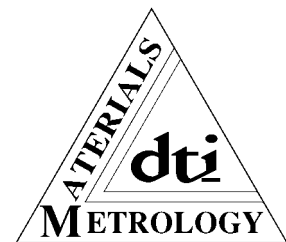


# **A Guide to the Preparation and Testing of Bulk Specimens of Adhesives**

## **Measurement Good Practice Guide**

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# Measurement Good Practice Guide No. 17

## Preparation and Testing of Bulk Specimens of Adhesives

Greg Dean and Bruce Duncan  
Centre for Materials Measurement and Technology  
National Physical Laboratory

### **Abstract:**

This Guide describes methods for preparing bulk specimens of adhesives and methods for testing these for the determination of mechanical property data needed for design. Emphasis is given in both specimen preparation and testing to the acquisition of accurate data. The Guide is applicable to one-part adhesives that cure by heating and to two-part systems that must be mixed prior to curing. It is not suitable for adhesives whose cure requires the evaporation of solvents or the liberation of gas.

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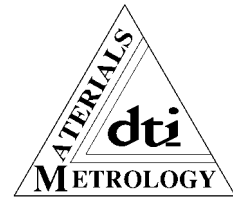
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# A Guide to the Preparation and Testing of Bulk Specimens of Adhesives

## A Best Practice Guide

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### Scope

This Guide describes methods for preparing bulk specimens of adhesives and methods for testing these for the determination of mechanical property data needed for design. Emphasis is given in both specimen preparation and testing to the acquisition of accurate data. The Guide is applicable to one-part adhesives that cure by heating and to two-part systems that must be mixed prior to curing. It is not suitable for adhesives whose cure requires the evaporation of solvents or the liberation of gas.

# A Guide to the Preparation and Testing of Bulk Specimens of Adhesives

## A Best Practice Guide



## Contents

<b>1. Introduction</b>	<b>1</b>
<b>2. Preparation of Bulk Specimens</b>	<b>2</b>
2.1 ISO STANDARDS	2
2.2 EXCLUSION OF AIR FROM SPECIMENS	2
2.3 THE MOULD	3
2.4 MIXING THE COMPONENTS OF TWO-PART SYSTEMS	4
2.5 DISPENSING THE ADHESIVE INTO THE MOULD	4
2.6 CURING THE ADHESIVE	4
2.7 TEST SPECIMEN PREPARATION AND CONDITIONING	5
2.8 SUMMARY OF SECTION 2	5
<b>3. Testing of Bulk Specimens</b>	<b>6</b>
3.1 TENSILE TESTS	6
3.1.1 Test methods, properties and standards	6
3.1.2 Specimen geometries	7
3.1.3 Methods for strain measurement	7
3.1.4 Summary of tensile strain measurement methods	9
3.2 SHEAR TESTS	9
3.2.1 Data requirements from shear tests	9
3.2.2 Notched Beam Shear (Iosipescu) Method	9
3.2.3 Notched Plate Shear (Arcan) Method	10
3.2.4 Torsion Method	10
3.2.5 Summary of shear test methods	11
<b>4. Rate and Temperature Dependent Behaviour</b>	<b>11</b>
<b>5. Acknowledgements</b>	<b>12</b>
<b>6. References</b>	<b>12</b>

# A Guide to the Preparation and Testing of Bulk Specimens of Adhesives

## A Best Practice Guide



## 1. Introduction

There are a wide range of test methods and associated test specimens that are used to evaluate the performance of adhesives and adhesive joints. A small fraction of these are able to generate data for design. This Guide is concerned with the preparation and testing of bulk specimens of adhesives and gives emphasis to the determination of data suitable for design. The issues covered in this guide were studied in Project 1 of a programme of research on adhesives funded by the DTI between 1993 and 1996. The title of Project 1 was "Measurement of Basic Properties for Design". A complementary publication (1), which is based on the findings of Project 2 of the programme, addresses failure criteria for adhesive bonds.

A particularly powerful design method involves the use of a finite element analysis to calculate stress and strain distributions in the adhesive under various loading conditions. This will locate regions of stress and strain concentration in the adhesive and also predict the overall deformation of the joint. It is then possible to explore how stress and strain levels can be reduced through changes to the size or geometry of the joint. In conjunction with a suitable criterion for failure of the adhesive, the analysis can also be used to determine safe operating loads and durations for different loading situations.

The data requirements for a finite element analysis depend on the materials model used by the analysis for describing the deformation behaviour of the adhesive. If the maximum strain developed in the adhesive is within the range of linear behaviour then an elastic model can usually be employed. The use of this model requires the determination of two material parameters. The tensile modulus and Poisson's ratio are commonly chosen, but, if shear test data are available, then the tensile and shear moduli are equally suitable. Polymeric adhesives are to some extent viscoelastic and therefore have properties that are time and rate dependent.

