a constant indentation, and

c) use of a spring resulting in variation of the indenting force with depth of indentation.

When standard methods were formulated, the measurement of force would have been much more of a complication than it is with modern force transducers, and consequently approach b) has not been seriously adopted. Bench instruments generally use approach a) but portable instruments, usually called durometers, always use a spring loading system. Because springs are not considered precision measuring elements, and because the force varies with indentation, standard reference methods use weights to apply a constant force.

A variety of indentor geometries are used, notably a ball, truncated cone and pyramid. The pyramid shape is derived from methods developed for metals and is commonly applied to rigid plastics, whereas a ball is favoured for rubbers and can be applied to relatively soft materials. The most widely used form of durometer uses a truncated cone.

In metals testing, the measurement of the indentation is normally made after removal of the indenting force, whilst in rubbers it is always made with the force applied. The difference is a consequence of the different levels of force involved and, particularly, because with metals the deformation is permanent, whereas with rubbers it is almost totally elastic. Plastics are somewhat between the two and both approaches are used.

The normal tests use indentors with dimensions of the order of millimetres but there are also microtests which are scaled down by approximately an order of magnitude which allow thinner test pieces to be used and, on rigid materials, produce less damage. With rubbers, hardness test are essentially non-destructive.

If test pieces are too thin in relation to the indentor and load used, the base material has an effect. Different results may also be obtained on curved surfaces. Measurements made on non-standard test pieces are sometimes referred to as apparent hardness.

5.2 Relationships

Despite the complexity of the deformation, approximate relationships between hardness and modulus have been derived in some cases. By far the most effort in this direction has been for rubbers [1] but plastics have been considered by, for example, Jirous [2] for a spherical indentor and by Rikards and co-workers [3] and Gubicza and co-workers [4] using the Vickers pyramid. Amitay-Sadovsky and Wagner [5] evaluated the measurement of Young's modulus from Knoop microtests.

Results are time dependent, subject to the non-linear response of strain to stress and a function of force and indentor geometry. The latter effects have been investigated by Crawford and Stephens [6]. Time and load dependence for the Vickers test was observed by Suwanprateeb [7] who found large differences in results calculated from diagonal length and indentation depth.

The total number of hardness methods which are used is considerable and a frequently asked question is how the different scales are related. Because of the dependencies mentioned previously, it follows that hardness values according to one method cannot generally be compared with those derived from another. However, a number of conversions have been established which, because of the arbitrary nature of the methods, are at best approximate. Computer software has been produced which includes all those that have been published [8].

Work by Fett and co-workers [9] and by Bowman and Bevis [10] has sought to relate hardness measurements to orientation conditions in a variety of thermoplastics, while Martinez-Salazar and co-workers [11, 12] have attempted to relate hardness to structure

and morphology in polyethylene and Balta-Calleja [13] to crystalline polymers generally. Selden and Gustafson [14] have attempted to correlate hardness and tensile properties for a number of materials. A comprehensive review of microhardness tests has been given by Lopez [15].

5.3 Standard Methods

5.3.1 Shore Durometer

The most popular scale is probably the Shore durometer hardness in its two main variants, Shore A and Shore D. These durometers are small, hand-held instruments with indentors of given geometry that are pressed into the surface of the material to be measured under a spring of given stiffness. Although originally designed to be hand held, they are often used on a stand which can improve reproducibility. The amount of penetration of the indentor is measured by a suitable scale marked directly in hardness degrees. Traditionally the scale was a dial gauge but modern instruments can have digital read out.

Both the common Shore variants are standardised in ISO 868 [16]. For soft plastics the Shore A scale is used (see **Figure 5.1a**). In this method, the indentor consists of a truncated

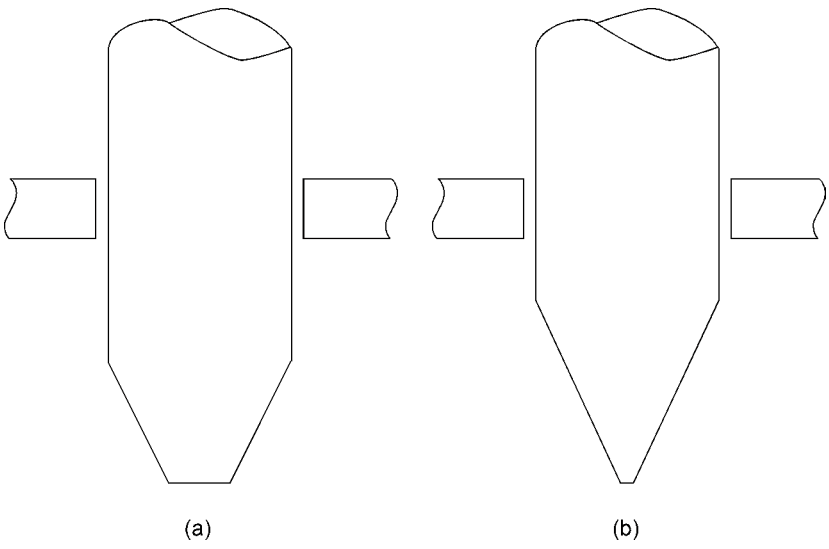

Figure 5.1 Shore indentors

cone of included angle 35° and diameter at the flat of 0.79 mm, operating under a spring pressure given by:

$$F = 550 + 75\ H_a$$

Where F is the applied force in mN and H_a is the hardness.

The Shore D scale (**Figure 5.1b**) is suitable for typical harder plastics materials. This has a sharper indentor of included angle 30° with only a slightly rounded (0.1 mm radius) tip and operates under a spring given by:

$$F = 445\ H_d$$

Both Shore scales are also specified for rubbers (although Shore D is not often applicable). The Shore A scale is essentially equivalent to the IRHD scale specified in ISO 48 [17] over the normal operating range.

For both durometers the test pieces must be at least 6 mm thick and measurements are made at least 12 mm from any edge.

The test is also standardised as BS EN ISO 868 [16] (identical to the ISO method) and ASTM D2240 [18] on which the ISO method was originally based. ASTM D2240 was originally for both rubbers and plastics but is now designated for rubbers only.

An important factor is the time of application of the load before a reading is taken because the measured hardness decreases with time of application. Unfortunately, there is not universal agreement over the time to be used. Currently, the ISO (and hence BS) standard sets this time at 15 seconds, although an 'instantaneous' reading may be estimated by making the reading after nominally a 1 second application of load. In the ASTM standard, the preferred time is 1 second but others may be used by agreement. In rubber testing an instantaneous reading is most common but some workers have used 30 seconds.

Durometers are best calibrated by measuring the spring force at various deflections and a crude method is given in ISO 868. Specific calibration methods are currently being developed for both plastics and rubbers, logically only one calibration standard is needed to cover both materials. Particularly for the A scale used for rubbers, standard blocks are available which are extremely useful for checks between formal calibrations.

5.3.2 Ball Indentation

A ball indentation test specifically for plastics is given in ISO 2039-1 [19]. The test piece is recommended to be 4 mm thick and the only limitation is that the underside of the test piece should show no signs of deformation after testing. A 5 mm diameter

hardened steel ball is pressed into the test surface under a specified load. An initial load of 9.8 N is applied and the indentation indicating device is set to zero. The major load is then applied, chosen from a given list such that the resulting indentation is between 0.15 and 0.35 mm. The time of application of the load is 30 seconds.

Although depth of indentation is measured, unlike the Shore or IRHD scales where hardness is directly related to the penetration of the indentor, the ball indentation hardness is related to the area of the impression and given in N mm^{-2}. First, a correction for deformation of the frame of the apparatus is made by a somewhat complicated procedure which incorporates empirical constants to give what are called reduced test load and reduced depth of impression. These figures are used to calculate the hardness.

Ten valid tests are required (with the indentation in the specified range) but one has to assume that they are averaged as this instruction is not given.

This test is apparently frequently used in continental Europe but is rarely found in the UK or USA. Nevertheless, it has been standardised as BS EN ISO 2039-1 [19].

At the time of writing a revision of ISO 2039 is being considered which proposes a new formula for expression of results which reduces the differences between results from measurements with different loads.

5.3.3 Rockwell

The Rockwell hardness test given in ISO 2039-2 [20] also uses a hardened steel ball as the indentor but 4 diameters are specified corresponding to 4 Rockwell scales, R, L, M and E. The minor and major loads also depend on the scale being used.

The standard test piece is a minimum of 6 mm thick for all scales.

Table 5.1 Rockwell hardness scales

Rockwell scale	Minor load (N)	Major load (N)	Indentor diameter (mm)
R	98.07	588.4	12.7 ± 0.015
L	98.07	588.4	6.35 ± 0.015
M	98.07	980.7	6.35 ± 0.015
E	98.07	980.7	3.175 ± 0.015

The minor load is applied and the indentation gauge zeroed. Within 10 seconds the major load is applied and removed after 15 seconds. After a further 15 seconds the indentation is measured. Hence, there is a fundamental difference in that the indentation is measured after removal of the load, unlike the other procedures above. The Rockwell hardness number is given by the rather curious expression 130 – e where e is the depth of impression after removal of the major load in units of 0.002 mm. The scale should be chosen such that the hardness is between 50 and 115. Five readings are taken and averaged.

The Rockwell alpha method is given as an annex to the standard. In this procedure the indentation is measured with the load applied and a similar correction to that used in the ball indentation method is applied (the error due to machine stiffness will increase with load). For reasons not explained, only the R scale is considered suitable. The Rockwell alpha hardness number is in this case given by 150 – d where d is the corrected indentation (not 130).

Fett [21] has shown that ball indentation hardness, H, and Rockwell alpha, R_α, are correlated through the expression:

$$H = \frac{(448.6)^{1.23}}{(150 - R_\alpha)}$$

The Rockwell test is also standardised as BS EN ISO 2039-2 [20] and in ASTM D785 [22]. The ASTM standard is said to be technically equivalent but it also includes the K scale with an even larger major load. Also, the Rockwell alpha method is included in the body of the standard as Method B.

Calibration of Rockwell hardness machines is normally by the use of standard blocks.

5.3.4 Softness

For flexible plastics there is a softness measurement standardised in BS 2782 Method 365A [23]. This is essentially the same as the hardness test for rubbers [17] but the expression of results is very different. Instead of converting the indentation to hardness degrees the softness number is recorded as the indentation in units of 0.01 mm. The test piece is required to be between 8 and 10 mm thick for the standard test to apply and no measurement should be made nearer than 10 mm from any edge. Non-standard dimensions can be used but these must be stated along with the softness number obtained. The standard contains special notes concerning plasticised polyvinyl chloride (PVC) which is known to vary in hardness with time after moulding and for results to depend on whether compression or injection moulded test pieces were used. To minimise the time effect the softness measurement must be made 7 ± 0.2 days after moulding.

5.3.5 Barcol Hardness

The Barcol impressor is a portable hardness meter or durometer. The method standardised in EN 59 [24] is specifically for glass reinforced plastics and uses the Barcol model 934-1. The indentor is a hardened steel truncated cone having an angle of 26 degrees and a flat tip of 0.137 mm. It is pressed onto the test piece under an unspecified spring pressure and the maximum indentation measured in units of 0.0076 mm.

BS 2782 Method 1001 [25] is identical and a Barcol is also specified in ASTM D2583 [26] where it covers rigid plastics generally.

5.4 Other Methods

It is somewhat curious that there are probably more references to the use of Vickers hardness on plastics than any other method bar Shore but it has not been standardised at international level for these materials. The Vickers method uses a right diamond pyramid on a square base as the indentor and the mean diagonal of the impression is measured, the hardness relationship being given by the expression:

$$HV = \frac{2F\sin(\theta/2)}{d^2}$$

where HV = Vickers hardness
F = applied load (kg)
d = mean diagonal width of the impression (mm)
θ = apex angle of the pyramid (= 136°)

The Vickers test is standardised for use with metals in ISO 6507 [27]. In a micro Vickers test, the same indentor is used but the loads are in the range up to 200 g.

The pyramid indentor usually gives a sharp impression in plastics which makes the diagonal measurement reasonably easy but the measurement is made after elastic recovery. Crawford [28] used a Wallace microhardness tester (adaptation of the rubber microhardness tester) with the standard Vickers pyramid which could measure the depth of indentation under load. He noted that the elastic recovery is greater in depth of indentation than it is for the diagonals and also that the hardness could depend on the level of load and the time of application.

The Brinell hardness method uses a ball indentor but, unlike Rockwell, measures the diameter of the impression. This is generally not very satisfactory with plastics and it is

far less popular that Vickers or Rockwell. The Brinell method is standardised for use with metals in ISO 6506 [29].

The hardness relationship being given by the expression:

$$HB = \frac{2F}{\pi D^2 \left[1 - \left(1 - \{ d/D \}^2 \right)^{1/2} \right]}$$

where HB = Brinell hardness
F = applied load (kg)
D = diameter of the indentor (mm)
d = diameter of the impression made (mm)

Crawford [6] demonstrated that small diameter ball indentors could be used in a sort of micro Brinell test.

There is little doubt that microhardness tests, particularly the Vickers method, have been successfully and usefully applied to plastics and are probably more effective that ball indentation or Rockwell. Hence, it is particularly surprising that no standard has been developed and that there has not been greater consideration of indentation being measured under load.

A considerable number of other hardness tests have been applied to plastics but have not been widely adopted.

The Knoop micro-hardness test is similar to the Vickers test but with the diamond indentor having rhombic cross section with diagonal lengths in the ratio 7:1. This gives rise to smaller indentations, making the shattering of brittle materials less likely, and also makes it possible for anisotropy to be detected. The Knoop method is standardised for metals in ISO 4545 [30].

The TNO test uses a polished sapphire pyramid as the indentor and apparently measures indentation in a manner similar to the Wallace tester.

Benabdallah and Chalifoux [31] constructed what might be termed a universal hardness tester which consisted of an indentation jig that fitted into an electro-hydraulic universal testing machine. They used a variety of indentors and investigated loading, creep and recovery curves, demonstrating the scope of information that can be obtained from an indentation test.

A universal method for metals is given in a ISO Technical Report [32]. A revision of this is in progress which deals with very tiny indentations and produces loading and unloading curves with indentation being measured under load. Such a microscale may not be so important for plastics but an instrumented approach would seem to have many benefits.

Tests like Moh's scale (using natural minerals) and pencil hardness (Kohinoor test) rate materials according to their scratch resistance, the harder the material the harder the mineral or pencil which will not scratch it. Although these sound crude, the pencil test can be useful in connection with, for example, floor coverings. Clearly, there is a qualitative relationship between the scratch resistance of a material and its hardness in the more conventional sense, but correlations between the two cannot be expected to be very high. Various mar and scratch resistance tests could be said to be a similar approach. A whole range of these together with conventional hardness tests was applied to plastics by Boor and co-workers [33].

The schleroscope in which a hemispherical headed striker falls onto the test piece under gravity is sometimes described as measuring hardness, when in fact it measures resilience.

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6 Tensile Stress-Strain

6.1 General Considerations

The short-term tensile characteristics of a material are probably the most commonly derived of all the properties that can be determined. Although there are many standards relating to short-term tensile testing (the most significant of these will be discussed later), they all endeavour to quantify a number of specific characteristics which relate to the strength and deformation of a material. Knowledge of these characteristics can give materials scientists, technologists, materials specifiers and designers an insight into the potential performance of a material.

Tensile stress-strain characteristics are derived by monitoring both the force required to pull a material apart and the displacement that the material undergoes as a result of the applied force at a constant deformation rate. In order to convert the force and displacement into stress-strain characteristics it is necessary to introduce a number of definitions:

Stress (MPa) = Force (N) / Area (mm^2)

So, if we have a rectangular bar, as shown in **Figure 6.1**, of cross sectional area 'ab' (mm^2) and this has an applied force of 'F' (N) acting upon it then the tensile stress (MPa) in the bar is simply F/ab.

It should be noted that the 'stress' that is normally measured in a tensile test is based upon the original dimensions of the material prior to the application of any force and that this is commonly known as 'engineering stress'. However, under an applied tensile force, a material,

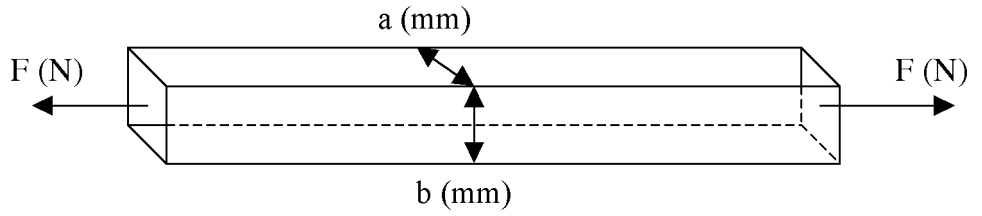


Figure 6.1 Tensile stress generated in a rectangular bar

or a bar such as that shown in **Figure 6.1**, would undergo a contraction of its cross sectional area (it gets thinner as you stretch it) resulting in an actual stress in the material greater than that indicated by an 'engineering stress' calculation. Consequently, if the actual cross sectional area of the material under the applied force is monitored and a stress calculation made based upon the observed dimensions then what is termed the 'true stress' will be calculated.

The definition of strain is:

Strain = Extension δl (mm)/Original Length l_0 (mm)

Consider the same rectangular bar that was used in the stress example, measure the length prior to the application of load l_0 (original length), see **Figure 6.2**, and then the length l_1 of the bar, see **Figure 6.3**, following the application of the force. Then since the extension δl of the bar is simply $l_1 - l_0$, then the strain induced in the bar is $l_1 - l_0 / l_0$.

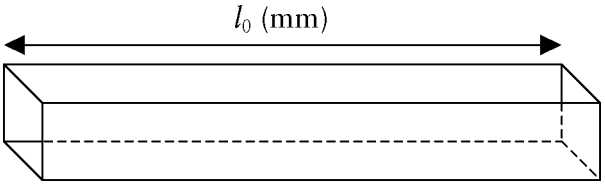


Figure 6.2 Rectangular bar prior to force application

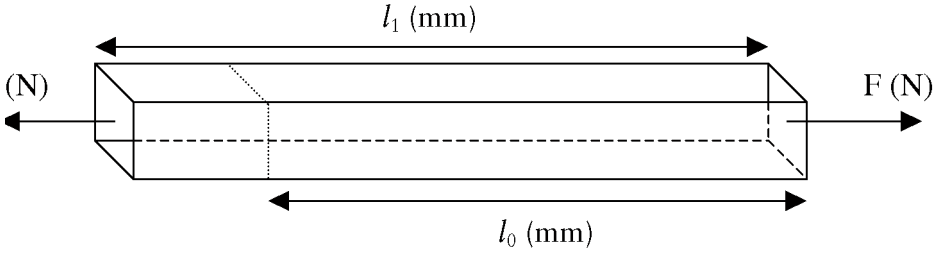


Figure 6.3 Tensile strain generated in a rectangular bar

It is common practice to express strain as a percentage and this is simply accomplished by multiplying by 100:

Strain (%) = (Extension δl (mm) / Original length l_0 (mm)) x 100

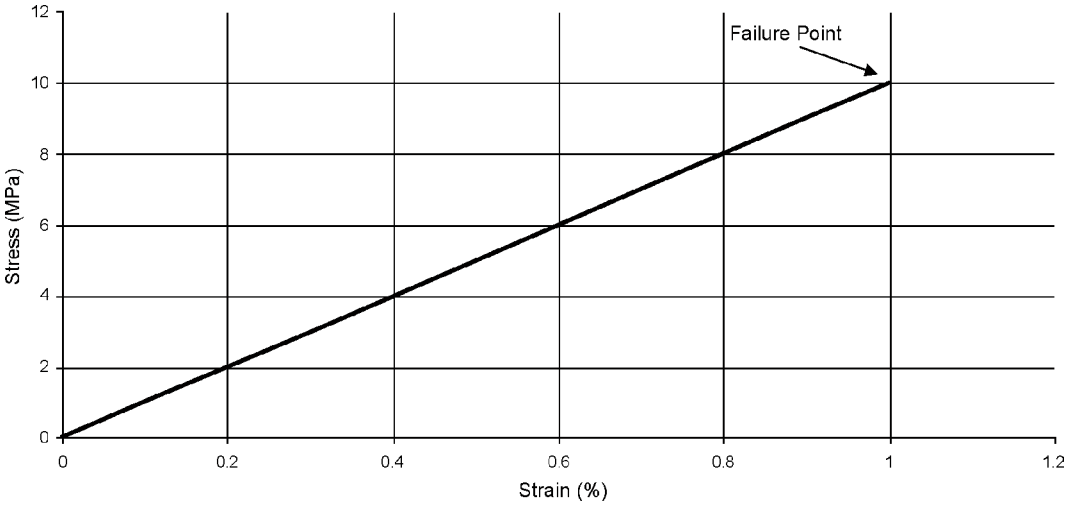


Figure 6.4 Typical stress *versus* strain characteristic for a perfectly elastic solid

Considering the stress-strain characteristic of a perfectly elastic solid through to brittle failure, **Figure 6.4**, it can be seen that the relationship between stress and strain is linear up to the failure point and as a consequence the relationship stress / strain = constant. This constant is known as the elastic modulus of the material and is usually measured in GPa.

Modulus (GPa) = Stress (MPa) / Strain

Unfortunately, polymeric materials are non-linear and consequently their stress-strain characteristic is not as simple as that shown in **Figure 6.4** and hence their modulus is not a constant. The stress strain characteristics of polymeric materials are quite diverse and the four main generic types are discussed in Sections 6.1.1-6.1.4.

6.1.1 Tough Materials with a Yield Stress Greater than the Failure Stress

An idealised stress-strain relationship for a typical tough plastic material with a yield stress greater than the failure stress is shown in **Figure 6.5**. This figure also introduces the concept of a yield point, the lowest stress at which strain increases without increase in stress.

From **Figure 6.5** a number of characteristic values associated with the material under test can be determined:

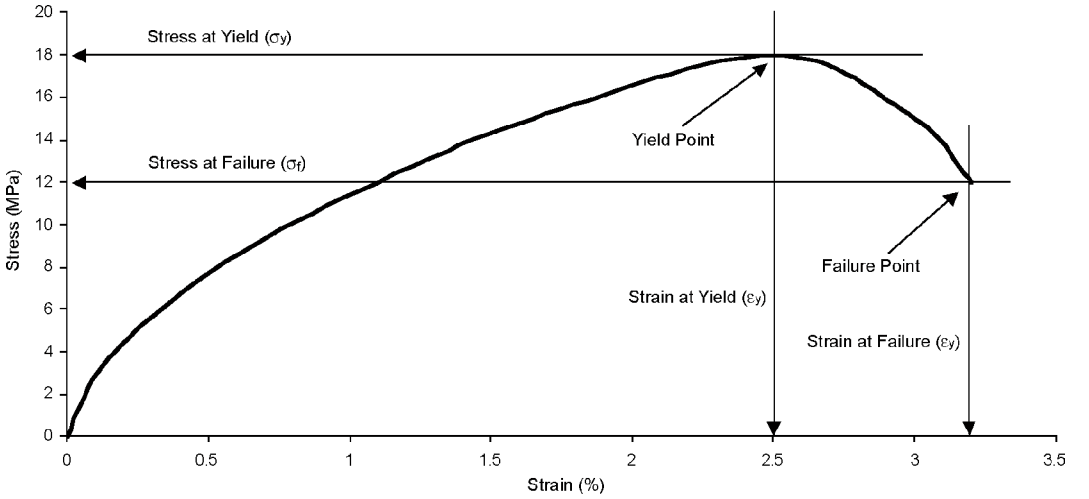


Figure 6.5 Typical non-linear stress *versus* strain characteristic for a material with a yield stress greater than the failure stress

- **Tensile Yield Stress (Yield Stress)**
  The lowest stress at which strain increases without a corresponding increase in stress.
  In the example given in **Figure 6.5**, Yield Stress (σ_y) = 18 MPa

- **Tensile Stress at Break (Failure Stress)**
  The tensile strength of the material at failure.
  In the example given in **Figure 6.5**, Failure Stress (σ_f) = 12 MPa

- **Tensile Strain at Yield (Yield Strain)**
  The corresponding tensile strain of the material at the yield stress.
  In the example given in **Figure 6.5**, Yield Strain (ε_y) = 2.5%

- **Tensile Strain at Break (Failure Strain)**
  The corresponding tensile strain of the material at the failure stress.
  In the example given in **Figure 6.5**, Failure Strain (ε_f) = 3.2%

6.1.2 Tough Materials with a Yield Stress Lower than the Failure Stress

Some materials will, after exhibiting a yield point, once again start to show an increase in stress with increasing strain and this characteristic is shown in **Figure 6.6**.

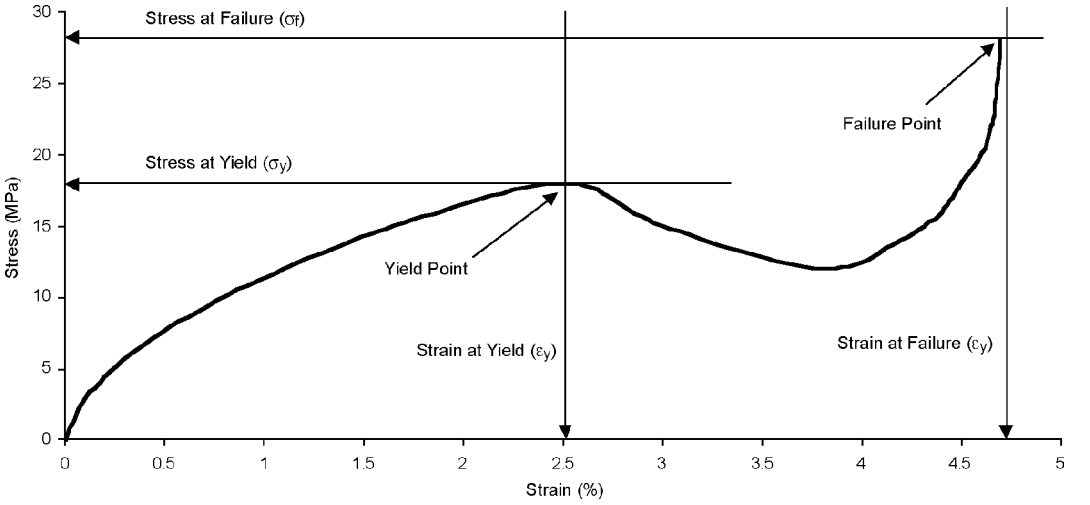


Figure 6.6 Typical non-linear stress *versus* strain characteristic of tough thermoplastic with a failure stress greater than the yield point

For this material the characteristic values that can be attributed are:

- **Tensile Yield Stress (Yield Stress)**
  The lowest stress at which strain increases without a corresponding increase in stress.
  In the example given in **Figure 6.6,** Yield Stress (σ_y) = 18 MPa

- **Tensile Stress at Break (Failure Stress)**
  The tensile strength of the material at failure.
  In the example given in **Figure 6.6,** Failure Stress (σ_f) = 28 MPa

- **Tensile Strain at Yield (Yield Strain)**
  The corresponding tensile strain of the material at the yield stress.
  In the example given in **Figure 6.6,** Yield Strain (ε_y) = 2.5%

- **Tensile Strain at Break (Failure Strain)**
  The corresponding tensile strain of the material at the failure stress.
  In the example given in **Figure 6.6,** Failure Strain (ε_f) = 4.7%

6.1.3 Tough Materials with the same Yield and Failure Stress

An interesting and sometimes perplexing characteristic for the test technician is the material that does not show a drop in stress at yield point, and does not exhibit brittle failure. An example of a typical characteristic is presented in **Figure 6.7** and is discussed further in connection with the strain rate.

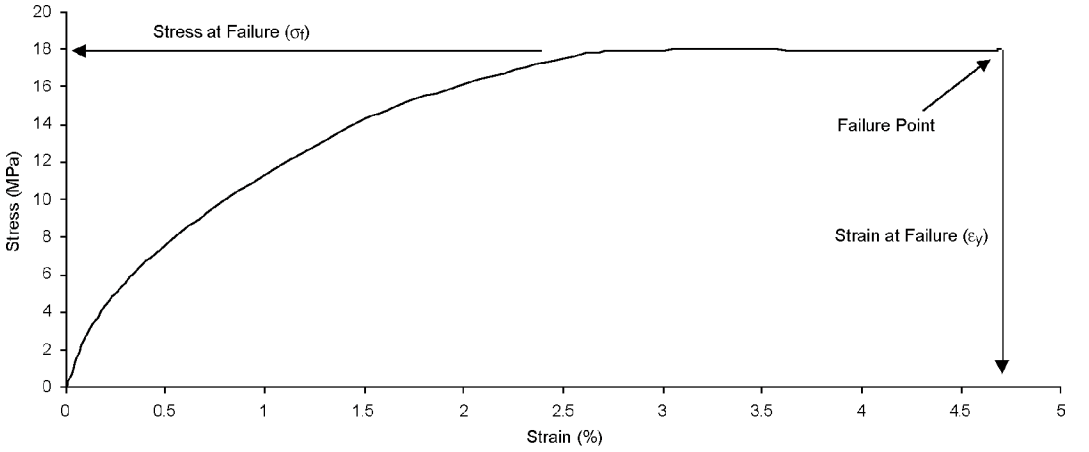


Figure 6.7 Typical non-linear stress *versus* strain characteristic of a tough thermoplastic showing no drop in stress at yield

6.1.4 Brittle Materials

If however, the material is not a tough ductile material, but brittle, then the stress strain curve of the material may look like the idealised curve presented in **Figure 6.8**.

In this instance only the failure stress and failure strain would be determined and in this case they are:

- **Tensile Stress at Break (Failure Stress)**
  The tensile strength of the material at failure.
  In the example given in **Figure 6.7**, Failure Stress (σ_f) = 15 MPa

- **Tensile Strain at Break (Failure Strain)**
  The corresponding tensile strain of the material at the failure stress.
  In the example given in **Figure 6.7**, Failure Strain (ε_f) = 1.6%

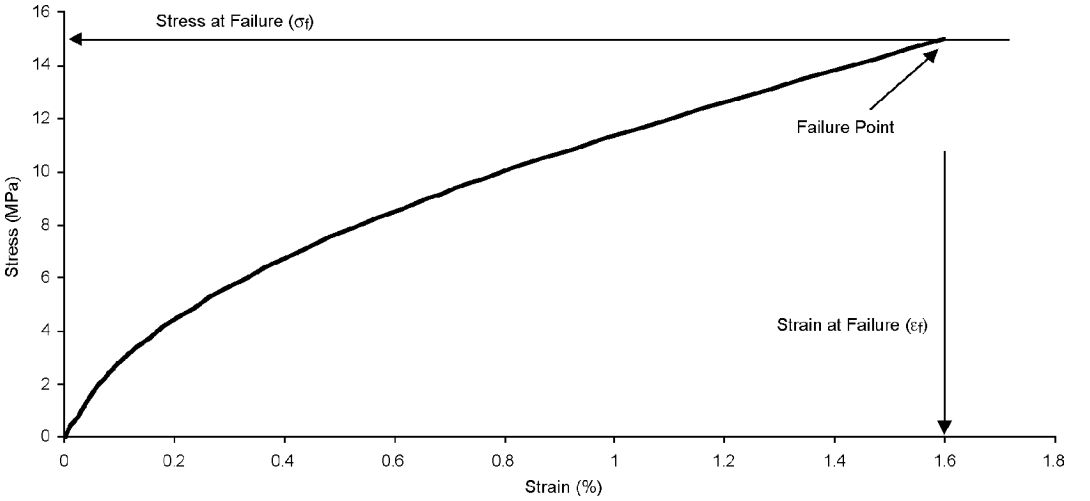


Figure 6.8 Typical stress *versus* strain characteristic of a brittle thermoplastic

With regard to modulus, there are two common types of modulus value that can be determined for a material such as that shown in **Figure 6.9**. The first is the tangent modulus, which is taken as the slope (stress/strain) of the best straight line that can be fitted to the initial portion of the curve and passes through the origin of the graph. The

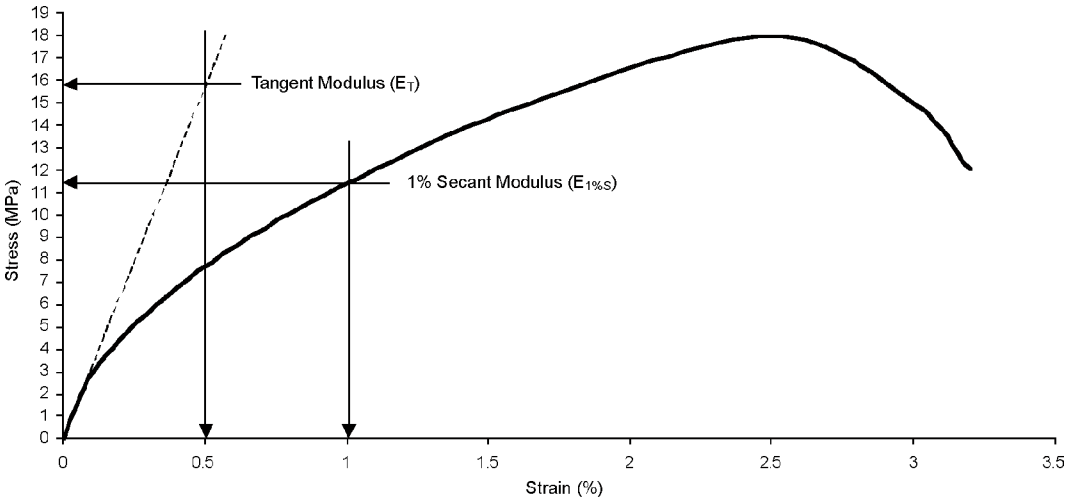


Figure 6.9 Determination of the modulus of a typical non-linear thermoplastic

second is the secant modulus, which is the modulus value that is calculated at a specified strain value, 1% being the most common for rigid materials. Many technologists prefer working with secant modulus because of the uncertainty of fitting a tangent to the stress strain curves found in practice. Referring to **Figure 6.9**:

Tangent Modulus (E_T) = 16.5 / 0.005 = 3300 MPa = 3.30 GPa

1% Secant Modulus ($E_{1\%S}$) = 11.2 / 0.010 = 1120 MPa = 1.12 GPa

NB: Remember to change from percentage strain to strain in the modulus calculations.

The significant differences in modulus that can be determined by using the two different methods are clearly shown in the previous example. It is also important to remember that the strain at which the secant modulus is taken can have a significant effect on the modulus value determined. This can be seen clearly in **Figure 6.10**, which shows how the secant modulus for the curve in **Figure 6.9** changes as the strain increases up to the yield point.

In addition to being non-linear, plastics are also viscoelastic and as a consequence exhibit time dependent behaviour. This can cause problems with tensile testing and care should be taken to establish the rate of extension that tests were conducted at if meaningful comparable data are to be generated. This point can be clearly seen in **Figure 6.11** which shows the effects on the tensile stress-strain response of a high density polyethylene (HDPE) pulled at a number of different rates at 23 °C.

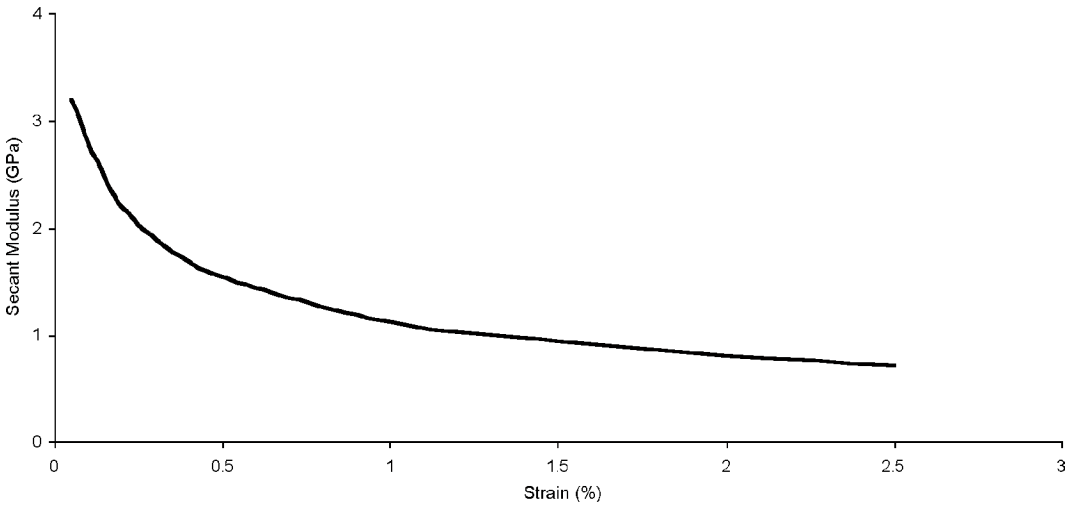


Figure 6.10 Secant modulus *versus* strain for a typical non-linear thermoplastic

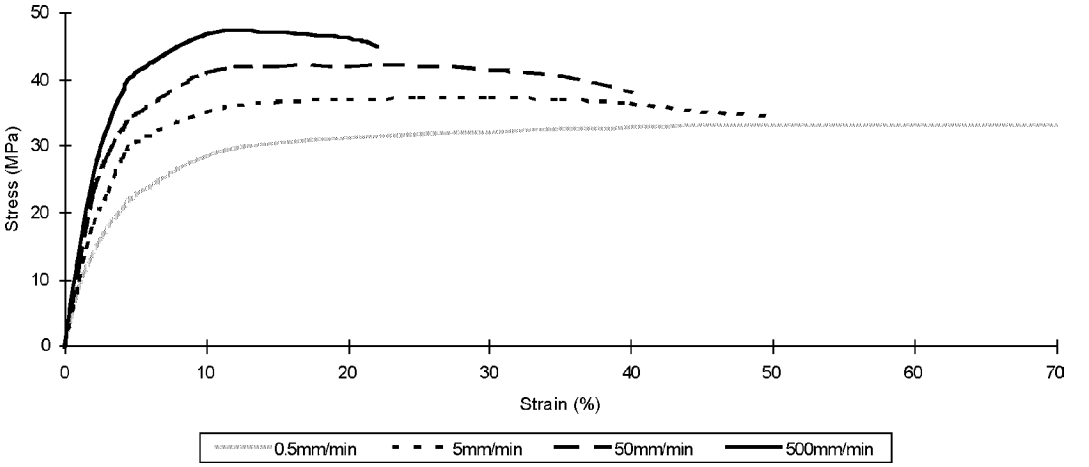


Figure 6.11 HDPE tensile stress *versus* strain curves at 23 °C and increasing strain rates

One of the first things to note in **Figure 6.11** is that the strength, and as a consequence the modulus of the material appears to increase with increasing rate of test. Another interesting feature, is the lack of any discernible yield point in the lowest rate of test and the almost level response of the material. In this instance the material is exhibiting cold flow, caused because the molecules of the HDPE, at this rate of extension, have sufficient time to align themselves just enough to accept the increase in deformation, whilst maintaining a constant resistance to the applied force (constant tensile stress). However, as we look at the other curves it apparent that as the rate of extension increases so does the propensity of the material to form a yield point; in fact if the rate of extension was increased sufficiently the material would start to exhibit brittle behaviour.

6.2 Test Methods

6.2.1 Standard Methods

From the plethora of standards that used to bewilder the uninitiated, a comprehensive set of standards that cover the tensile testing of plastic materials, in most of their converted forms, has been drawn up and covered in the various parts of ISO 527 [1-5]. The plastic materials that are normally excluded from ISO 527 are rigid cellular materials or sandwich structures containing cellular materials.

The British and European standards are identical and dual numbered. The ASTM general test methods are given in D638 [6] for general purpose plastics and D882 [7] for films

and sheeting. D638 is technically equivalent to ISO 527-1 and D882 is similar to ISO 527-3 but with less test piece configurations and differences in test speed. There is provision for the use of microtensile test pieces given in ASTM D1708 [8] where only small quantities of material are available, but only for use where there is a history of data using this test piece; otherwise the very small type V specimen of D638 is recommended. There is a British standard specific to tensile properties of polytetrafluoroethylene (PTFE) [9]

ISO 527-1 links all of the other parts of ISO 527 together and sets out the general principle to be applied.

Building on those given earlier, the following comprehensive set of definitions apply to ISO 527:

- **Gauge Length:** the initial distance between the gauge marks on the central part of the test specimen.
- **Speed of Testing:** the rate of separation of the grips of the testing machine during test.
- **Tensile Stress (engineering):** the tensile force per unit area of the original cross section within the gauge length, carried by the specimen at any given moment.
- **Tensile Stress at Yield (Yield Stress):** the first stress at which an increase in strain occurs without an increase in stress.
- **Tensile Stress at Break:** the tensile stress at which the test specimen ruptures.
- **Tensile Strength:** the maximum tensile stress sustained by the test specimen during a tensile test.
- **Tensile Stress at x% Strain:** the stress at which the strain reaches the specified value x expressed as a percentage. It may be measured, for example, if the stress-strain curve does not show at the yield point. In this case x must be defined either in the relevant product standard or as agreed upon by the interested parties.
- **Tensile Strain:** the increase in length per unit of original length of the gauge. It is used for strains up to the yield point. For strains beyond this limit see **nominal tensile strain.**
- **Tensile Strain at Yield:** tensile strain at the yield stress.
- **Tensile Strain at Break:** the tensile strain at the tensile stress at break, if it breaks without yielding.
- **Tensile Strain at Tensile Strength:** the tensile strain at the point corresponding to the tensile strength, if this occurs without or at yielding.

- **Nominal Tensile Strain:** the increase in length per unit original length of the distance between grips (grip separation). It is used for strains beyond the yield point. It represents the total relative elongation that takes place along the free length of the test specimen.

- **Nominal Tensile Strain at Break:** the nominal tensile strain at the tensile stress at break, if the specimen breaks after yielding.

- **Nominal Tensile Strain at the Tensile Strength:** the nominal tensile strain at the tensile strength, if the specimen breaks after yielding.

- **Modulus of Elasticity in Tension (Young's Modulus):** the ratio of the stress difference to the corresponding strain difference. These strains are defined in the standard as being 0.05% and 0.25%. Also known as Young's modulus. This definition is not applicable to films.

- **Poission's Ratio:** the negative ratio of tensile strain in one of the two axis normal to the direction of pull to the corresponding strain in the direction of pull within the initial linear portion of the longitudinal versus normal strain curve.

6.2.2 Test Apparatus

The core piece of apparatus is the tensile testing machine (which generally can also be used for compression, flexural and shear tests) which incorporates fixed and driven members together with a load cell for measuring force. Grips are required to hold the test piece and when elongation or modulus is to be measured an extensometer is necessary. Also, a suitable micrometer and/or dial gauge is needed to measure the test piece dimensions.

ISO 527-1 specifies that the tensile testing machine shall comply with ISO 5893 [10]. This standard covers constant rate of traverse, tensile, flexural and compression test equipment for rubber and plastics materials.

The accuracy of the force measuring capability of machines complying to ISO 5893 is designated as either grade A or B. The maximum permissible values for precision and accuracy of grade A and B machines are as presented in **Table 6.1** and the error is presented in **Figure 6.12**. ISO 527 calls for ± 1% which equates with grade A.

Table 6.1 Force – Measurement grades					
Grade	**Accuracy of verification device**	**Certified Range**			
		One-fifth of full scale to full scale		**Below one-fifth of full scale**	
		Precision requirement	**Accuracy requirement**	**Precision requirement**	**Accuracy requirement**
		At each verification force, maximum permissible difference between highest and lowest readings, expressed as a percentage of the verification force	At each verification force, maximum permissible error, expressed as a percentage of the verification force	At each verification force, maximum permissible difference between highest and lowest readings, expressed as a percentage of the full scale reading	At each verification force, maximum permissible error, expressed as a percentage of the full-scale reading
	%	%	%	%	%
A	± 0.2	1.0	± 1.0	0.2	± 0.2
B	± 0.3	2.0	± 2.0	0.4	± 0.4

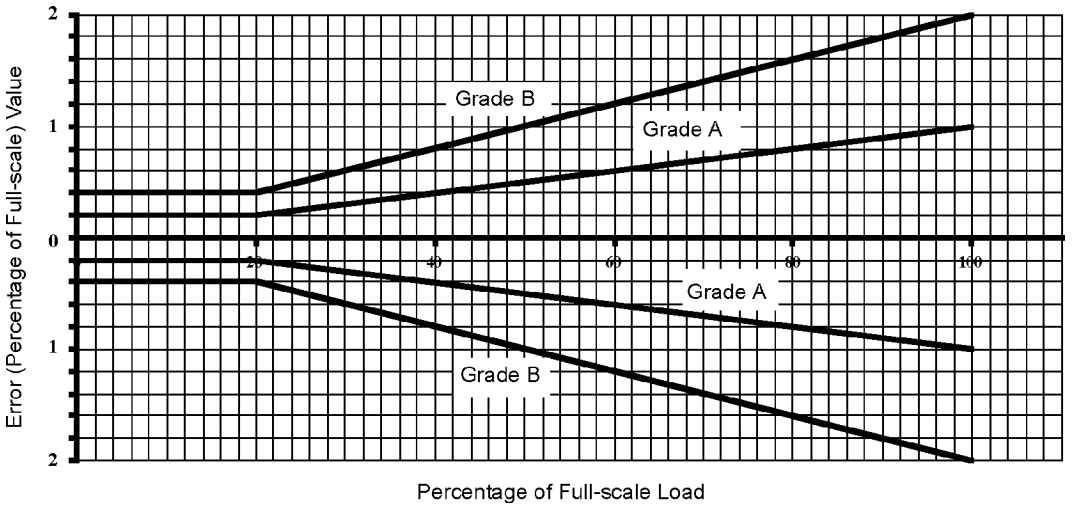


Figure 6.12 Machine accuracy gradings

6.2.2.1 Tensile Testing Machines

There are essentially only two types of tensile testing machines used commercially, electromechanical and servohydraulic.

Electromechanical machines are available in two configurations, twin-screw and centre-screw; with the most common being the twin-screw, shown schematically in **Figure 6.13**. The moving crosshead is operated by a positional servomechanism that incorporates an amplidyne power drive and synchro-control elements

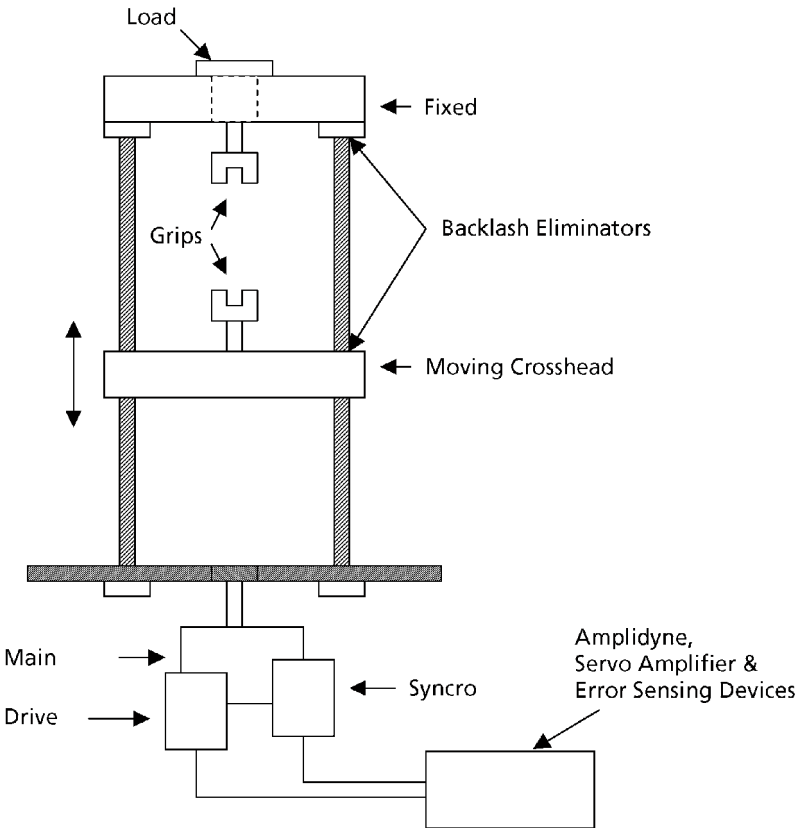


Figure 6.13 Achematic of an electromechanical twin-screw tensile test machine

Servohydraulic machines do not use a moving crosshead, they apply tensile or compressive forces through a hydraulic ram, as shown schematically in **Figure 6.14**.