An elastic analysis may however still be of adequate accuracy if properties are measured at an effective rate or time appropriate to the application. Exceptions arise if the load is intermittent or the adhesive is significantly viscoelastic. In these cases a linear viscoelastic model may need to be used for which creep and recovery data are needed.

Many modern adhesives are tough materials and can sustain large strains before fracture. Relationships between stress and strain are then non-linear, and if finite element methods are to be used to investigate stress and strain distributions then a suitable materials model must be used for the analysis. Discussion of the types of model that are available for describing non-linear behaviour is not covered in this Guide, but a review of these is given in reference 2. The data needed by these models must be derived from measurements of stress/ strain curves and, in general, under more than one stress state.

The data required for a linear elastic analysis and some of the data required for a non-linear analysis can be determined from tests on bonded test specimens. However, it is difficult to

# A Guide to the Preparation and Testing of Bulk Specimens of Adhesives



## A Best Practice Guide

achieve high accuracy in the measurement of strain in the adhesive because the associated displacements are very small. If bulk specimens of the adhesive can be prepared, then high accuracy can be readily achieved in the measurement of adhesive properties using test methods and test specimens that have been developed and standardised for engineering plastics. Furthermore, measurements can be made over a wider range of stress states.

In this guide, precautions needed for the preparation of bulk specimens of suitable quality are discussed. Various methods are then described for determining the properties of the adhesive using bulk specimens of suitable size and geometry.

## 2. Preparation of Bulk Specimens

### 2.1 ISO Standards

Recommended procedures for the preparation of bulk specimens of adhesives have been described in the ISO standard ISO 15166 (3). The standard consists of two parts; part 1 deals with two-part adhesives that involve the mixing of two chemicals prior to curing, which takes place subsequently at ambient or elevated temperatures. Part 2 is concerned with single component systems that require an elevated temperature to cure the adhesive.

In this section, methods for preparation are summarised and emphasis is given to the preparation of specimens of suitable quality for mechanical property determination.

### 2.2 Exclusion of Air From Specimens

One of the factors that influences the quality of bulk specimens of adhesives is the presence of air bubbles. The presence of small amounts of air in test specimens will not significantly influence mechanical properties except possibly the strain to failure. In a specimen under stress, small bubbles will give rise to regions of strain concentration at which failure can initiate prematurely. Air may be present in the adhesive or its components as supplied by the adhesive manufacturer. It may also be introduced during the mixing of two-part adhesives or during casting into the mould. The preparation method outlined in section 2 aims to minimise the inclusion of any additional air during preparation of bulk specimens.

Air already present in the adhesive will also occur in any joints manufactured. Therefore, it can be argued that removing this air during the preparation of bulk test specimens may make the samples unrepresentative of the material in the joint. A counter argument can be made that air bubbles in the centre of the bond will not be in critically strained regions of the joint and will not significantly effect failure but that bulk specimens containing air will fail prematurely leading to underestimates of the load bearing properties of the joint. The normal practice

# A Guide to the Preparation and Testing of Bulk Specimens of Adhesives



## A Best Practice Guide

should be that air need not be removed from the as-supplied adhesive unless the presence of air is likely to cause problems with measurements of material properties.

If required, the concentration of trapped or dissolved air in the as-supplied adhesive can be reduced by stirring the adhesive or its components under vacuum. This is a laborious exercise which usually involves emptying several cartridges into the vacuum stirrer and then refilling them under vacuum. This approach should not be taken if there is a risk of losing volatile substances in the process. The procedure is also not very effective for high viscosity adhesives unless they can be warmed slightly to lower the viscosity. The adhesive manufacturer should be consulted for advice in these circumstances. Some workers (4) have reported success with reducing entrapped air by spinning a vessel of the material in a centrifuge. The air becomes located preferentially in material closest to the axis of revolution. One serious problem with centrifuging is that the mix of the constituents of the adhesive may be destroyed and the resulting material will not be representative.

### 2.3 The Mould

In its most simple form, the mould consists of two plates separated by spacers as shown in figure 1a. The two plates, which form the upper and lower faces of the mould, should be flat, in order to produce a moulding of uniform thickness, and thick enough to avoid significant bending under the pressure needed to close the mould. This type of mould is suitable for viscous adhesives that will not flow out of the mould during casting and curing. If the adhesive has a low viscosity, either before curing or at some stage during an elevated temperature cure, then the spacers should be replaced by a frame, as shown in figure 1b or 1c, to prevent the adhesive from flowing from the mould. The closed frame in figure 1c is used for low viscosity materials that would flow out of the open frame before the mould could be closed. The simple mould designs shown in figure 1 produce a plate moulding from which specimens of required shapes and dimensions can be cut. A shaped frame can alternatively be used to prepare specimens of desired geometry and thereby avoid any machining.

The surfaces of the mould should be coated with a release agent or covered with a polytetrafluoroethylene (PTFE) film to prevent the adhesive from adhering to the mould. Alternatively, the spacer or frame can be made from PTFE or another low surface energy material.