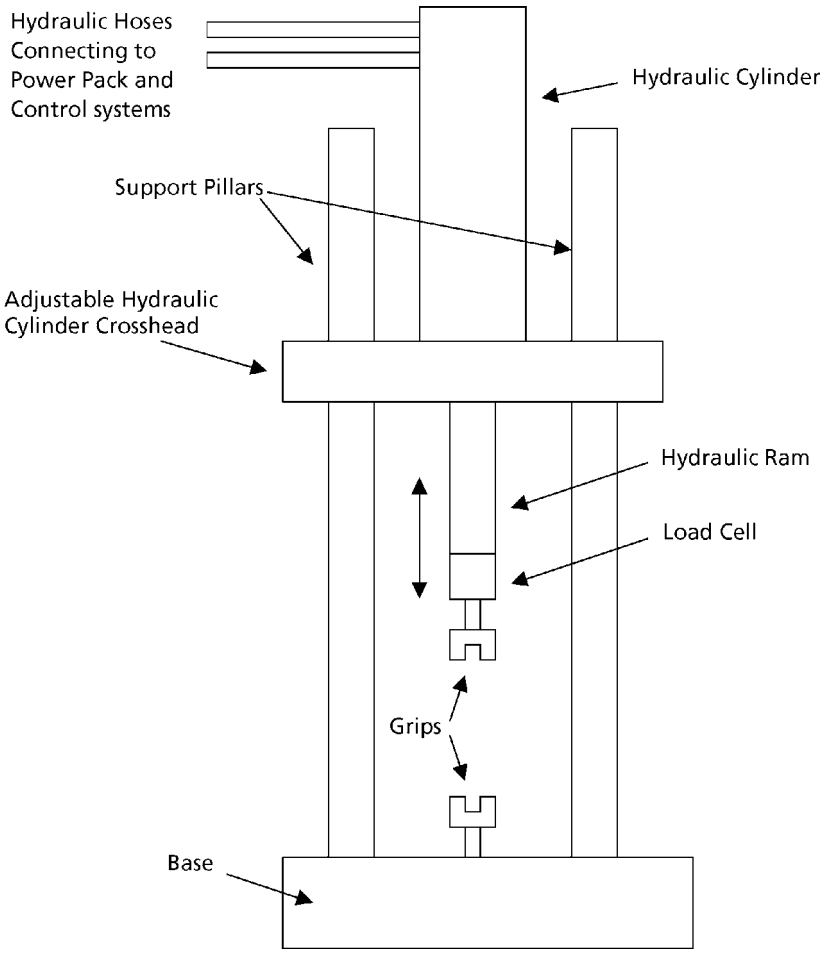


Figure 6.14 Schematic of a servohydraulic tensile test machine

6.2.2.2 Speed of Testing

ISO 527-1 specifies that any testing machine used shall be capable of maintaining the speeds of testing given in **Table 6.2**.

Table 6.2 ISO 527 part 1 recommended testing speeds	
Speed, mm/min	**Tolerance, %**
1	± 20
2	± 20
5	± 20
10	± 20
20	± 10
50	± 10
100	± 10
200	± 10
500	± 10

6.2.2.3 Grips

The standard, quite reasonably, requires that:

- The grip system is arranged such that the major axis of the test specimen coincides with the direction of pull through the centreline of the grip assembly.
- That the specimen is prevented from slipping relative to the grips and that the grip maintains or increases pressure on the test specimen as the force applied to the specimen increases.
- And that the grip system shall not cause premature failure at the grips. This can be quite difficult at times when one is dealing with particularly brittle or unidirectional continuous fibre-filled materials.

The types of grip systems available are far to numerous to detail here, however an example of the most common general-purpose system for rigid materials is presented schematically in **Figure 6.15**. The operation of wedge grips is quite simple and reminiscent of 'Chinese finger traps'. As the sample is put under a tensile load, the sample and wedges try to move out of the grip housing, however, the tapered grip housing forces the wedges closer together, exerting an ever greater pressure on the sample. Consequently, as the tensile load increases during the test so the force holding the sample firmly in place increases.

A similar principle is used in various designs of grip for flexible materials. An alternative way to maintain force on the test piece is to use grips with the jaws closed under pneumatic pressure.

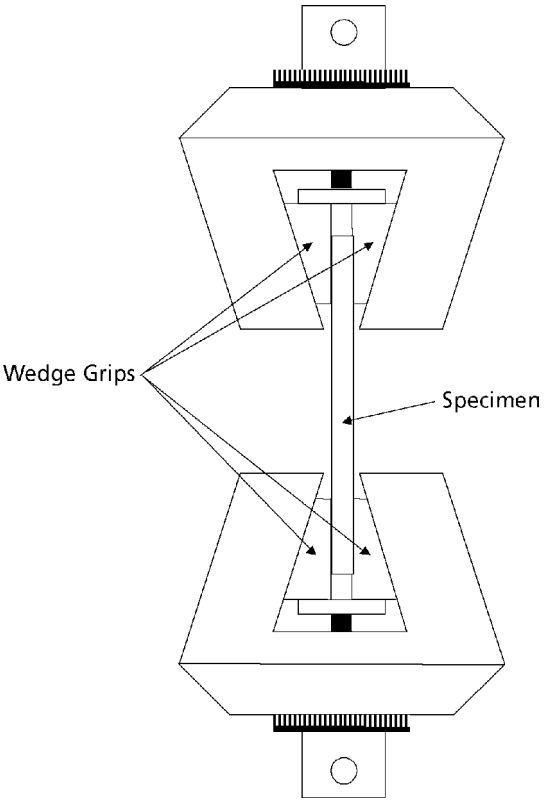


Figure 6.15 Typical wedge grip assembly

6.2.2.4 Extensometers

ISO 527-1 requires that the extensometer complies with ISO 5893 and should measure the change in gauge length with an accuracy of 1% of the relevant value or better. This is confusing as ISO 5893 specifies a series of extensometers which differ only in the maximum elongation they will measure and the gauge length, the accuracy in each case being ± 2%. Clearly, the accuracy required in length units depends on the level of elongation being measured, which varies greatly between rigid and flexible materials.

As with grips, there are many different types of extensometer that can be used to monitor the strain in a tensile test. For fairly high modulus engineering plastics, the so called 'clip on strain gauge' type, a schematic of which is given in **Figure 6.16**, is suitable. The principle of operation is very simple, four matched foil type resistance strain gauges are bonded to the curved spring steel, two on the outer surface and two on the inner surface.

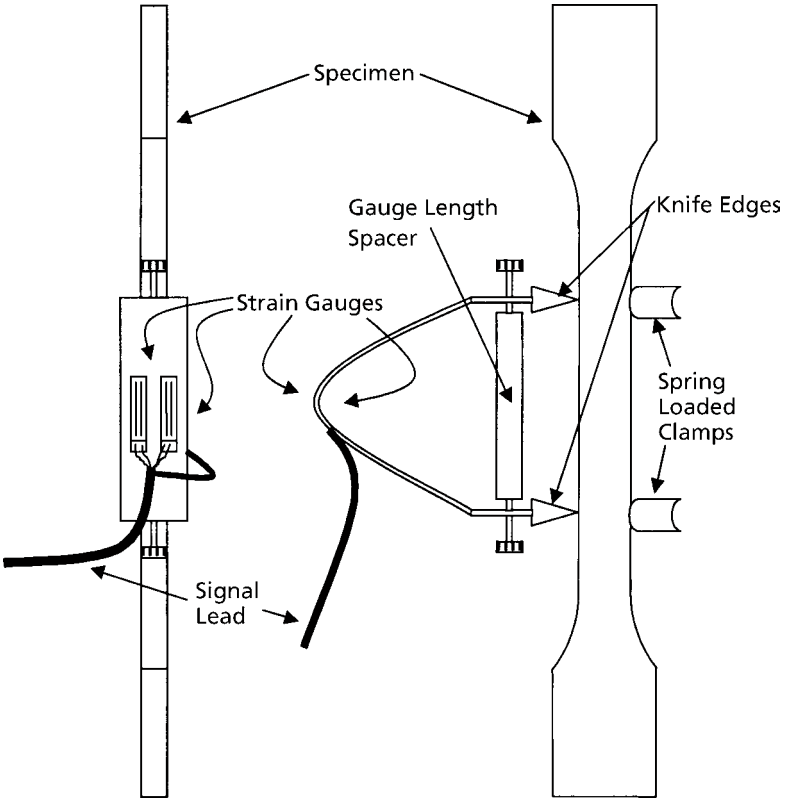


Figure 6.16 Schematic of a clip on strain gauge extensometer

In this arrangement, as a specimen extends so the arms of the spring move further apart causing the gauges on the outer surface of the spring to go into compression and the gauges on the inner surface of the spring to go into tension. The resultant variations in resistance are proportional to the level of strain in the specimen.

However, for low modulus and/or thin materials, where the weight of a clip on the extensometer would significantly distort the specimen even before the application of a test load, the preferred type of extensometer is what is termed the non-contacting type. As the name implies, the non-contacting type of extensometers are not attached physically to the specimen under test, rather they use some form of light to illuminate and follow gauge marks which have been made on the specimen. Usually these gauge marks have been drawn, painted or even stuck onto the surface of the specimen prior to testing. There are three primary types of non-contacting extensometer, optical, laser and video and these are briefly described next:

- **Optical Extensometers**

Optical extensometers, see **Figure 6.17** below, use either visible or infrared light to illuminate gauge marks on the specimen. Photoelectric sensing devices, using a servo mechanism, then follow the gauge marks as the specimen extends or contracts and the resulting movement is monitored using a displacement transducer, the output from which is sent to some form of recording device.

Laser extensometers, see **Figure 6.18**, use reciprocating or rotating mirrors to sweep a laser beam between the gauge marks on the specimen. If the angle subtended between the gauge marks and either the original gauge length or the distance between the specimen

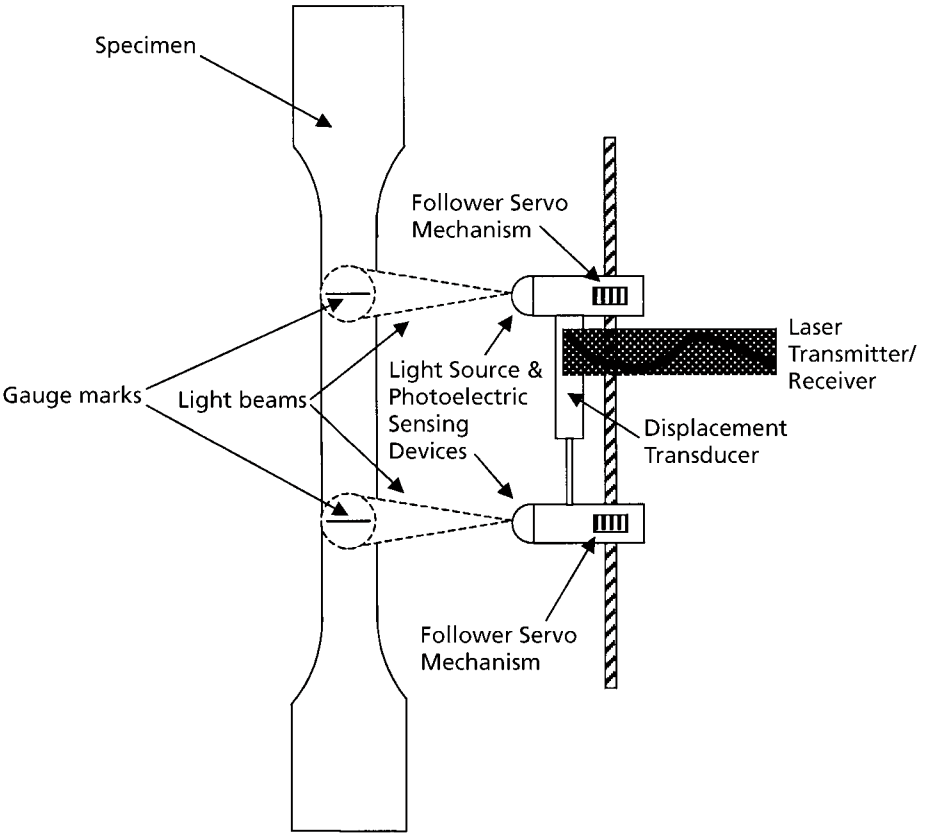


Figure 6.17 Optical extensometer

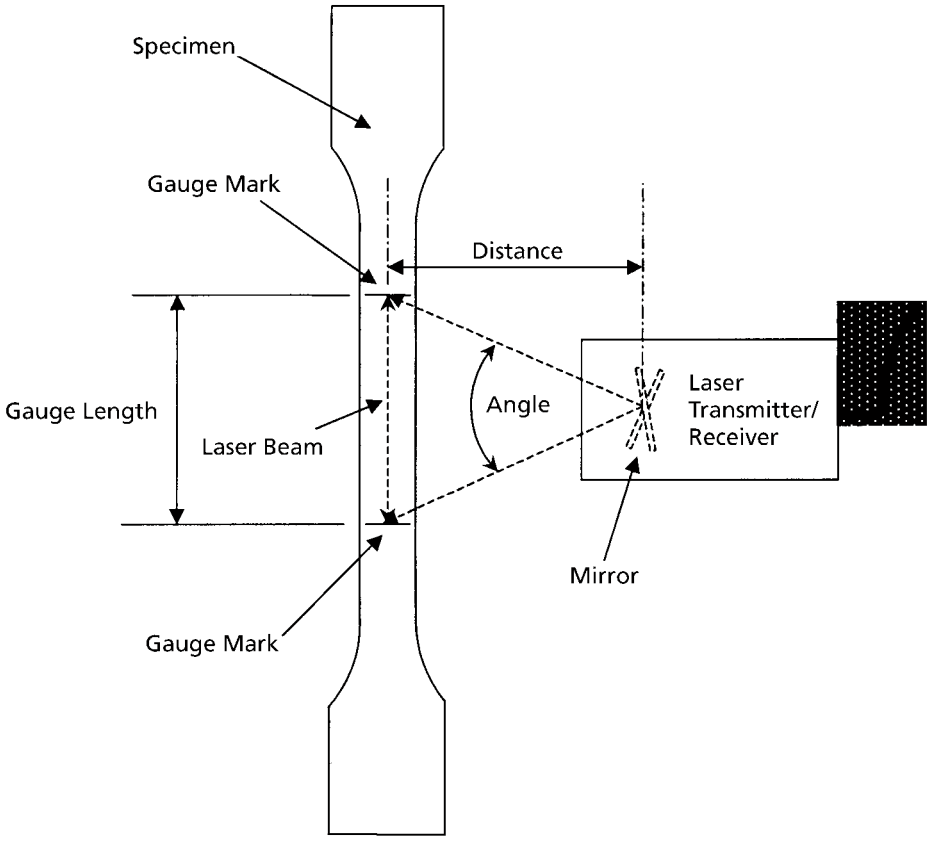


Figure 6.18 Laser extensometer

and the mirror is known, then as the gauge length changes, so the angle subtended between the gauge marks will change and the displacement of the specimen when subjected to a load can be calculated.

Video extensometers, see **Figure 6.19**, produce a real time image of the specimen and associated gauge marks. The resultant images are transmitted to a computer that then uses special software to process the images, in particular the original accurately measured gauge marks and the subsequent relative positions of the gauge marks to determine their displacement. The displacement can then be used to calculate the strain induced in the specimen. One advantage of the video system is that it produces a visual record of the test that has been conducted.

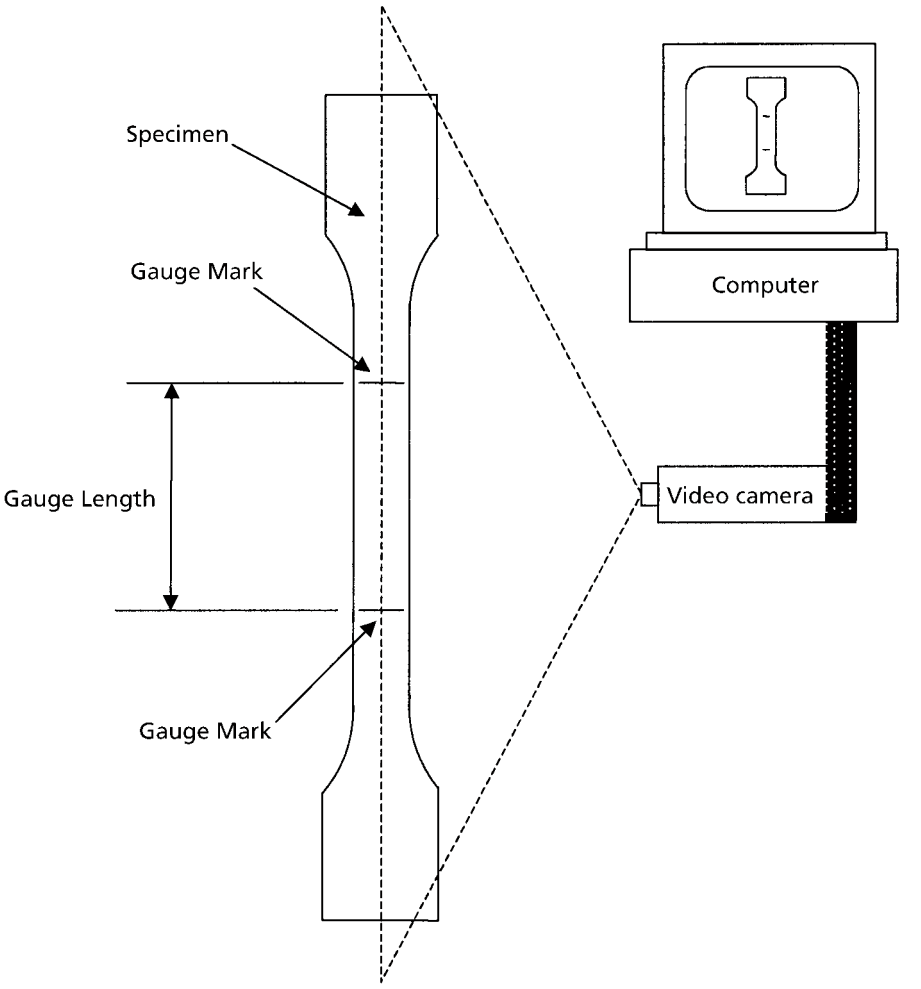


Figure 6.19 Video extensometer

6.2.3 Test Pieces

A minimum of 5 test pieces are required by ISO 527-1 for each direction tested and if different speeds are used for modulus and ultimate strength then separate sets of test pieces will be needed. The shape and size of test pieces is specified in ISO 527 Parts 2-5 and is depending on the material being tested.

6.2.3.1 ISO 527-2 Test conditions for Moulding and Extrusion Plastics

This standard covers the following materials:

- Rigid and semirigid thermoplastics moulding, extrusion and cast materials, including compounds filled and reinforced by for example, short fibres, small rods, plates or granules but excluding textile fibres (see ISO 527-4 and ISO 527-5) in addition to unfilled types.

- Rigid and semirigid thermosetting moulding and cast materials, including filled and reinforced compounds but excluding textile fibres as reinforcement (see ISO 527-4 and ISO 527-5).

- Thermotropic liquid crystal polymers.

The preferred test specimens that are applicable to this standard are either Type 1A or 1B, see **Figure 6.20**, with type 1A being used for directly moulded specimens and type 1B for machined specimens. However, if it is not possible to use type 1 specimens, then specimens of the type 1BA, 1BB (see **Figure 6.21**), 5A or 5B (see **Figure 6.22**) may be used, provided that the speed of testing is adjusted to the value given in the recommended speeds of testing (see Table 6.2) which gives the nominal strain rate for the small test specimen closest to that used for the standard-sized specimen.

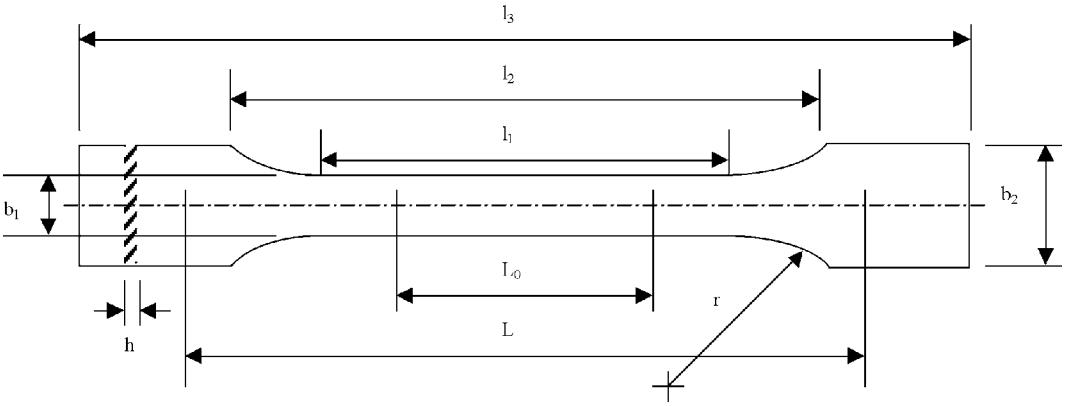
For example, if one normally used a type 1A test specimen with a gauge length of 50 mm at a speed of test of 50 mm/min, then the nominal strain rate would be 100%/min. If, however, one had to use a 1BB test specimen with a gauge length of 10 mm, then the speed of test would have to be dropped from 50 mm/min. which would give a nominal strain rate of 500%/min, to the nearest rate from Table 6.1 that would give a nominal strain rate of 100%/min, which in this example conveniently turns out to be 10 mm/min.

These test pieces are essentially for rigid materials and it would be more common to use the type 5 test piece of ISO 527-3 (see Section 2.3.2) for flexible materials more than 1 mm thick.

6.2.3.2 ISO 527-3 Test Conditions for Films and Sheets

This standard covers the following materials:

- Plastic films or sheets less than 1 mm thick, however cellular materials and plastics reinforced by textile fibres are not normally considered suitable for testing using this standard.



	Type 1A (mm)	Type 1B (mm)
l_3	≥150	≥150
l_2	104 to 113	106 to 120
l_1	80 ± 2	60 ± 0.5
b_2	20 ± 0.2	20 ± 0.2
b_1	10 ± 0.2	10 ± 0.2
h	4.0 ± 0.2	4.0 ± 0.2
L_0	50.0 ± 0.5	50.0 ± 0.5
L	115.0 ± 1	l_2 + 5
r	20 to 25	≥60

Figure 6.20 Test specimens type 1A and 1B

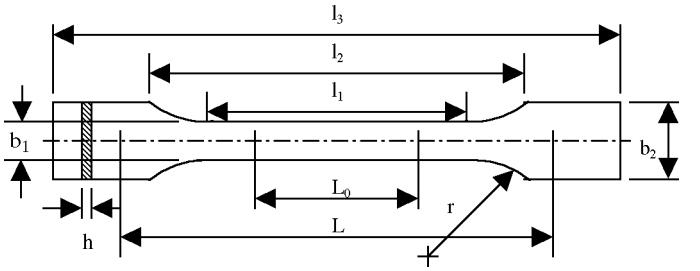
The preferred test specimen that is applicable to this standard is Type 2, see **Figure 6.23**.

For routine quality control tests the following specimens are recommended:

Type 5 Recommended for film and sheet with a very high strain at break, see **Figure 6.24**.

Type 1B Recommended for rigid sheets, see **Figure 6.25**.

Type 4 Recommended for other types of flexible thermoplastic sheet, see **Figure 6.26**.



	Type 1BA (mm)	Type 1BB (mm)
l_3	≥75	≥30
l_2	58 ± 2	23 ± 2
l_1	30 ± 0.5	12 ± 0.5
b_2	10 ± 0.5	4 ± 0.2
b_1	5 ± 0.5	2 ± 0.2
h	≥2	≥2
L_0	25 ± 0.5	10 ± 0.2
L	l_2 + 2	l_2 + 1
r	≥30	≥12

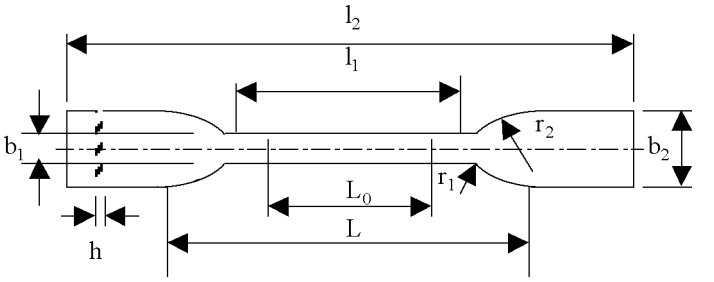
Figure 6.21 Test specimens type 1BA and 1BB

6.2.3.3 ISO 527 Parts 4 and 5 Test Conditions for Fibre Reinforced Plastics

ISO 527-4 covers isotropic and orthotropic materials whereas ISO 527-5 covers unidirectional materials. Usually, parallel strip test pieces are used with tab ends adhered to the strip to aid gripping.

6.2.4 Procedure

The general procedure for tensile testing is given in ISO 527-1.



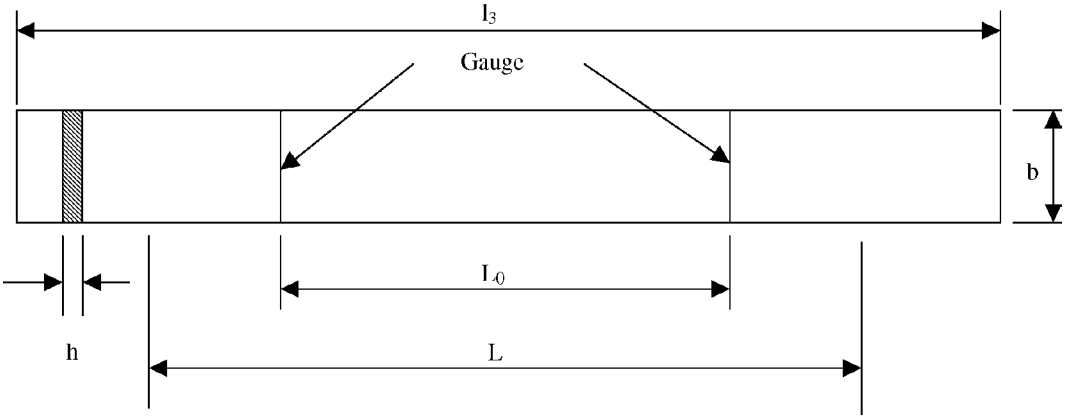
	Type 5A (mm)	Type 5B (mm)
l_2	≥75	≥35
l_1	25 ± 1	12 ± 0.5
b_2	12.5 ± 1	6 ± 0.5
b_1	4 ± 0.1	2 ± 0.1
h	≥2	≥1
L_0	20 ± 0.5	10 ± 0.2
L	50 ± 2	20 ± 2
r_2	12.5 ± 1	3 ± 0.1
r_1	8 ± 0.5	3 ± 0.1

Figure 6.22 Test specimens type 5A and 5B

Specific recommendations are not made for conditioning and test temperature but reference is made to ISO 291 [11].

The need to ensure good alignment of the test piece with the axis of the machine and to avoid pre-stresses, particularly with less rigid materials is emphasised. Quite sensibly, ISO 527-3 of the standard states that when testing thin sheets or film material, the specimen shall not carry the weight of the extensometer.

Testing speed is supposed to be selected in accordance with the relevant material specification except that for modulus the speed should be such as to give a strain rate of 1% of the strain rate per minute. This is an over-generalisation and would not be appropriate for all materials. ISO 527-3 does, however, state that films and sheets are



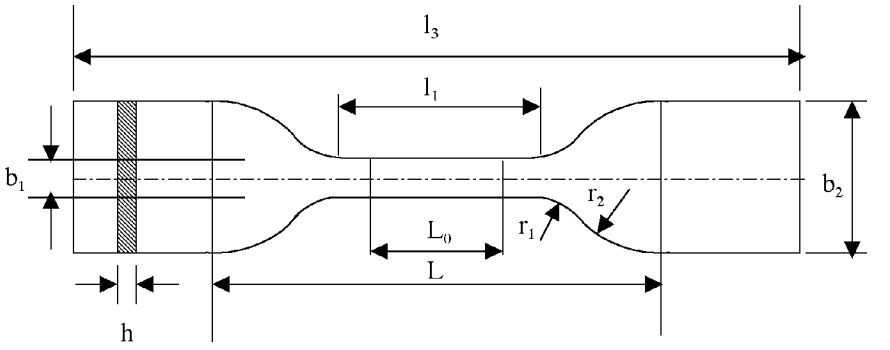
	Type 2 (mm)
l_3	≥150
b	20 to 25
h	≤1
L_0	50.0 ± 0.5
L	100 ± 5

Figure 6.23 Test specimens type 2

usually tested at 5, 50, 100, 200, 300 (this is an additional speed of testing not specified in Table 1 of ISO 527-1), 500 mm/min, but this is little help for selecting a speed in any particular case.

The results from test pieces that yield or break outside of the parallel portion are rejected. However, it should be noted that if at yield, necking occurs outside of the gauge length extremely odd elongation results can be obtained when using an extensometer. Generally, there is little practical meaning to elongation beyond yield but the definition of nominal tensile strain that takes account only of the grip movement can be used.

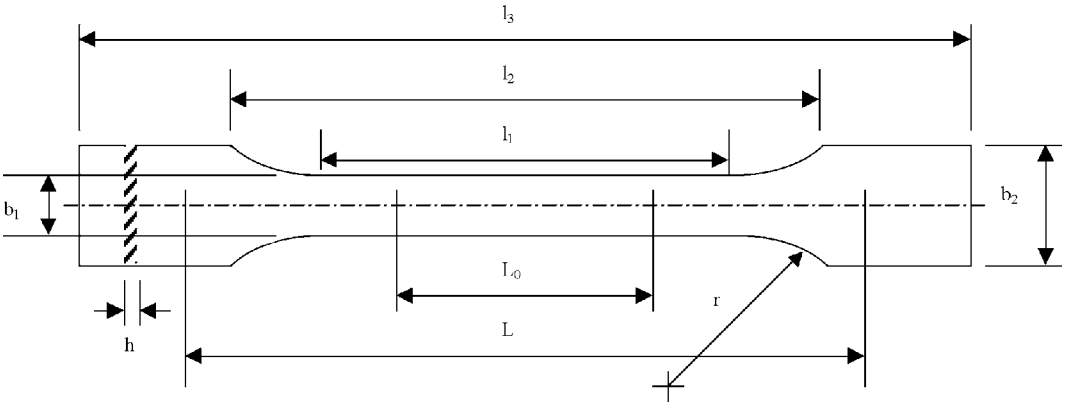
The force–elongation curve is normally recorded automatically. From this any of the defined stress, strain and modulus figures can be calculated although in most cases they will not all be wanted. The specific definition of Young's modulus determined between



	Type 5 (mm)
l_3	≥115
l_1	35 ± 2
b_2	25 ± 1
b_1	6 ± 0.4
h	≤1
L_0	25 ± 0.25
L	80 ± 5
r_2	25 ± 2
r_1	14 ± 1

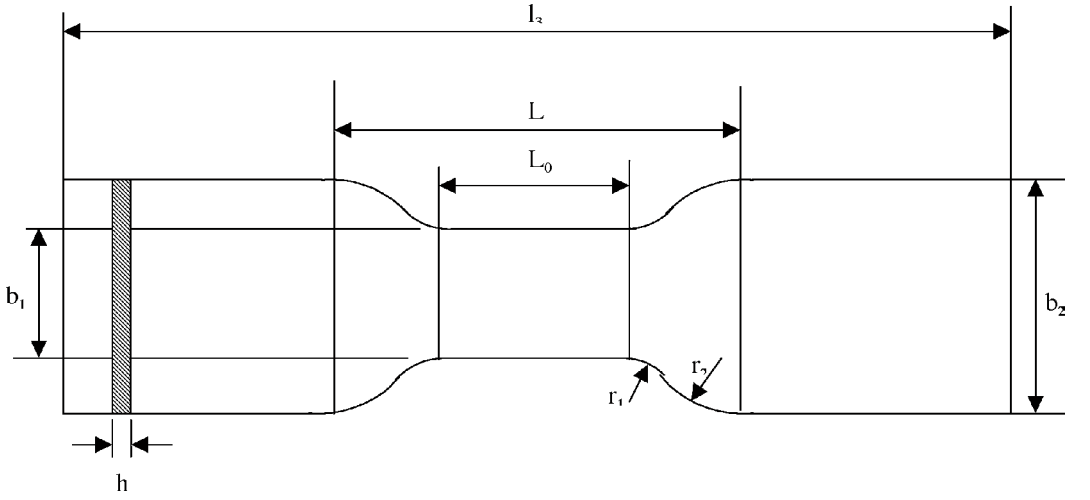
Figure 6.24 Test specimens type 5

two arbitrary strain levels (0.05% and 0.025% of gauge length) requires very high accuracy of the extensometer and is probably unrealistic in many cases. Although not specified, secant modulus may be preferred. Provision is made for measuring Poisson's ratio but this is not common and requires a means of accurately recording the change in width or thickness of the test piece as well as the elongation measurement.



	1B
l_3	≥150
l_1	60 ± 0.5
b_2	20 ± 0.5
b_1	10 ± 0.2
h	≤1
L_0	50.0 ± 0.5
L	115 ± 5
r	≥60

Figure 6.25 Test specimens type 1B



	Type 4 (mm)
l_3	152
b_2	38
b_1	25.4 ± 0.1
h	≤1
L_0	50.0 ± 0.5
L	73.4
r_1	22
r_2	25.4

Figure 6.26 Test specimens type 4

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7 Compression Stress-Strain

7.1 Introduction

It is logical to determine compression stress-strain characteristics of materials when they are to be used in this mode of deformation. For softer materials such as foams and rubbers this is often the case and the experimental approach is relatively straightforward, although complicated by shape factor effects. Rigid plastics are also frequently subjected to compressive stresses but in most applications, shear or tensile stresses are more important. However, even if the shear modulus is not of prime interest the geometry of components can be such that buckling is a potential problem.

Standard compression tests on rubbers and foams are carried out on test pieces with height significantly smaller than compressed area, usually a disk or short cylinder. Although buckling is not then an issue, the ratio of height to area is important, together with whether or not the test piece is lubricated, because of shape factor effects.

In theory there are two conditions under which a test piece can be compressed: either with perfect slippage between the test piece and compressing members of the apparatus or with complete absence of slip. If there were perfect slippage, every element of the test piece would be subjected to the stress and strain and a cylindrical test piece would remain a true cylinder without barrelling. Relationships have been derived for rubbers to take account of shape factor [1] but this does not appear to have been investigated for plastics. Because the stress-strain relationship is generally not linear and because of shape factor effects, care must be taken to compare only measures of compressive stiffness defined in the same way.

For soft plastics, compressive stress-strain properties are sensibly made by adaptation of rubber testing methods [2]. Standard test methods for rigid plastics often use test pieces, which may be a right prism, cylinder or tube, with the height greater than the diameter or width. This geometry will improve the accuracy of modulus measurements of stiff materials where the strains are small but the slenderness ratio must be chosen to avoid buckling at the strains likely to be reached during the test.

If the compressive force is applied to failure this can occur by the test piece shearing at an angle to its height. With more slender test pieces buckling would occur, whereas with a squat test piece failure by crushing would only be achieved at extremely high forces.

7.2 Test Apparatus

There are test machines available which are specifically designed for compression testing but generally a universal test machine, also used for tensile, flexural tests, etc., is used. The apparatus is equipped with a compression load cell and compression takes place between parallel steel plates which may incorporate a self alignment mechanism. It will be appreciated that the alignment of the loading platens is extremely important. An alternative is to use a compression cage in a tensile machine to effectively reverse the motion of the machine. However, these are now seldom seen as they can introduce considerable friction errors and there is always difficulty in alignment.

The forces generated in compression tests is generally much larger than for tension and, consequently, if one machine is used for both it will need to be of greater capacity than for tensile alone. Because of the high forces and relatively small deformations it is also necessary that the machine has high stiffness.

Strain is preferably measured by movement of the compression platens (or gauge marks on the test piece) by means of a convenient transducer, which in its simplest form could be a dial gauge. Machine crosshead movement can be used if the machine is sufficiently stiff in relation to the test piece to avoid the introduction of errors.

7.3 Standard Tests

The measurement of compressive properties of plastics generally is covered by ISO 604 [3]. It is specifically not intended to cover textile reinforced plastics, cellular materials and sandwich constructions involving cellular materials and, although its scope includes semi-rigid materials, it is not suitable for highly plasticised materials.

With the obvious substitution of decrease in length for increase in length, many of the definitions of parameters are similar to those for tensile properties. A distinction is made between compressive strain (based on the change in length between gauge marks) and nominal compressive strain (based on change in length of the test piece), but the significance of this is not obvious.

The shape of the test piece can be quite varied in terms of the cross section and the slenderness ratio. The standard permits the use of right prisms, cylinders or tubes for cross section. Test pieces for compression testing must have very flat and parallel end faces, parallelism to 0.025 mm normal to the long axis of the test piece being required. Lathes or milling machines are recommended for their preparation. As mentioned earlier, buckling is an important consideration in compression tests. Short, squat, test pieces are much less prone to buckling than tall, slender, test pieces and hence there are certain restrictions placed on

the ratio of key dimensions. The two key dimensions are the test piece length (the direction along which the compressive force is applied) and the critical dimension at right angles to this. The relationship between the parameters which govern buckling is given as:

$$\varepsilon_c^* \leq 0.4\left(\frac{x}{l}\right)^2$$

where ε_c^* is the maximum nominal compressive strain experienced during the test;

l is the length of the test piece;

x is the diameter if the test piece is a cylinder, the outer diameter if it is a tube, or the thickness, i.e., the smaller lateral dimension, if it is a prism.

This equation is based on linear stress-strain behaviour and some explanation of critical strain to cause buckling is given in an appendix.

In notes rather than in the text, an *x/l* ratio of not less than 0.08 is recommended for measuring modulus and for other tests not less than 0.4. The smaller ratio for the modulus test is given since the strain range required for the test is so small and hence the point of buckling is unlikely to be reached.

Two preferred test piece sizes are given (see **Figure 7.1**):

for modulus: length = 50 mm, width = 10 mm and thickness = 4 mm
for strength: length = 10 mm, width = 10 mm and thickness = 4 mm

These test pieces can be prepared from the multipurpose test piece given in ISO 3167 [4] and this source of test piece is preferred. Two smaller test pieces are defined in an appendix for use when little material is available.

Gauge marks are required to measure compressive modulus if an optical method is used to measure change in length but the magnitude of the gauge length is not specified, only that the marks should be approximately equidistant from the mid point of the test piece.

This treatment of test pieces could hardly be called elegant standardisation.

The requirements for test speed are similarly complicated. The speed is chosen from 1, 2, 5, 10 or 20 mm/min, the choice depending on the nature of the material and the measurement being made. For modulus, the speed should be that closest in value to 5% of the specimen length. For strength tests on brittle materials the speed should be closest to 10% of the specimen length and for ductile materials to 50% of this length. Hence,

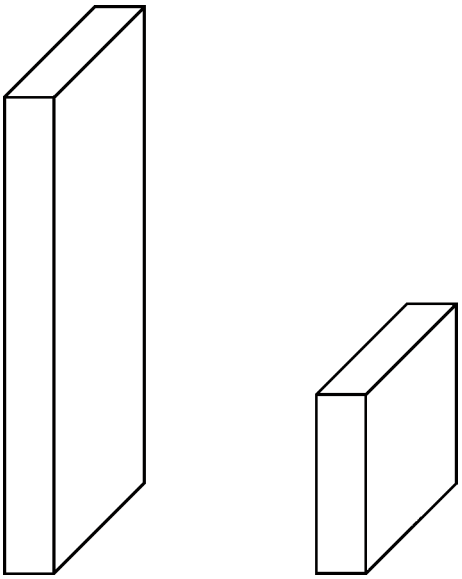

Figure 7.1 Compression test pieces

for the standard test pieces, the most appropriate speed for modulus and for the strength of brittle materials is 1 mm/min, and 5 mm/min for the strength of ductile materials.

The effect of whether or not the ends of test pieces are lubricated is mentioned in a note and for most precise measurements it is recommended that they are either lubricated or discs of fine abrasive paper be used between test piece and platens!

It is intended that the stress-strain curve is recorded so that all of the defined parameters can be deduced. In practice, only those relevant or of interest would be extracted. The only compression modulus considered is that obtained from the difference between the stresses at 0.05 and 0.25% strain, which would only be reasonable for materials within a particular band of stiffness.

At the time of writing, a revision of ISO 604 is under consideration which appears to be concerned with changes of relative detail and editorial order rather than any major change of concept.

The corresponding standard British standard is identical and numbered as BS EN ISO 604 [3]. The ASTM standard is D695 [5] which is said to be technically equivalent to the ISO method but, as is often the case with ASTM standards so described, has a number of differences.

ASTM D695 gives rather more information on the nature of compression stress-strain curves and more detail of suitable compression devices. The test pieces are different and include provision for fibre reinforced materials and syntactic foam as well as a dumbbell shape (with supporting jig) for thin materials. The standard test piece is a right cylinder or prism with length twice the principal width or diameter. The standard test speed is 1.3 mm/min. The expression of results is different to ISO, in particular modulus is obtained from a tangent to the initial linear portion of the stress-strain curve.

BS 2782-3 Method 346A [6] has a compression test which measures the cohesion between layers of laminated tube.

7.3 Other Tests

Tests on fibre reinforced composites are outside the scope of this book but the many references in the literature indicates that considerable attention has been given to compression testing of these materials in recent times. The current status of compression tests for composites is discussed by Welsh and Adams [7] and the problems of inconsistencies in results highlighted in a bulletin [8].

In the 1960s, Williams and Ford [9] evaluated the plain strain compression test, which was developed for metals, for determining total and residual deformation in plastics up to high levels of stress that could occur in some engineering applications. In this method compression is applied through the thickness of a strip test piece by two lubricated metal bars (**Figure 7.2**). Although it appears to be a useful practical test it has not been taken up by standards committees.

Also several decades ago, Warfield and co-workers [10] devised a compression test where the test piece was enclosed such that both compression and bulk modulus could be measured and used it on polystyrene.

A particular form of test for composite cylindrical structures is to load a ring diametrically in compression. Stiffness can be derived but such a test can be considered as a practical evaluation of integrity. The same approach can be taken to make *ad hoc* compression tests can conveniently on a number of products.

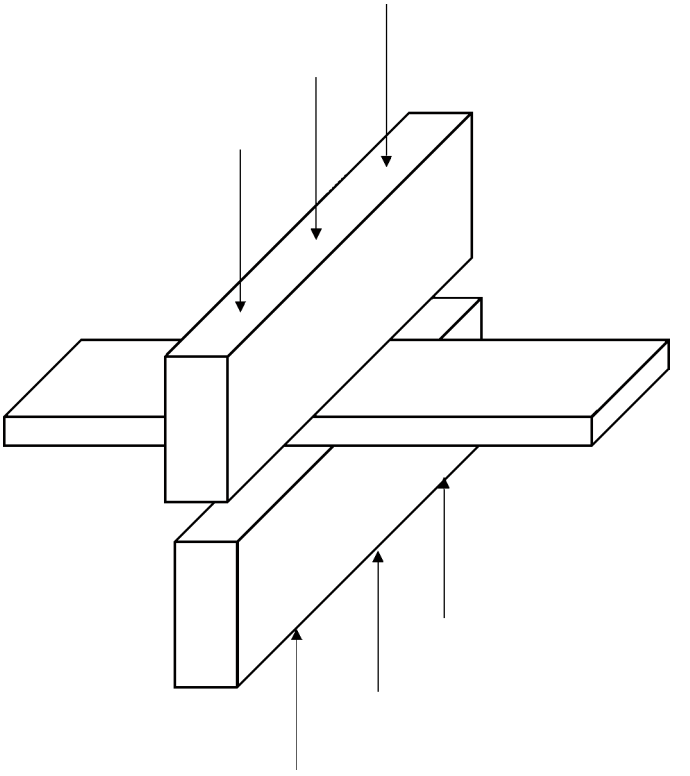

Figure 7.2 Plain strain compression test

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8 Shear Properties

8.1 Introduction

Tensile and compressive forces are normal to the plane on which they act but shear forces are parallel to the plane. Simple shear can be represented by planes sliding parallel to a given plane by an amount proportional to their distance from that plane. In **Figure 8.1**:

The shear stress is $\tau = \frac{F}{lxw}$ where w is the width (not shown).

The shear strain is $\gamma = \frac{x}{h}$

Pure shear is represented in **Figure 8.2** and is defined as a homogeneous strain in which one of the principal extensions is zero and the volume is unchanged. If the extension ratio $l_1 = \alpha$ while $l_2 = l$ then l_3 is $1/\alpha$.

Shear stresses are involved in many applications of plastics but very little shear testing is carried out except on fibre reinforced materials.

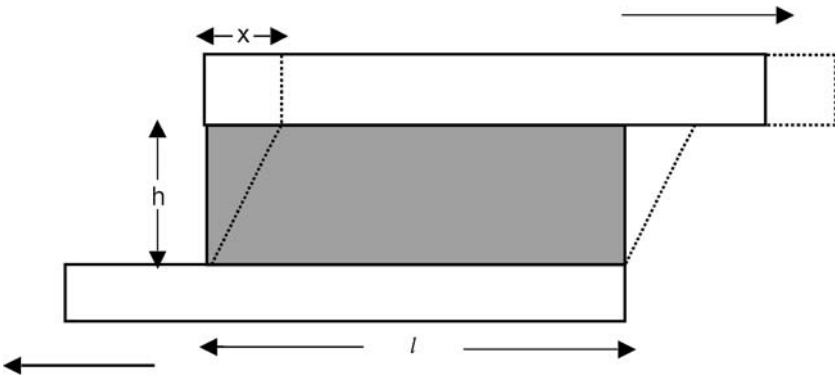

Figure 8.1 Simple shear

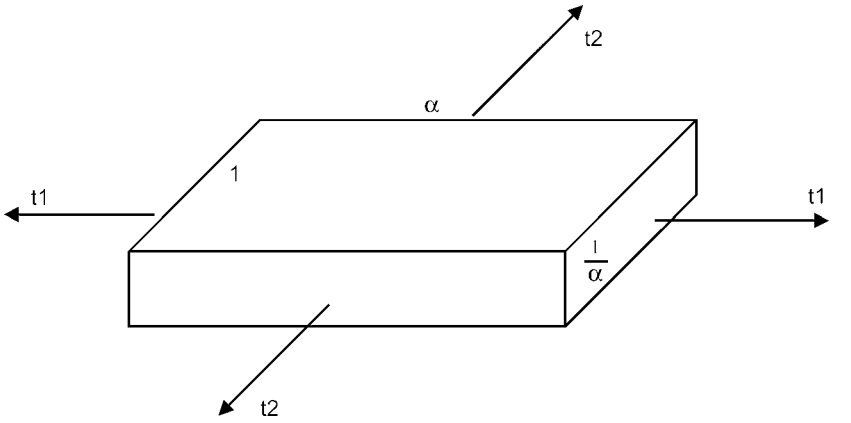

Figure 8.2 Pure Shear

Both shear modulus and the shear strength are of interest, but not necessarily measured in the same test. Shear modulus is usually measured at small strains where the stress-strain relationship is essentially linear. There are a number of loading systems which give rise to shear stresses including lap shear, punch shear, torsion and four point loading.

For rubbers and foams the most usual approach is based on the lap or sandwich, shear geometry [1]. This has not been standardised for quasi-static tests on plastics, probably because of the difficulty of bonding test pieces, although it has been used for dynamic tests. There can be one, two or four elements as shown in **Figure 8.3**. but clearly the four element design is the most stable. In this geometry, there will be increasing bending strains as the thickness of the elements is increased and the thickness/ area ratio is controlled to ensure that bending is insignificant Although in principle strength can be measured as well as modulus, adhesion to the metal plates is likely to be the limiting factor.

For strains where the shear stress-strain relationship is linear:

$$\frac{F}{A} = G\gamma$$

where F = force, A = area, G = shear modulus and γ = shear strain.

Punch shear geometry is relatively popular to measure shear strength of plastics. The approximation to pure shear conditions are achieved by a punch bearing on a sheet of material supported by a die. The smaller the difference between the internal diameter of the die and the external diameter of the punch the nearer the approximation.

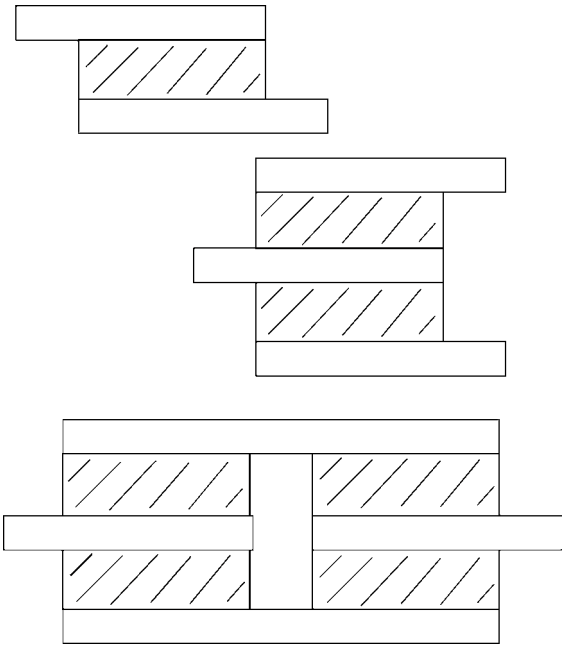

Figure 8.3 Lap and sandwich shear test pieces

Shear modulus can be measured in torsion, although in practice it is largely restricted to measuring stiffness of rubbers, flexible plastics and coated fabrics as a means of characterising low temperature performance. A strip geometry is used when force and deflection are related by:

$$\tau = kbt^3G\theta/l$$

where τ = applied torque, k = shape factor, b = width of test piece, t = test piece thickness, G = shear modulus, θ = angle of twist and l = effective length of test piece.

Stress strain relationships for other shear, shear/compression and torsional configurations can be found in Freakley and Payne [2] and Payne and Scott [3].

In bending or flexural tests (see Chapter 9) the objective is normally to choose a geometry that makes shear stresses negligible. Alternatively, the geometry can be chosen to make shear dominant and this is the object of the so called interlaminar shear strength test for fibre reinforced plastics. The span is reduced to six times the test piece thickness to encourage shear failure. Shear can also be induced in directionally reinforced materials by suitable arrangement of the orientation of the reinforcement relative to the direction of straining in a tensile test.

Shear tests with the sandwich type geometry, punch shear tests and tests where shear is induced from straining in tension are carried out using a universal test machine with appropriate jigs and grips to mount and strain the test piece. Strain can be measured with a transducer or by crosshead movement in a similar way to compression tests. Bending tests also use a tensile machine with a specially designed bending jig in accordance with the relevant standard. Particular equipment to apply a torsional strain is described in relevant standards but it is possible to adapt a tensile testing machine [4].

8.2 Standard Tests

The only ISO tests for shear of plastics in general are torsional methods [5, 6]. ISO 458-1 is not polymer specific and says in the scope that it is intended for measuring stiffness in torsion at various temperatures, particularly at temperatures below 0 °C. Most people think of it as a method for evaluating the effect of sub-ambient temperatures.

The apparatus, commonly known as Clash and Berg apparatus, is a somewhat crude mechanical device using weights and pulleys to apply a torque to a strip test piece. A wide tolerance on test piece thickness is given, between 1 and 5 mm, so that materials of different stiffness can be tested (more flexible materials use the higher thickness). The temperature is controlled by immersing the test piece and the grips into a suitable liquid contained in a Dewar flask. The flask is cooled with dry ice or by putting it into a freezer to below the minimum temperature of interest and then raising the temperature in steps by intermittent use of a heater.

After conditioning for 180 seconds, a torque is applied to give an angular deflection of between 10° and 100° for Method A and between 50° and 60° for Method B. The significance of two methods is not explained but it is known that the response is often not linear with strain, and Method B would seem to be an attempt to minimise this effect. In fact, a note does say that materials should only be compared if approximately the same angle of deflection was used. The angle of deflection is measured after an arbitrary time of 5 seconds, which is standardised to avoid effects of creep. Measurements are then made at successively higher temperatures as required. Torsional modulus is calculated using a modification of the equation given earlier.

ISO 458-2 is the same test applied to plasticised polyvinyl chloride (PVC). The angle of deflection is in this case limited to between 55° and 65° (so much for standardisation) and the temperatures corresponding to moduli of 300, 23 and 4 MPa obtained. The thickness of the test piece is changed for the different modulus levels.

BS 2782 Methods 340A&B [7, 8] cover the determination of punch shear strength for moulding material and for sheet material, respectively. In Method 340A the test pieces

are moulded discs 25.3 ± 0.1 mm in diameter and 1.6 ± 0.1 mm thick. Method B uses a rectangular bar test piece of length 32 mm and width 6.4 ± 0.2 mm at the thickness of the sheet under test, up to a maximum of 6.35 mm. If the sheet is more than 6.35 mm it is machined on one surface only to reduce the thickness to 6.10 ± 0.25 mm.

The test is carried out in compression by placing the test piece in a special bolster with a close fitting punch which bears against the test piece surface. The jig is placed in a universal testing machine and the load on the punch is increased so as to cause failure in the test piece within 15 to 45 seconds from first application of the load (see **Figure 8.4**). It should be noted that the diagrams defining the punch tool were amended in 1987.

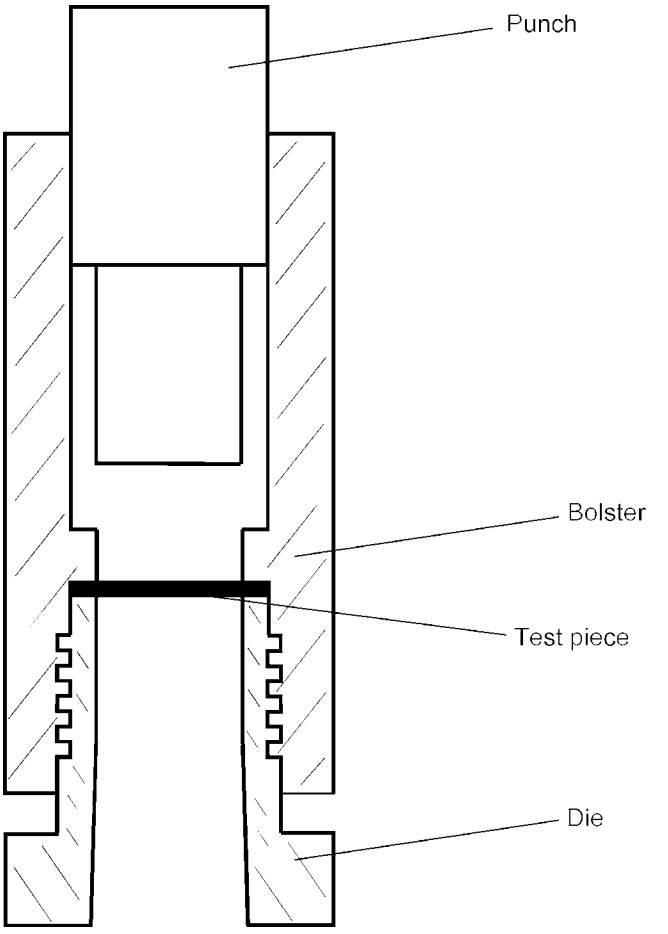

Figure 8.4 Arrangement for punch shear

The calculation of strength for Methods A and B is given by the expressions 8.1 and 8.2, respectively:

$$S = \frac{F}{\pi DT} \tag{8.1}$$

$$S = \frac{F}{2.096BT} \tag{8.2}$$

where S is the shear strength, F is the force at break, D is the diameter of the punch, B is the mean width of the test piece and T is the mean thickness of the test piece.

ASTM D732 [8] follows the same pattern as Method A, but uses a 50 mm diameter test piece and permits any thickness between 0.125 mm and 12.5 mm. Also, the test piece is drilled centrally to locate a guide pin on the punch. It warns that shear strength calculated using the sheared area should not be interpreted as indicating that shear strength is proportional to thickness.

8.3 Other Tests

Many references can be found to shear testing of fibre reinforced materials and there are several standards. The short beam interlaminar flexural test mentioned earlier is covered by ISO 14130 [9]. BS EN ISO 14130 is identical but, curiously, there is also another British method BS 2782 Method 341A [10].

A survey of standard methods for delamination resistance was made by Davies and co-workers [11] and Adams and Lewis consider the current status of composite material shear tests [12]. Aichele and Fischer describe a method for obtaining shear moduli of three dimensionally orthotropic laminates [13].

Hedner and co-workers [14] reference a number of geometries which have been used to produce shear data and describe a method which uses a geometry akin to lap shear. A square test piece has cut outs as shown in **Figure 8.5** and is clamped on the shaded portions and strained as indicated. The stress distribution is not even but they achieved useful results. They also tried a variation on this with small corner cut outs but, although it gave better stress distribution, it did not appear to offer improvements in determining shear modulus.

The Iosipescu test piece (**Figure 8.6**) is usually associated with testing fibre reinforced materials but in fact it was first developed for use with metals and can be used with

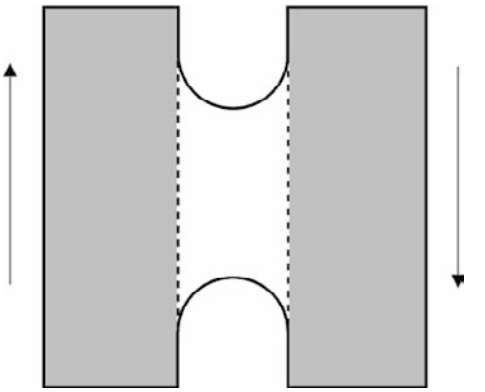

Figure 8.5 Shear Test Piece after Hedner [14]

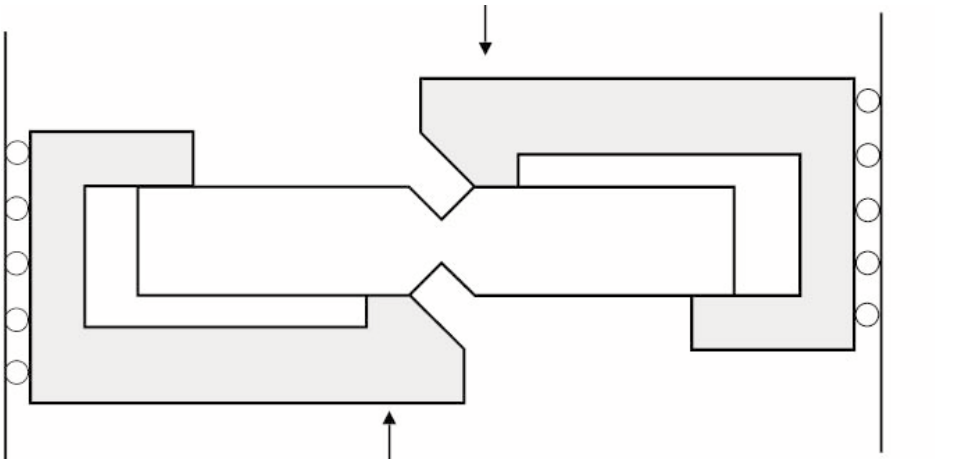

Figure 8.6 Loading arrangement for Iosipescu test piece

unreinforced plastics [15]. Perhaps surprisingly, the stress across the test piece between the notches is pure shear and is uniform. The shear stress is simply given by:

$$\tau = P/A$$

where P is the applied force and A the cross sectional area between the notches.

Strain is measured with a two element strain gauge bonded to one face of the test piece.

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9 Flexural Stress-Strain

9.1 General Considerations

The short-term flexural characteristics of a material are almost as commonly measured as the tensile properties considered in Chapter 6. This is not surprising considering the fact that most components are subject to a mixture of loading modes, and flexing or bending often occurs by intent or accident. Flexural tests also have the advantage that a strip test piece is easier to produce than a dumbbell and there are no gripping problems as can occur in tensile tests.

Flexural stress-strain characteristics are derived by monitoring both the force required to flex a material and the displacement that the material undergoes as a result of the applied force at a constant deformation rate. In flexure, there is a maximum tensile force on one side of the test piece graduating to a compressive force on the other. It should be noted, that the stress and strain that are calculated are the maximum outer fibre stresses and strains and that the stress calculations given later in the chapter are only valid up to a maximum fibre strain of 5%. Hence, although in principle the same parameters are measured as in a tensile test, they refer to the outer layer of the material rather than the bulk. Because plastics are seldom completely isotropic through the thickness, the results will only approximately equate to those from tensile tests.

The mode of loading can take one of three forms:

- Three point
- Four point
- Simple cantilever

By far the most common is three point loading. As the name implies, this mode of loading is achieved by applying the force to the specimen at three points, see **Figure 9.1**. The central loading point being equidistant from the outer two supporting points.

In practice the specimen usually sits on the two outer supporting rods and the force is applied through the central loading rod, which will have both a force transducer and some form of displacement measuring device attached.

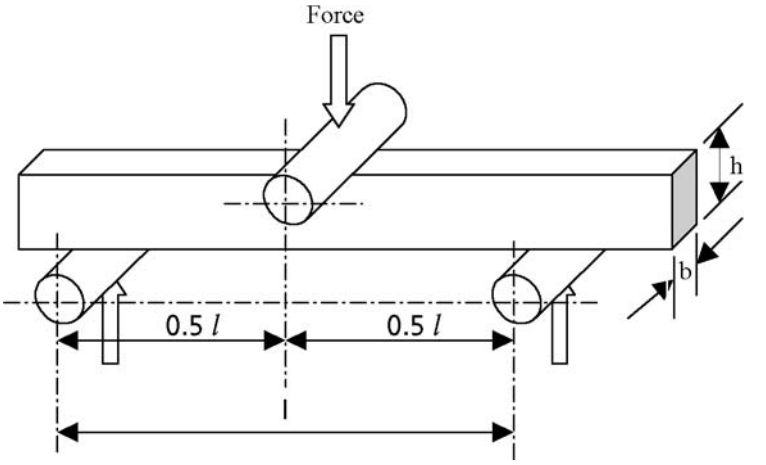

Figure 9.1

As with the case of tensile testing, to obtain any comparative data, the force and displacement has to be converted into stress-strain characteristics and to do this for rectangular bars the following equations are used:

Flexural stress (σ_f) = $3Fl/2bh^2$

Flexural strain (ε_f) = $6hs/l^2$

Where l = Support span - the length of the beam between the centres of the two outer supporting rods (mm)
h = The thickness of the beam (mm)
b = The width of the beam (mm)
F = Force (N)
s = Deflection of the specimen at mid span (mm)
σ_f = Flexural stress (N mm^{-2})
ε_f = Flexural strain

From these expressions modulus can be obtained from:

$$E_f = \frac{l^3}{4bh^3} slope$$

where *slope* is the slope of force-deflection curve between reference strains (0.05% and 0.25% in ISO 178 [1]).

A more accurate expression for the stress, which takes into account the horizontal component of the flexural moment, is given by:

$$\sigma_F = \frac{3Fl}{2bh^2}\left(1 + \frac{4s^2}{l^2}\right)$$

Since *s* is typically very much less than *L* the second term in brackets makes only a very small contribution to the stress and can be ignored. Other formulae have been proposed for more accurate calculation of stress, see for example the detailed consideration of bending by Heap and Norman [2].

For a circular rod, the expressions for stress and modulus are:

$$\sigma_F = \frac{8Fl}{\pi D^3}$$

$$E_f = \frac{4l^3}{3\pi D^4} slope$$

Where D is the diameter of the rod.

Less commonly, four point loading is used. Again, as the name implies, see **Figure 9.2**, this mode of loading is achieved by applying the force to the specimen at four points, usually with the loading span set to either one-third or one-half the support span. The advantage of four point loading is that the stress is uniformly distributed between the loading supports rather than being a maximum at the central loading point in three point bending.

In practice the specimen usually sits on the two outer loading rods and the force is applied through the two central loading rods, which will have both a force transducer and some form of displacement measuring device attached.

Relationships for the stress and strain in a rectangular bar subjected to 4-point bending depend on whether the total, inner or outer span is used, on how the measured deflection is defined and on whether F is the total force or that on one support. The equations below are as given in ASTM D6272 [3].

For a load span one-third of the support span ($3l_L = l$):

Flexural Stress (σ_f) = Fl/bh^2

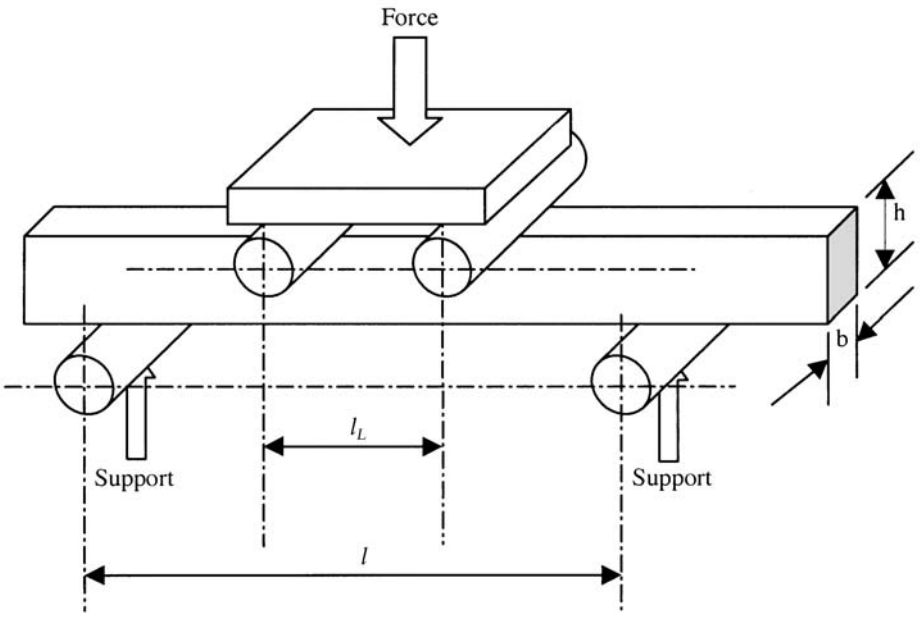

Figure 9.2

Flexural Strain (ε_f) = $4.7hs/l^2$

For a load span one-half of the support span ($2l_L = l$):

Flexural Stress (σ_f) = $3Fl/4bh^2$

Flexural Strain (ε_f) = $4.36hs/l^2$

Where l = Support span - the length of the beam between the centres of the two outer supporting rods (mm)

l_L = Loading span – the distance between the centres of the two loading rods (mm)

h = The thickness of the beam (mm)

b = The width of the beam (mm)

F = Force (N)

s = Deflection of the specimen at mid span (mm)

σ_f = Flexural stress (Nmm^{-2})

$\varepsilon_{f.}$ = Flexural strain

Heap and Norman used:

$$\varepsilon_f = 4hs/l_L^2$$

where s is defined as the deflection of the centre of the beam with respect to the inner supports.

A third type of loading is a simple cantilever with the test piece fixed at one end and loaded at the other. This was relatively popular at one time in the form of a simple test where the load was applied by hanging weights, but is now rarely seen. The stress and modulus for a rectangular beam are given by:

$$\sigma_f = 6Fl/bh^2$$

$$\varepsilon_f = 4Fl^3/bh^2s$$

9.2 Test Methods

9.2.1 Standard Methods

The international standard for flexural properties is ISO 178 [1] and the British and European standards are identical. Currently, ISO 178, only considers three point bending tests although four point bending is said to be under consideration for certain textile-fibre-reinforced plastics.

The corresponding ASTM standard is D790 [4] which follows very much the same lines as the ISO although it is not technically equivalent. One extra feature is an appendix which advises on dealing with toe compensation. This is where there is an artifact at the beginning of the stress-strain curve due to take up of slack in the test system. There is also a four point loading method given in ASTM D6272 [3] which uses a configuration with the loading span half of the support span. A further test is ASTM D747 [5] in which a strip of material is clamped in a vice and the load applied through a pivot point at the end of the vice, where the free length of the test piece starts. The greater the angle of bend, the lower the stiffness and hence modulus of the material. The test is better suited to estimating relative modulus than in making absolute determinations.

ISO 178 covers the following materials:

- Thermoplastic moulding and extrusion materials, including filled and reinforced compounds in addition to unfilled types; rigid thermoplastic sheets;

- Thermosetting materials, including filled and unreinforced compounds; thermosetting sheets, including laminates;

- Fibre reinforced thermoset and thermoplastic composites, incorporating unidirectional or non-unidirectional reinforcements such as mat, woven fabrics, woven rovings, chopped strands, combination and hybrid reinforcements, rovings and milled fibres; sheets made from pre-impregnated materials (prepregs);

- Thermotropic liquid crystal polymers.

The method is not normally suitable for use with rigid cellular materials and sandwich structures containing cellular material.

Most of the flexural definitions covered in the standard are analogous to the tensile test definitions set out in ISO 527-1 General Principles [6]. There are however two terms which are not covered and these are presented next:

Conventional deflection, s_C: Deflection equal to 1.5 times the thickness, h, of the specimen. It is expressed in millimetres (mm).

Flexural stress at conventional deflection, σ_{fc}: Flexural stress at the conventional deflection s_C. Using the span l = 16h, the conventional deflection corresponds to a flexural strain of 3.5%.

The term stress at the conventional deflection is unique to the flexural test. Generally with ductile materials the test piece does not reach a point of fracture, it simply keeps bending until eventually is slips from the outer supports. The conventional deflection is defined as 1.5 times the test piece thickness, which for the standard span of 16 times the thickness, equates to a strain of 3.5%. The stress at this point forms a useful, if arbitrary, characteristic for ductile materials which occurs before the peak in the force-deflection curve is reached.

9.2.2 Test Apparatus

As in the case for tensile testing, the main apparatus is a test machine complying with ISO 5893 [7] with force measurement to grade A. The requirements for measurement of force and deflection are given as within 1% of full scale. One can assume that grade A for force is intended but the requirement for deflection is badly worded.

Table 9.1 Recommended values for the speed of testing	
Speed mm/min	**Tolerance %**
1 [1]	± 20
2	± 20
5	± 20
10	± 20
20	± 10
50	± 10
100	± 10
200	± 10
500	± 10
[1] The lowest speed is used for specimens with thicknesses between 1 mm and 3.5 mm	

The test machine should be capable of maintaining the speed of testing as defined in **Table 9.1**.

A jig is required to support and load the test piece and the requirements for the supports and striking edge are arranged as in **Figure 9.3**. The radius R_1 of the striking edge and the radius R_2 of the supports are as follows:

$R_1 = 5.0 \text{ mm} \pm 0.1 \text{ mm}$

$R_2 = 2.0 \text{ mm} \pm 0.2 \text{ mm}$ for thicknesses of test specimen $\leq$ 3 mm, and

$R_2 = 5.0 \text{ mm} \pm 0.1 \text{ mm}$ for thicknesses of the test specimen > 3 mm

The span L should be adjustable.

In addition, a suitable micrometer and vernier calliper or equivalent is needed to measure the test piece dimensions and the span.

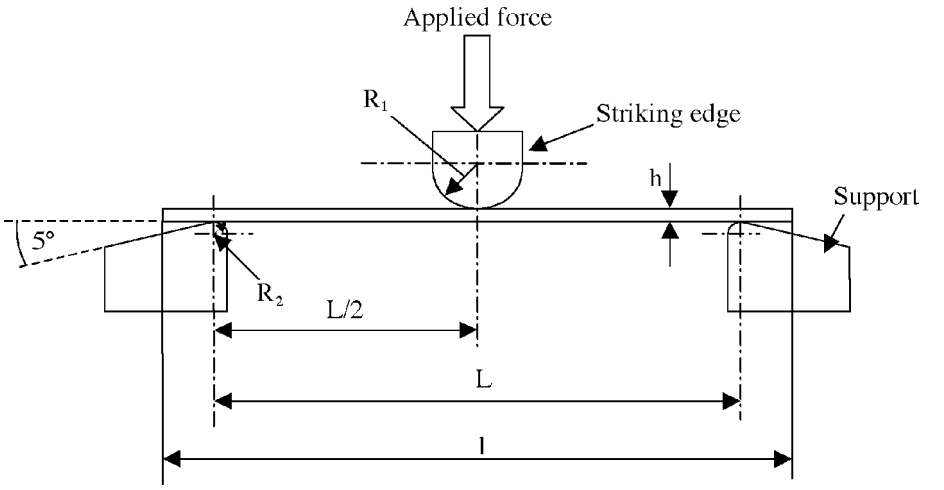

Figure 9.3 ISO 178 test arrangement

9.2.3 Test Pieces

A minimum of five test pieces are required by ISO 178 for each direction tested. The preferred test piece is a strip with the following dimensions:

Length: $l = 80.0 \pm 2.0$
Width: $b = 10.0 \pm 0.2$
Thickness: $h = 4.0 \pm 0.2$

and

1) The thickness of the central third of the specimen length shall not deviate by more than 2%.

2) The width of the central third of the specimen length shall not deviate by more than 3%.

3) The specimen must have a rectangular cross section with no rounded edges.

If the preferred test piece cannot be used then the following limits apply:

1) The length to thickness ratio shall be 20 ($l/h = 20 \pm 1$)

2) The width of the specimen shall be as defined in **Table 9.2**.

Table 9.2 Values for the width, b, in relation to the thickness h		
Nominal thickness, h	**Width b ± 0.5 [1] (mm)**	
	Moulding and extrusion compounds, thermoplastic and thermosetting sheets	**Textile and long-fibre-reinforced plastics materials**
$1 < h \leq 3$	25.0	15.0
$3 < h \leq 5$	10.0	15.0
$5 < h \leq 10$	15.0	15.0
$10 < h \leq 20$	20.0	30.0
$20 < h \leq 35$	35.0	50.0
$35 < h \leq 50$	50.0	80.0
[1] For materials with very coarse fillers, the minimum width shall be 20 mm to 50 mm.		

The span is normally set to 16 ± 1 times the thickness although for soft thermoplastics (to avoid indentation from the supports) and certain fibre-reinforced materials (to avoid delamination) higher span to thickness ratios may be needed. For very thin test pieces a lower ratio may be needed in order to keep the forces generated within the measuring range of the equipment.

9.2.4 Procedure

ISO 178 does not give specific recommendations for conditioning but reference is made to ISO 291 [8].

In the absence of a material specification, the test speed is selected to give a strain rate as near as possible to 1% per minute which for the standard test piece is 2 mm/min. The higher speeds given in **Table 9.1** are unlikely to be used.

The force–deflection curve is normally recorded automatically. The flexural stress is calculated, but one has to guess that this can be at break, conventional deflection or whatever. Modulus is defined in a similar way to tensile tests with strains of 0.05% and 0.25% again taken as the limiting values between which it is determined. This places very severe constraints on the accuracy of the test equipment and the test piece itself. For the standard test piece, 0.05% strain corresponds to a deflection of the outer surface of only 0.08 mm from its starting point. This does not make much allowance for any backlash

in the test jig, lack of flatness in the test piece, or lack of alignment in any of the three loading bars. Although not specified, modulus could be calculated at other strains, or secant modulus measured.

References

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3. ASTM D6272. *Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials by Four-Point Bending*, 2000.

4. ASTM D790. *Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials*, 2000.

5. ASTM D747. *Standard Test Methods for Apparent Bending Modulus of Plastics by Means of a Cantilever Beam*, 1999.

6. ISO 527-1. *Plastics – Determination of Tensile Properties – General Principles*, 1993.

7. ISO 5893. *Rubber and Plastics Test Equipment – Tensile, Flexural and Compression Types (Constant Rate of Traverse) – Description*, 1993.

8. BS EN ISO 291. *Plastics – Standard Atmospheres for Conditioning and Testing*, 1997.

10 Impact Strength

10.1 General Considerations

10.1.1 Introduction

The decidedly *ad hoc* field of impact testing has received considerable attention in official standards, materials data sheets and the literature because the impact properties of plastics materials are directly related to the overall toughness of the material. The concept of 'toughness' is one that most people can readily appreciate and a broadly accepted definition is the work done in breaking a test piece or object.

The one advantage impact tests offer is a ready measure of the actual energy required to break the test piece. This information can of course be calculated from stress-strain diagrams in, say, tensile (Chapter 6) or flexural (Chapter 9) tests. In the past this could only be achieved through some considerable effort, but with the widespread use of computers to control testing and to process test data, it has become as easy to derive work energy from these tests as from the impact test. While such data are quite useful for describing the behaviour of plane-faced objects, the area under a conventional tensile or bending test curve is of limited value because in practice one is frequently interested in toughness under conditions of rapid deformation. There is, however, much technical complexity in carrying out tensile tests and other modes of deformation at high speed. Thus, the concept of impact resistance or 'strength' and the introduction of impact or shock resistance tests, and its assessment follow naturally from these concerns.

It should be remembered, however, that the result of an impact test is basically no more than one point on the general curve of studying strength properties as a function of speed of testing. There are a large number of standardised impact tests, which is in stark contrast to the scarcely standardised subject of high-speed mechanical testing using tensile, flexural, compressive or shear geometries.

10.1.2 Modes of Failure

Energy is required both to create a crack and to allow this crack to be propagated through the material. The energy to initiate a crack is called the crack initiation energy. If the

available energy in the system undergoing impact exceeds the crack initiation energy, the crack will continue to propagate and complete failure will occur if the system has sufficient energy to also exceed the crack propagation energy. Thus, both crack initiation and crack propagation contribute to the measured impact energy. Vincent [1] has identified four basic types of failure that are encountered under impact and the result of an impact test may result in different types of failure. It is important in interpreting the results of a test that test pieces exhibiting different kinds of failure are not pooled together.

- Brittle fracture is where the part fractures extensively without yielding and typically has sharp 'glassy' edges. General-purpose PS typically exhibits this type of failure under impact conditions.

- Ductile failure is where there is a definite yielding of material, often indicated by stress whitening, along with cracking. Polyolefins are generally considered to be ductile materials.

- Yielding is where the part exhibits obvious and permanent deformation and stress whitening but no cracking takes place.

- Slight cracking is where the part shows evidence of some cracking and yielding but without losing its shape or integrity.

The distinction between the four types of failures is not always very clear and some overlapping is quite possible. For particular standardised test geometries, defined modes of failure may be given and test pieces must be assigned to one of these categories. For Charpy and Izod tests, for example, the following definitions along with their letter abbreviations need to be adopted:

C complete break; a break in which the specimen separates into two or more pieces.

H hinge break; an incomplete break such that both parts of the specimen are held together only by a thin peripheral layer in the form of a hinge having no residual stiffness.

P partial break; an incomplete break that does not meet the definition for a hinge break.

NB non-break; in the case where there is no break, and the specimen is only bent, possibly combined with stress whitening.

10.1.3 Factors Affecting the Impact Strength

10.1.3.1 Rate of Loading

Because plastic materials are viscoelastic, the speed at which the test piece or part is struck has a significant effect on the behaviour of the polymer under impact loading. At

low rates of impact, relatively stiff materials can still have good impact strength, but at high enough rates of impact, even rubbery materials will exhibit brittle failure. All polymer materials seem to have a critical velocity above which they behave as glassy, brittle materials. This has important consequences for designing with plastics. If in a particular application the product will 'see' impact speeds of 50 m/s then it is dangerous to base a material selection on normal impact tests like Charpy or Falling Weight because the impact velocities are an order of magnitude lower and a material that behaves in a ductile manner at these speeds may become brittle at the application speed. After using these tests for screening potentially useful candidate materials a more specialised and probably *ad hoc* test, perhaps involving firing a projectile at the materials, will be needed. Otterson and co-workers [2] report the effect of testing speed on blends of Nylon 6 and acrylonitrile-butadiene-styrene (ABS) for crack growth resistance while Douglas and Leevers [3] consider the effect of test speed and geometry on pipe grade materials using a recently developed dynamic and fracture model. The effect of deformation rate on fracture toughness values has been considered by Pinardag and co-workers [4].

10.1.3.2 Temperature

Again, the viscoelastic nature of plastics makes the effect of temperature much more significant than it is for materials like ceramics and metals. Decreasing the temperature tends to promote the onset of brittle failure. It is therefore important to take account of the temperature range that the article might see in service and to conduct impact tests over that temperature range as far as practically possible. Note that increasing temperature has the opposite effect of increasing speed and so there is not a single temperature at which brittleness occurs, but a locus of temperature/speed values where the transition from ductile to brittle behaviour takes place. Every polymer has its own characteristic locus. Weier and Hemenway [5] consider a number of factors affecting the process of energy absorption during impact testing with temperature being one of these factors while Takeda and co-workers [6] focus on the effect of temperature and water content on Nylon 6 composites.

10.1.3.3 Notch Sensitivity

A sharp corner in a fabricated part or a notch in a test specimen can dramatically lower the impact strength of the material. This is because a notch creates a localised stress concentration where the true stress can be many times higher than the bulk stress being imposed on the test piece or object as a whole. Hence failure under impact loading is promoted. All plastics are notch-sensitive, but the notch sensitivity varies with the type of plastic being considered. Both notch depth and notch radius have an effect on the

impact behaviour of materials. A larger radius of curvature at the base of the notch will have a lower stress concentration and therefore will tend to give a higher impact energy for the material in question. It can be seen from this that when designing a plastic part, notches, sharp corners, and other factors that act as stress concentrators should be avoided. The science of fracture mechanics seeks to quantify these types of effects and the interested reader is referred to [7] for an overview of the subject, [8] provides a more detailed development of the mathematical concepts and [9-17] give information on particular aspects of the subject.

10.1.3.4 Fillers

The inherent impact properties of a polymer may be modified simply by adding some form of filler. Polymeric impact modifiers may be incorporated to act as barriers or crack blunting regions to the advancing crack front. A good example of this is the addition of polybutadiene (PB) rubber to styrene-acrylonitrile (SAN) plastics to produce ABS. In other instances lower molecular weight plasticisers are added (for example to polyvinyl chloride (PVC)) to improve the impact behaviour. In the case of Nylons, increasing the moisture content significantly improves the toughness of the plastic. Very dry Nylons like Nylon 6 and 6-6 are quite brittle and sufficient time must be allowed for them to gain atmospheric moisture prior to testing if the 'equilibrium' impact strength is to be measured. The down-side of plasticisation is that it results in loss of rigidity. Another way to improve the impact strength may be to use fibrous fillers that appear to act as stress transfer agents. Good coupling between the fibre and the polymer matrix is necessary for the effect to take place. Other fillers may be used simply to make the product cheaper and typically these result in some impairment of impact strength compared to the base resin. Recent papers which consider some of the aspects of fillers on impact properties include calcium carbonate [18, 19], pigments [20], glass beads [21], glass fibres [22, 23] and talc [24] on polypropylene (PP), mica on polybutylene terephthalate (PBTP) and Nylon [25] and alumina particles in epoxy resins [26]. Effects of moisture on Nylon are given in [6] and on polycarbonate in [27].

10.1.3.5 Orientation

Polymer molecules are long 'spaghetti-like' structures and as such their orientation within an object is highly dependent on the flow patterns of the molten polymer during the moulding phase. The properties of the molecules themselves are also highly directional: properties along the main backbone chain are quite different to those along side chains and between the molecules. For this reason, the manner in which the polymer molecules are oriented in a part will have a major effect on the impact behaviour of the polymer - the impact strength

is always higher in the direction of flow. Molecular orientation is deliberately introduced by drawing films and fibres, for example, to give extra strength and toughness along the stretch direction compared to the isotropic material. However, at right angles to the flow or drawing direction the impact properties can be significantly reduced as it is predominantly inter-molecular forces rather than intra-molecular forces that are involved. Such directional orientation of polymer molecules can result in dramatically different impact properties in different areas or directions of a moulded part. Impact stresses are usually multiaxial and so tend to automatically 'find' the weakest direction in the moulding.

10.1.3.6 Processing Conditions

Processing conditions also play a key role in determining the impact behaviour of a material. Inappropriate processing conditions can cause the material to fail to attain its inherent toughness. Poor processing conditions may create voids, for example, that will act as stress concentrators; high processing temperatures may cause thermal degradation and, therefore, reduce the impact strength; inadequate drying of plastics that have a tendency to absorb moisture can have a dramatic effect in reducing the resulting impact strength. Improper mould design may create a weak weld line that will almost certainly reduce the overall impact strength. Test pieces taken from compression-moulded plaques usually show a lower impact resistance than test pieces that are directly injection-moulded and test pieces with moulded notches give higher impact strengths than test pieces with machined notches [28]. Some of the effects of annealing can be found in [29], and of weld lines in [30, 31]. Rotational moulding is discussed in [32] and the processing variables of reaction injection moulding (RIM) in [33]. Processing temperature effects are given in [34] and the issue of reprocessing/recycling is covered in [35]

10.1.3.7 Molecular Weight and Degree of Crystallinity

All other things being equal, a reduction in the average molecular weight reduces the impact strength and *vice versa*, although above a certain critical molecular weight the effect is relatively slight. The papers by Ibhadon [29] and Schriver and co-workers [35] include the influence of molecular weight on impact properties.

For semi-crystalline plastics, like polyolefins, increasing the percentage crystallinity decreases the impact resistance and increases the probability of brittle failure, so the thermal history of the product will influence the outcome of an impact event. Material that has been quenched from the melt will be tougher than the same material that has been allowed to cool slowly. The crystallinity and molecular weight effects for polyether ether ketone (PEEK) is dealt with in a paper by Chivers and Moore [36].

10.1.3.8 Impact Methodology

The geometry of the test piece and the manner in which the test piece is struck can significantly alter the impact results. Thus a pendulum impact test will produce a different result from the one produced by falling-weight. What is more surprising to many people who are not familiar with the complexity of plastics materials is that different types of pendulum test, e.g., Charpy and Izod, also produce different results and there is no simple correlation between them. Although these pendulum tests 'normalise' the impact energy for a given test piece by dividing the energy taken out of the pendulum by the cross sectional area behind the notch, it is found that different sized test pieces tend to give different impact data. All of this makes the application of impact data into design calculations in a direct, quantitative, way fraught with difficulty. It also means that even when simply comparing databases for materials we must be sure we are comparing like with like or false conclusions will inevitably follow. Mention may be made in this context of the use of ISO standard protocols for the generation of single point data [37] and multipoint data [38] and of the Campus [39] system of representing comparable data from different materials producers. A comparison between pendulum devices and drop weight impact tests for long glass fibre reinforced PP has been made by Paakkonen and co-workers [23] and the effect of geometry on pipe grade plastics by Douglas and Leevers [3]. Rogers and Plumtree [40] compare the Izod and Charpy tests for polystyrene (PS).

10.2 Specific Tests

As has been touched on previously, the impact testing of plastics tends to fall into two basic categories: the pendulum tests and the falling weight tests. Each of these may then be further sub-divided into more specific classes.

10.2.1 Pendulum Methods

10.2.1.1 Charpy Test

The Charpy test is detailed in BS EN ISO 179-1 [41], and the related ASTM D6110 [42]. The following discussion is based on the ISO test method, with comments on the ASTM variant at the end.

In the Charpy test the test piece is supported as a horizontal beam and is broken by a single swing of a pendulum, the line of impact being midway between the supports. Both notched and unnotched test pieces may be tested and the test piece may be oriented in the edgewise or the flatwise direction. The two geometries are illustrated in **Figure 10.1**.

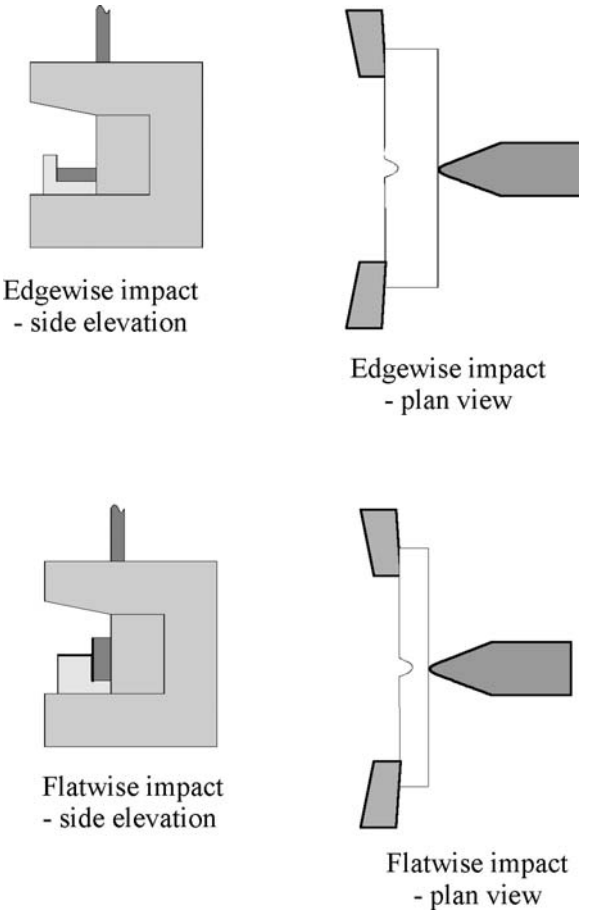

Figure 10.1

The directionality of the test is best understood in relation to the test piece dimensions themselves. The standard test bar is 80 x 10 x 4 mm which can be cut from the centre parallel portion of the multipurpose test specimen [43]. In the flatwise test the direction of the pendulum at impact is in the 4 mm direction of the test piece so that bending takes place over the 80 x 10 mm surface. In the edgewise test it is the 80 x 4 mm plane that is bent and the pendulum travels in the direction of the 10 mm dimension. The edgewise test is now the preferred form of geometry for most testing purposes. In former times it was the flatwise test that was typically used and the edgewise test was reserved for investigating the effect of fibre-reinforcements on impact strength. Now the flatwise test is reserved for investigating surface effects such as might occur when the material is weathered by UV light or exposed to chemicals.

For laminated test pieces tests may be performed both flatwise or edgewise and for each of these there exists the possibility of having the laminations parallel or normal to the direction of blow. These variations are illustrated in **Figure 10.2**. All of these are permitted and a suitable coding scheme is defined to enable the options chosen in a given test to be defined very succinctly.

The test can be performed using either unnotched or notched test pieces, although the notched test is the more common. Three types of notches are standardised (**Figure 10.3**), the preferred one having a radius at the notch base of 0.25 mm (the type A notch). A blunt 1.0 mm (type B notch) and a very sharp 0.1 mm (type C) notch are also covered. Notches of different base radius are useful for more extensive characterisation of plastics than a simple quality test or data sheet entry, in that they enable an estimate of the notch sensitivity of the plastic to be investigated, as was mentioned in 10.1.3.3. The flatwise test can also be performed notched or unnotched, except here the notched test has two notches machined across the 4 mm direction and directly opposite each other to give a 6 mm width to the test piece between the notches. All three types of notch may be used in the edgewise test.

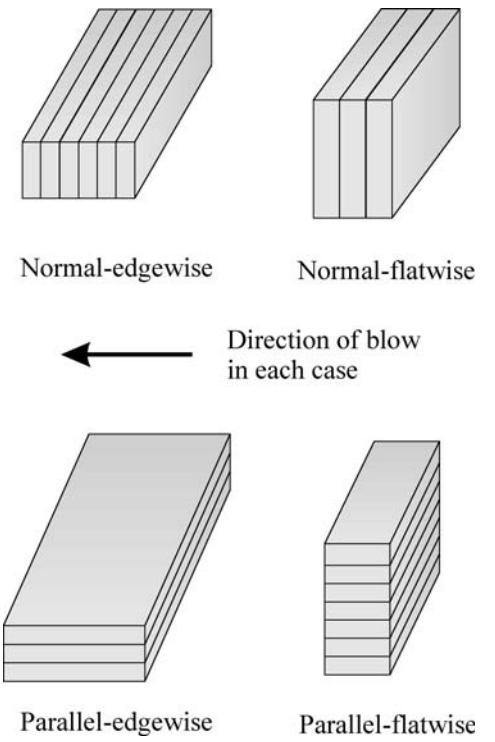

Figure 10.2

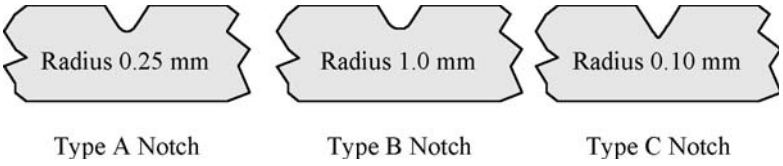

Figure 10.3

The standard test piece is suitable for general purpose testing, although where sheet material is to be tested it is permissible to test the full thickness of the sheet up to a maximum of 10.2 mm. Above this the sheet should be machined on one surface to reduce the thickness to 10 mm. Where the sheet is reinforced in some way the reinforcement must be regularly distributed and be of only one type. Thin samples are not suited to this test as buckling of the test piece can occur when tested edgewise or bending without failure when tested flatwise. For long-fibre reinforced plastics alternative geometries are permitted that have no specified specimen sizes. Instead it is the span to thickness ratio that is the controlling parameter. For type 2 test pieces the L/h ratio is 20 and for type 3 a ratio of 6 is preferred, however where thinner sheet materials are being tested and the apparatus does not allow such a small ratio to be accommodated, then a ratio of 8 is permitted. The test piece width for a flatwise test is either 10 mm or, for large stitch or irregularly manufactured structures this is increased to 15 mm. When an edgewise test is performed the dimension perpendicular to the direction of impact is that of the sheet from which the test piece has been machined.

The ISO standard covers two pendulum lengths, giving different velocities at the point of impact. The one most frequently used has an impact velocity of 2.9 m/s and five pendulums having energies of 0.5, 1.0, 2.0, 4.0 and 5.0 J are specified. The larger machine, having an impact velocity of 3.8 m/s, has pendulums of energy 7.5, 15.0, 25.0 and 50.0 J.

The impact strength is the energy removed from the pendulum as a result of work done in breaking the test piece divided by the cross sectional area of the test piece in the direction of swing. In fact, because the test piece is bending during the impact event, there is a deformation volume rather than simply an area and so test pieces of different size do not give results which are proportional to the cross-sectional area, but rather to some indeterminate volume. For this reason results obtained from test pieces of different size cannot be compared.

The ASTM D6110 [42] test follows the same principles, but differs in detail, as the first note of the standard points out. The preferred test specimen dimensions are based on imperial units, but unfortunately the current version of the standard contains an error such that the Figure referred to as giving the test piece dimensions actually gives the geometry of the anvil used for the micrometer. It may, however, be inferred that the preferred test piece is probably

127 mm long, by 12.7 mm wide by a thickness between 12.7 and 3 mm. The span between the supports is 95.3 mm compared to the ISO standard of 62 mm. The details of the apparatus used are the same as for the Izod test which is covered in the Section 10.2.1.2. Unlike the ISO test, the preferred form of expressing the result is different being based on the energy normalised with respect to the length of the notch only, and not on the area behind the notch. The alternative normalisation with respect to area is also now permitted. This only serves to add to the difficulty in making comparisons between data obtained by the ASTM standard, with its different test piece sizes and impact conditions, to that of the ISO standard.

BS EN ISO 179-2 [44] covers the instrumentation of the Charpy pendulum so that force-time (and by integration, force-deflection) curves can be obtained. This allows for a fuller characterisation of the impact behaviour of the plastic than can be derived only from the energy to break of the typical test. Otherwise the test procedure follows much the same details as for the non-instrumented version, the same test piece sizes being used and the 2.9 m/s pendulum being the preferred one. There has been an instrumented version of the falling weight impact test (see later in this section) for several years and the same principles apply to both.

Nakamura and co-workers [45] have used the instrumented Charpy test to examine the effect of silica fillers on epoxy resins while Wang and co-workers [46] have used the same technique for examining RIM parts. Trantina and Oehler [47] discuss the application of Charpy (and Izod) tests to the prediction of impact resistance for use in design calculations. Sharpe and Boehme [48] have used a small Charpy test to investigate dynamic fracture toughness measurements.

10.2.1.2 Izod Test

The Izod test is notionally very similar to the Charpy test, except that the test piece is clamped at one end just below the notch, or the centre of the specimen if it is unnotched, and struck by a pendulum close to the other end. It is therefore a cantilever bending test (**Figure 10.4**). Traditionally the Izod test has been more favoured in North America, while the Charpy test has been more popular in Europe. The test details are given in BS EN ISO 180 [49] and ASTM D256 Method A [50] (also methods C, D and E).

Considering the ISO standard first, the standard test piece is the ubiquitous 80 x 10 x 4 mm test piece taken from the multipurpose test piece [43] so widely used in ISO. Three variants are permitted: unnotched, notched with a 0.25 mm radius notch (type A) or notched with a 1.0 mm radius notch (type B). These match the same conditions as for Charpy, but the type C notch is undefined for Izod. The test is almost always carried out edgewise, although where laminated plastics are to be tested it is possible to test flatwise as well and using the same parallel or normal arrangements as for Charpy.

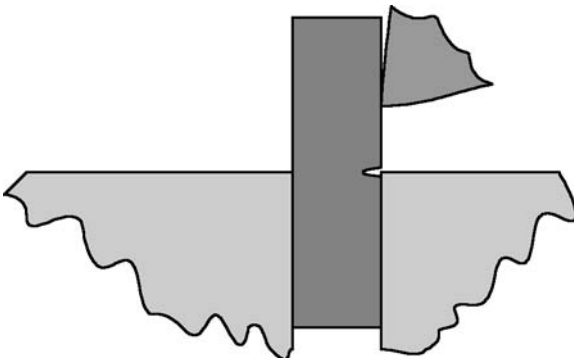

Figure 10.4

Unlike the Charpy test, the notched Izod is capable of being tested either with the notch on the same side as the point of impact, which is the normal way round, or on the opposite side when it is called the reverse notched test. Thus, in the normal test the side containing the notch is placed under tension and the notch fulfils its purpose as a stress concentrator. In the case of the reverse notch it is the unnotched face which is under tension and no stress concentration occurs; in fact the notch is placed under a compressive deformation. This arrangement is possible in the Izod because the pendulum strikes the test piece at a point remote from the notch and the advantage of having the reverse notch is that the test piece is otherwise identical. For the Charpy test the cross section of the unnotched test piece must be greater than the notched test piece.

The impact velocity for the test is 3.5 m/s and pendulums of energy 1.0, 2.7, 5.5, 11.0, and 22.0 J are used. As for the Charpy test, the energy absorbed by the impact should be between 10% and 80% of the capacity of the pendulum.

Certain plastics can give results which vary according to clamping pressure, a problem from which the Charpy test is free, and the standard recommends that when testing such materials some means of standardising and recording the clamping pressure should be used. However, it gives no advice on which plastics are so affected nor on how to determine whether the effect is significant or not.

The test is often applied to plastics at sub-ambient temperatures but is far from ideal for this. Again, the Charpy test is preferred. There are serious practical problems in carrying out the test with the apparatus itself at the low test temperature due to icing of the bearings, etc. It is therefore common practice to soak the test piece at the test temperature and then quickly remove it and test it. However, the test piece must be clamped into a large metal heat source, the clamping vice, at a point adjacent to the critical notch region

where the bending takes place. The actual test temperature is therefore quite indeterminate and likely to be variable from test piece to test piece.

The ASTM test follows the same principles, but, as for the Charpy test, differs in certain details. Again, the test is based on imperial units with the preferred impact resistance characterised by the length of the notch rather than the area behind the notch. The details of the apparatus itself mirror very closely the requirements of the ISO test method which was largely derived from the ASTM standard. Method A covers the normal test procedures which are applied to materials having an impact resistance in excess of 27 J/m. For lower values than this, Method C is applied which attempts to make a correction for the energy required to 'toss' the test piece. This involves carrying out a secondary test on the broken test piece, wherein the halves of the test piece are reassembled and the energy value obtained when this broken test piece is impacted is taken to be the energy absorbed in accelerating the initially stationary test piece. Since this energy is not due to the impact event as such, it is then subtracted from the apparent energy obtained during the first impact event, when the test piece was unbroken. Objections to the scientific principles behind this idea can be raised, and it has never found acceptance within the ISO community.

Method D deals with the estimation of notch sensitivity by having the test carried out at two notch radii, 0.25 mm and 1.0 mm. The ratio of the difference in the two energy values to the difference in notch radii is then taken as the index of notch sensitivity. Where the 1.0 mm radius leads to test pieces which do not break, a 0.5 mm radius notch may be substituted.

Method E covers the reversed notch test and although this is intended to represent an unnotched test piece, the standard warns that this method may not give identical results to a completely unnotched test piece. Genuinely unnotched test pieces are covered in the method given in ASTM D4812 [51] which is stated to be particularly suitable for testing reinforced materials, where a notch may mask the effects of orientation.

ASTM D4508 [52] is an Izod-like cantilever beam test but using a small test piece, 19.05 mm long by 12.7 mm wide and 1.02 to 3.18 mm thick (1.8 mm is preferred). It appears to be particularly favoured for assessing the effect of weathering on the impact resistance of plastics and for testing test pieces taken from finished products, where its small size is a significant benefit. There is no near-equivalent in ISO to this test.

Fu and co-workers [53] have investigated the toughening of polyethylene by calcium carbonate using the Izod test while Grocela and Nauman [54] have tried to derive quantitative models for Izod to predict strength for impact modified PS. The Izod test has been used to investigate the toughening mechanism of low molecular weight PB in PS [55, 56]. Weier and Hemenway [5] describe the use of the instrumented Izod test on PVC/acrylic composites.

10.2.1.3 Tensile Test

Both the previous two methods require the test piece to be sufficiently rigid for buckling of the specimen under test to be negligible. For thinner section materials and for those exhibiting a high elongation before fracture, the tensile impact test may be the only viable pendulum method. The test is standardised in BS EN ISO 8256 [57] and ASTM D1822 [58].

There are two basic types of tensile impact test: the specimen-in-bed type (illustrated schematically in **Figure 10.5**) and the specimen-in-head type. Method A of ISO 8256 covers the first of these and Method B the second. Two pendulum lengths are given in the standard, one of which gives an impact velocity of 2.8 m/s and the other of 3.7 m/s. The former is applied to pendulums having an energy of 2 and 4 joules, while the latter is applied to pendulums having an energy of 7.5, 15, 25 or 50 joules.

For Method A the test piece is clamped into a suitable holder mounted onto the bed of the apparatus. One end of the holder is rigidly mounted on the bed and the other, the cross-head, is free to move along the bed. The test piece forms a bridge between them. A pendulum is released and at the bottom of its swing it makes contact with the arms of the cross-head. Kinetic energy is transferred to the test piece which extends to rupture and the absorbed energy is determined from the height of swing of the pendulum. However, some energy is also expended in tossing the cross-head and so a correction must be

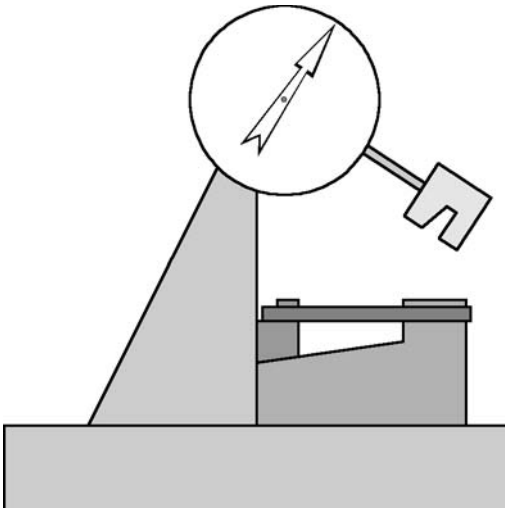

Figure 10.5

applied for this. The correction is a constant for a given pendulum and cross-head and can be determined from the equation:

$$E_q = \frac{E_{\max}\mu(3+\mu)}{2(1+\mu)}$$

where $\mu = \frac{1}{4}\frac{m_{cr}}{E_{\max}}\left(\frac{gT}{\pi}\right)^2(1-\cos\alpha)$

E_q is the energy correction due to the plastic deformation and the kinetic energy of the crosshead;

E_{max} is the maximum impact energy of the pendulum;

m_{cr} is the mass of the crosshead;

g is the acceleration due to gravity;

T is the period of the pendulum;

α is the angle between the positions of the maximum and minimum height of the pendulum.

The desired energy to rupture the test piece is then simply the difference between the uncorrected energy read from the maximum swing of the pendulum after impact and the above correction energy. As for the Charpy and Izod tests the result is normalised with respect to the area of the test piece cross-section, although unlike the other pendulum impact tests, there are several types of test piece that are used (see **Figure 10.6**).

For Method B the test piece is clamped into the compound head of the pendulum which is released from its raised position. As it reaches the lowest point of its swing the rear of the pendulum strikes rigid supports on the frame of the apparatus and is arrested. The front of the pendulum continues its swing, extending and rupturing the test piece. As for Method A, corrections must be applied to the energy read from the swing of the pendulum to compensate for the crosshead bounce energy. In this case the correction is added to the reading from the pendulum because immediately after impact the two halves of the pendulum are travelling in opposite directions. The correction for a given apparatus is determined by means of a special calibration procedure which is detailed in the standard.

ASTM D1822 is essentially Method B of the ISO standard, the specimen-in-head geometry being that favoured in North America, while the specimen-in-bed geometry has been typically favoured in Europe.

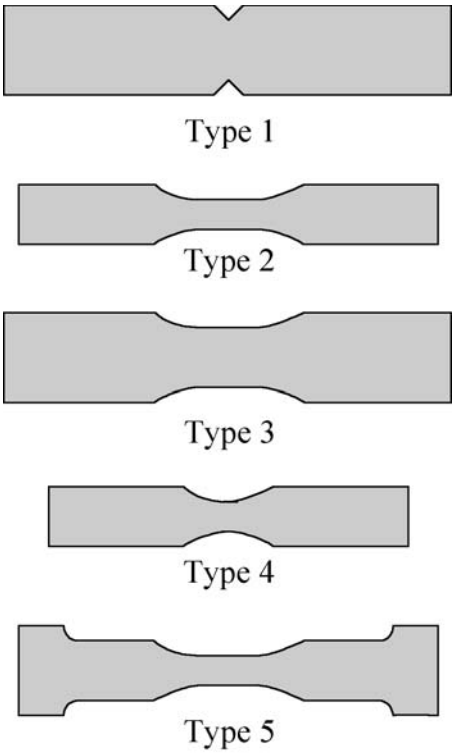

Figure 10.6

Tensile impact has been used to characterise the effect of molecular weight on impact and stress-strain properties [59] while Dijkstra and co-workers [60] have used tensile impact to investigate the toughening effects of rubber in Nylon 6.

10.2.2 Drop Methods

10.2.2.1 Falling Dart Methods

The traditional falling dart methods require a large number of test specimens because for each drop there can be only two outcomes: the test piece fails according to some agreed criteria, or it passes. The amount by which it passes or fails cannot be judged. Results must therefore be analysed statistically in order to quantify the mean energy or mass or height which causes failure. With the newer methods, piezoelectric or resistive transducers are built into the dart so that the force during impact can be monitored directly and a quantitative result obtained for each test piece tested.

The general test for plastics is covered in BS EN ISO 6603-1 [61]. Test pieces of preferred size 60 mm square (or circular) by 2 mm thick are supported on an annular base of inside diameter 40 mm and the dart with a 20 mm diameter striker is released from a preferred height of one metre. The test piece may be clamped or unclamped on the support, although the standard indicates that different results are likely to occur from these two techniques and it is permitted also to use a 10 mm diameter striker.

Two methods of analysis are covered. The preferred method is the 'staircase' method in which the mass of the dart is varied in given increments according to whether the test piece previously tested passed or failed. If it passed, the mass is increased to increase the probability of failure next time, and if it failed, the mass is decreased to decrease the probability of failure. At least 20 test pieces are required plus an additional 10 used as preliminary specimens to select a suitable starting mass and increment. The increment by which the mass is changed must be kept constant throughout a given test run. In Method B, the 'statistical' or 'probit' method, a minimum of 40 test pieces is required, although in practice 60 or more tend to be needed. Here 10 test pieces are tested under given conditions and the percentage of failures recorded. The mass is then altered and a further 10 are tested and so on until at least three results are obtained with percentage failures greater than 0% and less than 100% with at least one result greater than 50% and at least one result less than 50%. In this test non-uniform increments of energy can be used so it is easier while the test is underway to ensure that a more even spread of results can be achieved.

For both tests it is permitted to vary the height rather than the mass, although this is not the preferred way to carry out the test as impact velocity is changing along with the impact energy. The variable falling height method is given for the testing of plastics pipes in BS EN 1411 [62], which is also dual numbered as BS 2782 Method 1108B [63].

The mean impact strength and standard deviation are determined by means of a rather complex calculation for the staircase method or for the statistical method by plotting the percentage passes (or failures) against impact parameter (energy, mass, or height according to the requirements) on probability paper and finding the best fit straight line. The parameter which corresponds to the 50% failure probability is the mean value and the difference between the 50% and the 16% (or the 84%) probabilities is the standard deviation.

The method detailed in ASTM D5420 [64] is somewhat unusual compared to other impact standards in that the test piece, which is placed on a support plate having a circular hole of given size, has a striker resting upon it and the striker is then impacted by the falling weight. This is the so-called Gardner impact test and a number of variations in geometry are allowed. It uses the 'staircase' approach to varying the energy of impact, with drop height rather than the drop mass being varied. ASTM D5628 [65] is rather more conventional and follows the same pattern as ISO 6603-1, albeit with different dart shapes and drop height.

Films and sheeting are tested by similar methods, although the test piece diameter tends to be rather larger, typically 125 mm or so, as does the impacting striker. The standardised tests are given in ISO 7765-1 [66], and the identical BS 2782 Method 352E [67], and in ASTM D1709 [68]. There is very little difference between these standards, although the ASTM method does permit either the staircase or the probit method of analysis while the ISO and BS only allow the staircase method to be used. The drop height is either 0.66 m for method A or 1.5 m for method B.

Instrumented impact tests are cited in BS EN ISO 6603-2 [69] for general purpose plastics testing and ISO 7765-2 [70] for films and sheeting. These tests are echoed in the corresponding BS 2782 Method 352F [71], which is identical to the corresponding ISO. The essential difference to the non-instrumented variant is that some load sensing transducer is built into the dart; it is preferable to have this transducer as close to the point of impact as possible to reduce interference from 'ringing' effects as the force wave sweeps up the dart from the moment of contact. **Figure 10.7** gives a schematic illustration of the dart arrangement. The transducer is generally a resistive or piezo-electric device, the latter being preferred as it has a higher natural frequency and is therefore capable of recording faster transitions without attenuation of the signal.

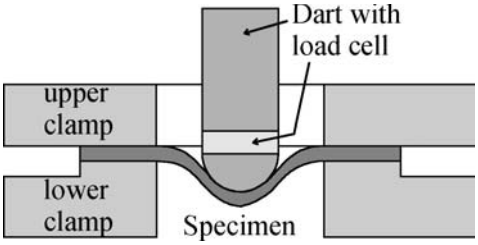

Figure 10.7

Much of the detail concerning test pieces and geometry are as given in ISO 6603-1. It is noteworthy that for the film and sheeting tests, the same 40 mm span is used as for the general test and not the much larger span of 125 mm as in the non-instrumented test. Much of these standards is devoted to the requirements for the instrumentation - the frequency response of the transducer and the band-width of the amplifier to ensure that attenuation or distortion of the signal generated by the impact event are not significant. For the instrumented option, the dart should have a large excess of energy so that the reduction in velocity during impact is small (less than 20%). This is unlike the simple falling weight test, where the energy of the dart has to be similar to the impact energy of the material being tested.

In addition to the impact energy, this test is also capable of delivering the peak force, the energy to peak force and the displacement at peak force. The shape of the force-displacement curve can itself be instructive in characterising the material's behaviour, and the standards give several examples of the type of curve that may be observed, as well as various failure criteria that can be applied. Clearly, therefore, much more data can be derived from this test than is possible from the simple test. It also requires far fewer test pieces: 10 is the norm, but for quality purposes five may be used. The negative side, of course, is that the apparatus is much more expensive and cannot be put together with a minimum of workshop facilities, as can the simple test.

ASTM D3763 [72] follows similar principles to ISO 6603-2 but with differences in striker, fixture, specimen geometry and impact velocity it is hardly surprising that the methods give different numerical values. Suggested impact velocities are 2.5, 25, 125, 200 and 250 m/min. A 12.7 mm dart impacts a test piece of diameter between the inside of the clamp faces of 76 mm.

A particular variation on the non-instrumented falling dart method which is applied to plastics pipes is the 'round-the-clock' method in which the same test piece (a 200 mm long section of pipe) is struck repeatedly at several points around its circumference. The number of impacts per test piece varies according to the diameter of the pipe. For pipes of less than 40 mm nominal size a single blow is administered. This then increases to three for pipes up to 63 mm and so on until 24 impacts are delivered to pipes of nominal size exceeding 355 mm. The impact 'resistance' is measured as the true impact rate (TIR) which is the total number of failures divided by the total number of blows, expressed as a percentage. This technique is standardised in BS EN 744 [73] which is the same as BS 2782 Method 1108C [74] and the older but very similar BS 2782 Method 1108A [75]. In each of these the maximum acceptable value for TIR is 10%. Since the TIR must be established with at least a 90% level of confidence, a very large number of impacts (several hundred) may be required for a pipe that is at all borderline. There must be at least 25 impacts as a minimum.

In ASTM D3420 [76] a pendulum impact test on plastics films is detailed. A 12.7 mm diameter dart-ended pendulum is released from a height which gives an impact velocity of 74 m/min and the energy absorbed from the pendulum as a result of the impact is measured by means of an indicating follower. As with many ASTM standards there are two variants of the apparatus with different manufacturers supplying one or other of the types.

10.2.2.2 Falling Product Methods

As well as tests on materials, impact tests may also be applied to finished products. BS 6642 [77] for example is a specification for plastic refuse sacks, although the specification

has now been declared obsolescent by BSI. One of the tests therein is an impact test in which the sack is partially filled with sawdust and dropped through a hang-man's trap door. Objects such as milk crates and chemical-containing drums are also frequently tested by loading the object in a way that simulates service and then releasing them from a given height – often at various angles, for corner, edge and face impacts – onto a firm foundation such as concrete. The test is often carried out at sub-ambient temperatures to characterise the behaviour under service conditions that might lead to more brittle failures.

ASTM D2463 [78] is applied to blow-moulded containers and essentially consists of a spring-loaded platform upon which the container rests at a pre-arranged height above the impacting surface (a 13 mm thick steel plate). The platform is released from its horizontal plane and falls rapidly away from the container so as not to impede its drop. In Method A, 20 containers are released from an agreed height and the number of failures recorded (primarily used for quality control purposes as it is a rapid test). In Method B the drop height is varied (The Bruceton Staircase method) about the approximate mean height to cause failure and the mean height to cause failure and the standard deviation are then calculated. ASTM D4504 [79] for open pails includes drop tests as does ASTM D1185 [80] for pallets.

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11 Tear Properties

11.1 Introduction

Tear tests are only applicable to flexible sheet and thin films. In a tensile test, taken to break, the force to produce failure in a nominally flawless test piece is measured. In a tear test, the force is not applied evenly but concentrated on a deliberate flaw or sharp discontinuity and the force to continuously produce new surface is measured. This force to start or maintain tearing will depend in rather a complex manner on the geometry of the test piece and the nature of the discontinuity.

Tearing can occur in almost any product made for sheet or film and is also involved in fatigue and abrasion processes in flexible materials. Because tear strength is very geometry dependent it is not surprising that a many different tests have been devised, many of them originating for rubbers. Tear strength from common tests is not an intrinsic property of the material and it can be difficult to directly correlate the results of laboratory tests with service performance. The test conditions which have been standardised are to a considerable extent arbitrary, although in many cases they are meant to represent the sort of stress concentrations found in service.

For rubbers, a fracture mechanics approach was developed many years ago which uses the concept of energy of tearing which is the energy required to form unit area of new surface during tearing. The tearing energy is in theory a basic material property and independent of test piece geometry so that if the tearing energy and the elastic characteristics of the material are known the force needed to tear a given geometry can be predicted. To obtain tearing energy it was necessary to use a test piece where the relationship between force and energy is relatively simple. Although, for example, the trouser test piece is suitable, the approach has not been adopted in standards. Now, other geometries can be treated by finite element analysis. Recently, a fracture mechanics approach has also been applied to tearing of plastic films.

Most tear tests involve applying a tensile force to a test piece and a tensile test machine with suitable grips is used. The response characteristics of the apparatus are particularly important because the force can rise very rapidly and in certain tests rise and fall dynamically.

The widely used Elmendorf apparatus takes a different approach and applies a tearing force by means of a pendulum. Energy stored in the pendulum is used to produce tearing

and the amount used is indicated by the energy lost compared to the total available. It is likely that the original incentive for this approach was to have a relatively cheap and simple standalone apparatus.

11.2 Test Piece Geometry

A very important distinction can be made between the force to initiate a tear as opposed to that to propagate a tear. Both are important because after a tear has started, perhaps because of an accidental cut, the resistance to propagation will determine whether the damage becomes catastrophic. With some materials the difference between force to start a tear is comparable with that to propagate it but in other materials the propagation force can be very much smaller.

The discontinuity at which a stress concentration is produced is formed either by a cut, a sharp re-entry angle or both. Most standard test pieces involve an artificially introduced cut and only in a method with a sharp angle and no cut would any measure of tear initiation force be possible. It can of course be argued that a cut is always possible and if the propagation strength is lower it will be the limiting factor.

The most common form of geometry is where the tear is induced at right angles to the direction of applied force and the stresses at the tip of the tear are essentially tensile. Three well known variants are shown in **Figure 11.1**. All of these are used for rubbers but only the angle test piece is now standardised for plastics. The other common geometry is the trouser test piece illustrated in **Figure 11.2**.

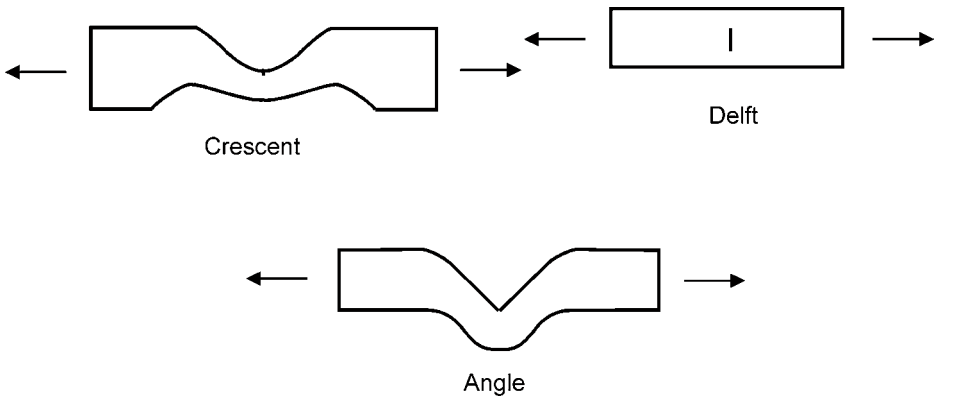

Figure 11.1 Tear test piece geometries

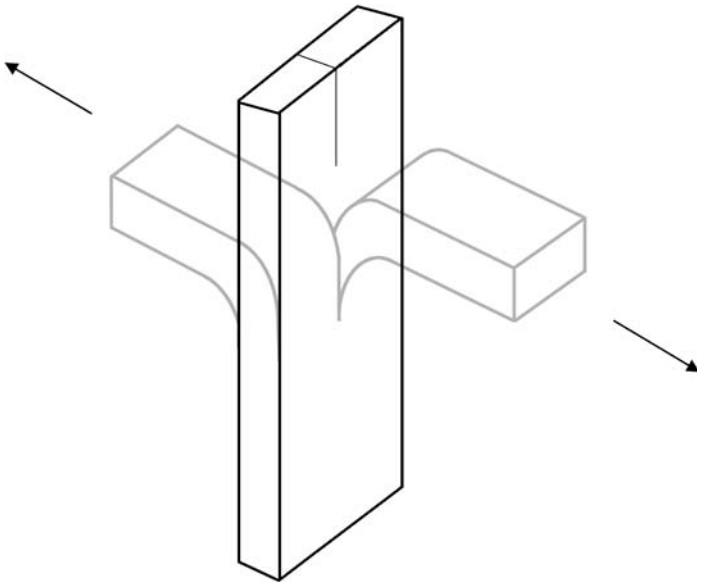

Figure 11.2 Trouser tear test piece

It is not immediately obvious why the crescent tear, so popular for rubbers, is not now used on plastics as it is a very simple concept. The Delft geometry is particularly useful if only small amounts of material are available. The angle tear without a cut is the only geometry in general use where an initiation force is measured but it requires the angle of the cutter to be very carefully maintained to get consistent results. In the trouser tear the stresses must include shear forces. It allows the course of tear propagation to be followed and is a relatively easy shape to cut. For rubbers it also has the advantage of being particularly convenient for calculating tearing energy. One problem is that there can be difficulties in highly extensible materials due to excessive leg extension and a variation is to reinforce the legs with a textile. Another problem can be the path of the tear deviating from the centre line and a special form with a groove has been suggested.

If cutting takes place whilst there is other stress on the material, tearing is being assisted by a sharp object. Cutting involves both the strength properties of the material and friction so that if a stress is applied whilst cutting friction is much reduced and with it the force needed to cause cutting. Puncture tests could be viewed as a sort of tear initiation but the geometry can be arranged so that the tear is propagated after puncture. Generally, cutting or puncture tests operate under *ad hoc* conditions intended to relate to the stress and geometry conditions of service.

11.3 Standard Tests

Only two methods are standardised internationally. The first part of ISO 6383 [1] specifies the trouser tear geometry, essentially the same as that used for rubbers. A test piece 150 mm by 50 mm is cut along the centre of its long axis, from one end to half way down its length. The two 'legs' so formed are gripped in the stationary and moving jaws of a universal testing machine and pulled apart at 200 or 250 mm/min. A note warns that the 250 speed may be deleted in future revisions.

Typically, an irregular wavelike trace results (**Figure 11.3**) and the standard defines the tearing force as the mean force after ignoring the first 20 mm and last 5 mm of the tearing trace. This tearing force is then normalised by dividing it by the film or sheet thickness to produce the tearing resistance value. BS ISO 6133 [2] specifically deals with the interpretation of tear and adhesion traces but although it is titled rubber and plastics it was developed by the rubber committee and has been ignored by the equivalent plastics committee.

The standard gives three procedures for traces having less than 5 peaks, 5-20 peaks and more than 20 peaks. For less than 5 peaks the median of them all is taken, for 5-20 peaks the median of the peaks in the central 80% of the trace is taken and for more than 20 peaks the trace is divided into tenths by 9 lines, the peak nearest to each line noted and the median of these taken. The basic question is whether the mean of the trace the peaks or the valleys are the most relevant criteria? Clearly, the mean is simplest but cases have been argued for the valleys (worst case) and the peaks (best case).

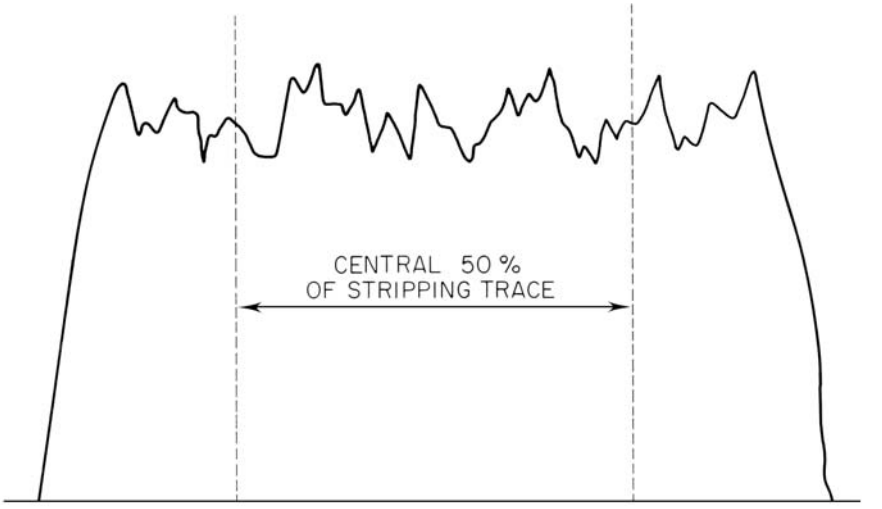

Figure 11.3 Tear or peel adhesion trace

ISO 6383-2 [3] specifies the Elmendorf method in which the test piece is held in the jaws of a pendulum, one jaw fixed and one attached to the pendulum arm. The pendulum is released and causes an initial cut in the test piece to propagate across it (**Figure 11.4**). The energy absorbed by this tearing process is measured by the height to which the pendulum swings once tearing is complete.

Two test pieces are specified, the rectangular and the constant radius test piece, but the latter is preferred on the grounds that it has been shown to give better reproducibility. As the name implies, the rectangular test piece is rectangular with sides 75 mm by 63 mm and a 20 mm long slit cut in the middle of the longer side and parallel to the shorter side. The constant radius test piece is of the same 75 mm length with the same 20 mm slit, but the edge facing the cut is circular with radius 43 mm rather than straight. This means that if the tear propagates at an angle to the motion of the pendulum the tear length remains constant, and so, in principle, should the tearing energy.

The standard allows additional masses to be added to the pendulum or for test pieces to be plied up so that the energy used falls between 20% and 80% of the pendulum capacity.

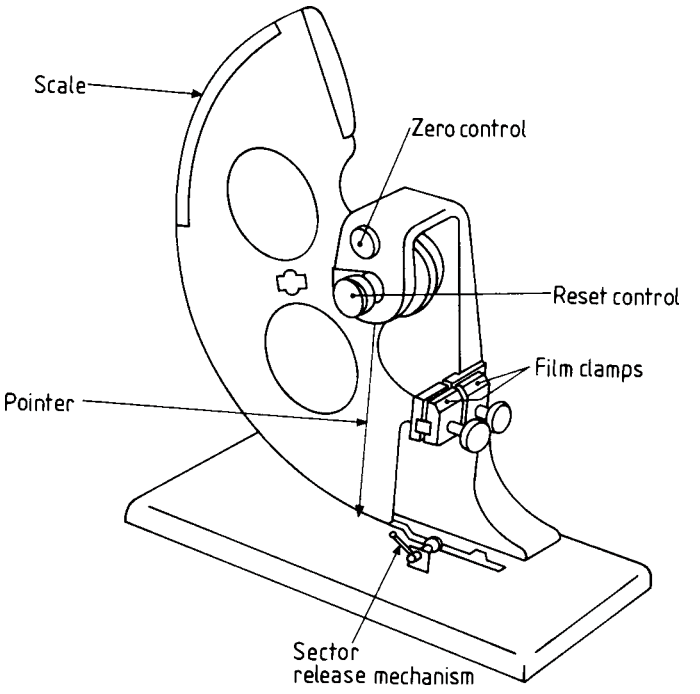

Figure 11.4 Elmendorf tear tester

Although the test is energy-based, it is a tearing force that is calculated from the scale reading and the conversion factors provided by the manufacturer. Interestingly, the tearing resistance is the tearing force and not the tearing force normalised to the test piece thickness as it is in Part 1 of the standard. Whilst normalising to the thickness invites people to believe that tear strength is proportional to thickness, ignoring thickness makes no compensation at all. If a product is being tested it is reasonable to argue that the result represents the strength of the product and the thickness is irrelevant.

An appendix gives a procedure for calibrating the pendulum by adding weights but appears to make the assumption that a pendulum factor is known and is correct.

In British standards, the same methods are standardised and dual numbered as BS 2782 Method 360B [4] and Method 360A [5].

A third British method, BS 2782 Method 360C [6], use the angle tear test piece without a cut similar to that specified for rubbers. The dumbbell is pulled in a tensile machine at 250 mm/min and the maximum force noted. The tear strength is calculated from force divided by thickness. There being no cut in the test piece before testing, this method is a measure of tear initiation.

ASTM has four tear test methods. D1938 [7] is based on the trouser method but has a smaller test piece than the ISO procedure and specifies only 250 mm/min. D1922 [8] uses an Elmendorf pendulum with the constant radius test piece similar to the ISO standard. D1004 [9] is the angle tear method but using a grip separation speed of only 51 mm/min. This is sufficiently different from the ISO and BS methods for one to expect differences in test results.

D2582 [10] uses a novel falling dart arrangement (**Figure 11.5**) which is intended to simulate snagging hazards. A weighted carriage is mounted in a guide channel on a tower and released from a standard height. Fastened to the side of the carriage and protruding horizontally from it is a cylindrical tearing probe with a truncated cone at the tearing end. This falls against the test piece which is clamped to a curved holder adjacent to the tower down which the carriage falls so that the distance from the film to the tower decreases the further down the tower the carriage falls. After falling 508 mm, the probe just touches the surface of the film and thereafter penetrates and rips down the film. The length of tear so produced is taken as the measure of tear resistance.

As with other mechanical tests, the properties of the film or sheet may vary significantly with direction. Consequently, it is common practice and often specified that tear strength is measured in both the machine or longitudinal direction and the transverse or cross direction.

11.4 Other Tests

As mentioned earlier, various *ad hoc* cutting and puncture tests have been used to simulate particular service conditions. Also, some people view the falling dart impact test on films as a puncture test that relates to tear resistance. An analysis of cutting methods as applied to rubber has been given by Lake and Yeoh [11].

In some applications a tear is propagated through dynamic stressing so it may be appropriate to apply a form of fatigue test. Cut growth fatigue tests standardised for rubbers can be viewed in this way [12] and an instrument known as the tear analyser has been specifically developed and fracture mechanics principles applied [13].

Dawson and Bowes [14] point out that falling dart impact and Elmendorf tear often give contradictory results and consider the use of single specimen J-integral analysis and crack

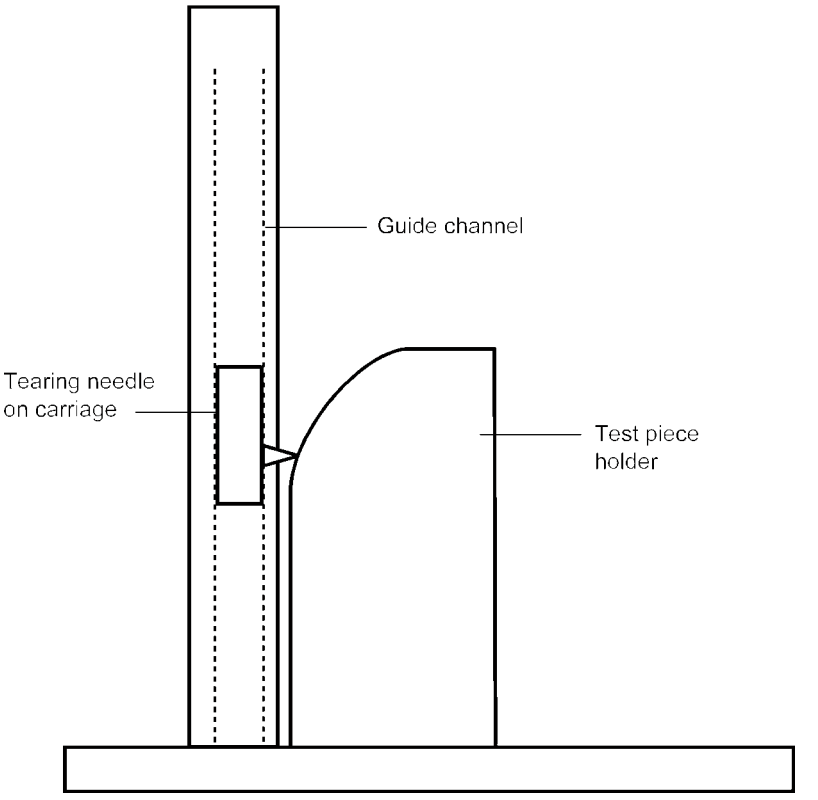

Figure 11.5 ASTM puncture propagation

tip opening displacement methods. The single specimen J-integral method was applied to blown film by Eason and co-workers [15] and comparison made with dart and Elmendorf tests.

Marzinsky and co-workers [16] argue that the widely used Elmendorf method does not yield results that relate directly to toughness or any other true material property but, if the apparatus was instrumented, data analogous to results from an instrumented impact test can be generated. They describe a set-up for instrumentation of the test.

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12 Fracture Toughness

12.1 Introduction

All the traditional standard stress-strain tests for plastics have a severe handicap for design purposes because the results are geometry dependent and they do not yield fundamental properties. Fracture mechanics provides a way of interpreting the material response independently of geometry through consideration of the loads or stresses that cause a crack to propagate. This approach was first proposed by Griffith as long ago as 1920, so it is somewhat surprising that this is the first Handbook of Plastics Test Methods to mention fracture mechanics. The reason is simply that the Handbook has been primarily concerned with the commonly used tests in industry and those that have been standardised. The first international standard for a fracture mechanics based test on plastics did not appear until 2000.

It is not appropriate for this book to include an account of fracture mechanics nor to cover all the test protocols and interpretation procedures that have been applied. Indeed, that would require at least one volume and there are many accounts already existing in the literature. Suitable texts include a chapter in *Handbook of Polymer Test Methods* [1] and *Fracture Mechanics Testing Methods for Polymers Adhesives and Composites* [2]. However, the basic concepts are briefly summarised next.

The basis of the fracture mechanics approach is that the material contains flaws or cracks at which stress concentrations occur when a load is applied. The crack will grow when the stress intensity at the tip of the crack exceeds the cohesive strength of the material. In a brittle material the energy needed to create new fracture surface is balanced by a reduction in the total elastic strain energy of the stressed sample. For ductile materials the energy balance will be with the work done to cause plastic deformation as well as the creation of new fracture surface.

In linear elastic fracture mechanics (LEFM) all energy dissipation is associated with the fracture process and the deformation that occurs is linear elastic. The energy release rate, G, is defined as the rate of energy released by the crack growth. The critical energy release rate, G_c, is the rate for fracture to occur and is expressed as energy per unit test piece thickness and unit crack growth (per unit fractured area). G_c may be constant or vary with crack length and in the latter case a curve of G_c against crack growth (resistance

or R curve) is needed to fully characterise the material. However, the initial (lowest) value is generally considered to be the most important.

In terms of stress intensity, the critical stress intensity factor, K_c, is the minimum stress intensity for fracture to occur and, although called a factor, has units of Pa $m^{0.5}$. G_c and K_c are related by:

$$K_c = (EG_c)^{0.5}$$

where E is Young's modulus.

To relate G_c and K_c to the measured load or energy requires a calibration factor which is a function of the crack length and the test piece width.

When the geometry of test is such that the crack faces move apart with the displacement being normal to the crack faces it is termed mode I propagation and G_c and K_c are termed G_{IC} and K_{IC}.

LEFM applies in many practical cases but in others significant plastic deformation takes place and elastic – plastic fracture mechanics apply. Then fracture toughness is characterised by parameters such as crack tip opening displacement (CTOD) or J-integral. With CTOD, a plot of load against crack mouth opening displacement is used to find the plastic component of the crack opening. J_c is the equivalent of G_c for a non-linear elastic material.

12.2 Standard Methods

There is currently only one international standard for measuring fracture toughness, ISO 13586, [3]. This is based on LEFM using either single edge notch bending (SENB) or compact tensile (CT) test pieces as illustrated in **Figures 12.1 and 12.2**. The methods are intended for rigid and semi-rigid plastics generally but limitations are given for the linearity of the load displacement curve and on the dimensions of test pieces so that conditions are reasonably valid for LEFM. For linearity, the limitation specified is arbitrary and equates to better than 10% non-linearity. The test pieces suggested are normally satisfactory in terms of dimensions but a way of checking validity is given.

It is essential that the initial crack is sharp enough that an even sharper crack would not make significant change to the values obtained. The normal process is to machine a sharp notch and then to open a crack by tapping with a razor blade. If this is not successful for a tough material the crack can be sharpened by sliding the razor blade across the notch, or the test piece cooled before tapping.

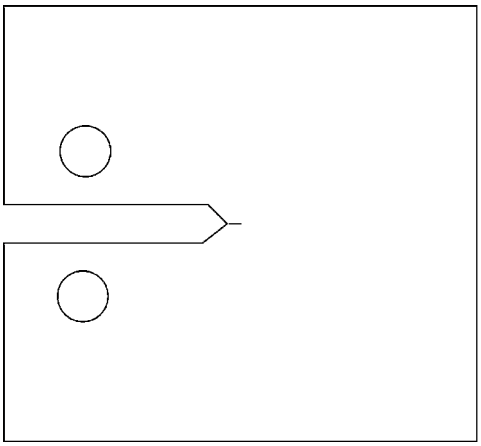

Figure 12.1

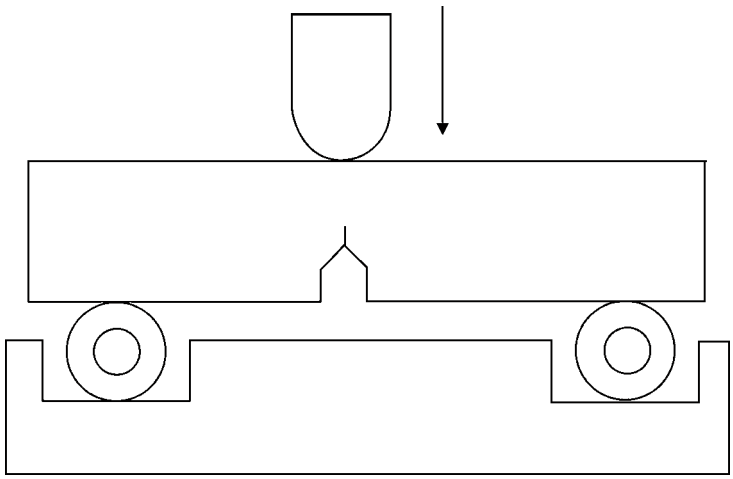

Figure 12.2

The basic test conditions recommended are 23 °C and a speed of 10 mm/min and it is advised that speeds greater than 0.1 m/s and loading times of less than 10ms are likely to cause dynamic errors. If a valid test cannot be obtained at 23 °C it is suggested that the temperature can be lowered to increase the yield stress but without greatly changing K_{IC}. Presumably, increasing the speed would have the same effect.

The displacement is measured with a transducer but no details are given. Corrections have to be made for indentation of the loading pins, compression of the test piece and compliance of the machine. It is not explicit that not all of these will apply to both test pieces or transducer used. Procedures are given for making the corrections.

The critical energy release rate and critical stress intensity factor are calculated using calibration factors given in an annex. These are validated for size criteria before being reported and also a cross check procedure is given based on the relationship of modulus with the fracture mechanics properties.

It becomes clear that a great deal more is involved in carrying out a test of this nature than for a traditional tensile of flexural test. Inevitably, this is one reason why fracture mechanics test are relatively little used in industry.

An amendment to ISO 13586-1 is in draft form which adds guidelines for determining fracture toughness of discontinuous reinforced moulded plastics. This was originally intended to form Part 2 of the standard but has been relegated to an annex to Part 1.

A method for determining fracture toughness at moderately high loading rates is also being developed in ISO [4]. This is described as supplementing ISO 13586 by applying the LEFM approach to higher loading rates. The same general principles, methods, rules and restrictions apply, and the same test pieces are specified. The basic loading rate is given as 1 m/s but the significance of the time to fracture rather than loading rate is pointed out.

Largely because of the lower accuracy expected at higher rates multiple test pieces with varying initial crack length are used to obtain G_{IC}. The means of applying the load can be a falling weight or pendulum impact device or a servo hydraulic testing machine. For the independent determination of G_{IC} the machine needs to be instrumented to record force and the load point displacement and the problems of dynamic effects at higher speeds and how to minimise them are explained.

The procedures for making the measurements and manipulation of the data obtained are inevitable somewhat complicated. Although quite detailed instructions are given in the draft, those without prior experience may find it relatively difficult to follow as the order of procedure, data handling and expression of results is not entirely logical.

The procedure given in ASTM D5045 [5] is technically very similar to ISO 13586-1 but there is a rather more full explanation of the principles, notably in a significance and use section.

ASTM also has a method, D6068 [6], for determining J-R curves which is intended to apply to materials that would not give a valid characterisation by LEFM. The same basic

test piece configurations as for LEFM are used and the method is based on a multi test piece approach with each being loaded to a different displacement and providing one point on the J-R curve (J integral versus crack growth). Corrections are made for non-fracture energy by tests on separate unnotched test pieces.

The procedures are more complicated than for the basic LEFM method and not particularly easy for the inexperienced to follow. This is not helped by there apparently being errors in the references to the diagrams.

12.3 Other Methods

The test approaches mentioned in the previous section have been developed over some 15 years by Technical Committee 4 of the European Structural Integrity Society (ESIS) and its forerunner the European Group on Fracture. Details of these and other test protocols developed by ESIS can be found in *Fracture Mechanics Testing Methods for Polymers, Adhesives and Composites* [2]. Apart from giving the background to fracture toughness measurements and protocols for further test procedures, this reference will be found helpful when using the standards discussed above.

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Abbreviations and Acronyms

ABS	Acrylonitrile-butadiene-styrene
ASTM	American Society for Testing and Materials
BS	British Standard
BSI	British Standards Institute
CEN	European Committee for Standardisation
CENELEC	European Committee for Electrotechnical Standardisation
CT	Compact tensile
CTOD	Crack tip opening displacement
DSC	Differential scanning calorimetry
EC	European Community
EFTA	European Free Trade Association
EN	European standard
ESIS	European Structural Integrity Society
HDPE	High density polyethylene
IEC	International Electrotechnical Commission
IRHD	International Rubber Hardness Degrees
ISO	International Standards Organisation
LEFM	Linear elastic fracture mechanics
PB	Polybutadiene
PBTP	Polybutylene terephthalate
PEEK	Polyether ether ketone
PP	Polypropylene
PS	Polystyrene
PTFE	Polytetrafluoroethylene
PVC	Polyvinyl chloride
RH	Relative humidity
RIM	Reaction injection moulding

rpm	Revolutions per minute
SAN	Styrene acrylonitrile
SEN	Single edge notch
SENB	Single edge notch bending
SI	Systeme internationale
T_g	Glass transition tempertaure
TIR	True impact rate
TNO	Netherland's Organisation for Applied Scientific Research
UKAS	United Kingdom Accreditation Service
UV	Ultra violet

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Author Index

L

M

N

O

P

Standards Index

Main Index

E

F

G

H

I

K

L

M

N

O

P

Q

R

S

T

U

V

W

Y

Rapra Technology Limited

Rapra Technology is the leading independent international organisation with over 80 years of experience providing technology, information and consultancy on all aspects of rubbers and plastics.

The company has extensive processing, analytical and testing laboratory facilities and expertise, and produces a range of engineering and data management software products, and computerised knowledge-based systems.

Rapra also publishes books, technical journals, reports, technological and business surveys, conference proceedings and trade directories. These publishing activities are supported by an Information Centre which maintains and develops the world's most comprehensive database of commercial and technical information on rubbers and plastics.

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