A spacer or frame thickness of between 2 mm and 3 mm is recommended. Below this thickness, specimens may be fragile and more prone to premature failure by the influence of extensometer contacts. A thickness greater than 3 mm should be avoided for adhesives that cure by a rapid exothermic reaction. For these materials, the upper and lower surface plates should be thick and made of a high conductivity metal in order to dissipate the heat of the reaction and prevent excessive temperature rises.

Thicker mouldings can be prepared of adhesives that can be cured slowly to allow any heat of the reaction to be conducted from the centre of the moulding without an excessive

# A Guide to the Preparation and Testing of Bulk Specimens of Adhesives



## A Best Practice Guide

temperature increase. To achieve satisfactory specimens in these circumstances, it may be necessary to cure the adhesive below the temperature range recommended by the adhesive manufacturer. There may then be some uncertainty regarding whether the properties of a thick specimen are similar to those of thinner specimens that have been cured under different conditions. Whether representative material has been prepared should be checked by comparing the properties of thin specimens with sections from the thicker specimen.

### 2.4 Mixing the Components of Two-part Systems

Mixing of components is most conveniently achieved using a static mixer. A diagram is shown in figure 2. Dispensing of the components can be carried out manually or by pneumatic assistance. It is generally wise to select a nozzle with a large number of blades (>30) in order to ensure complete mixing of the components. If the working life of the mixture is sufficiently long at ambient temperatures, it should be possible to mix the components by hand in a separate vessel under vacuum. Mixing by hand in air is not recommended because of the high probability of incorporating air into the mixture.

### 2.5 Dispensing the Adhesive into the Mould

Adhesives that are available in cartridges can be conveniently dispensed into the mould through a nozzle attached to the cartridge. The adhesive should be cast into the centre of the mould, as shown in figure 3, in a single process and without removing the tip of the nozzle from the cast. This will minimise the possibility of introducing air into the cast. If the adhesive is viscous, the cast should be higher than the spacer or frame thickness. As the mould is closed, the adhesive will then be spread into a sheet of uniform thickness.

If the adhesive is able to flow freely, it should be poured into the centre of the mould, care being taken to avoid turbulence that may trap air. After closing the mould, it should be tilted to allow the adhesive to fill the space between the plates and produce a sheet of uniform thickness. Sufficient pressure is then needed between the plates to prevent leakage.

### 2.6 Curing the Adhesive

Depending on the adhesive type, cure may take place at ambient temperature, generally soon after mixing the components, or at an elevated temperature. As noted earlier, when curing adhesives that have a rapid exothermic reaction, it is necessary to ensure that the temperature never exceeds the maximum cure temperature specified by the adhesive manufacturer. If the specimen thickness is such that it is uncertain whether the temperature rise is excessive, trial experiments should be carried out using a mould in which temperature sensors are located in the mould cavity. If necessary, the use of lower initial cure temperatures and longer cure times should be explored. A post-cure should also be considered to ensure that the cure reaction is complete.

# A Guide to the Preparation and Testing of Bulk Specimens of Adhesives

## A Best Practice Guide



### 2.7 Test Specimen Preparation and Conditioning

A mould similar to those depicted in figure 1 will give mouldings in the form of a plate or sheet from which specimens of suitable size and geometry must be prepared by machining. Preparation by stamping is viable only for highly ductile adhesives but, even then, ultimate property measurements may be influenced. The ISO standard ISO 2818 (5) gives guidance on the preparation of specimens by machining. The standard ISO 572 part 2 (6) gives details of shapes and sizes that have been standardised for tensile tests.

Where measurements of stress/strain curves to failure are to be made, specimens should be cut from regions of the moulding that are relatively free of air bubbles. For transparent or translucent materials, suitable regions can be identified by visual inspection, possibly aided by a light source behind the plate. For opaque materials, an ultrasonic scan can be used to indicate the clearest regions. In addition, for measurements of ultimate properties, it is recommended that the machined edges of the specimen be polished to remove sites for premature failure that may have been introduced by machining. This is particularly important for low-ductility adhesives.

Many adhesives absorb small quantities of water. Epoxies and acrylics for example may typically contain about 5% by weight of water when in equilibrium with an atmosphere of 50% relative humidity at 23°C. This weight content can be far higher for some materials especially when saturated. The water content generally has a significant effect on properties, and so, for comparable data, specimens may need to be conditioned in atmospheres of known humidity prior to mechanical property measurements.

### 2.8 Summary of Section 2

Air bubbles in bulk specimens may influence mechanical properties, especially strain to failure. Precautions should be taken to avoid the inclusion of air during the mixing of two-part adhesives and when casting the adhesive into the mould. Small quantities of air are acceptable if specimens can be prepared from regions in a moulding that are essentially free from bubbles.

Care should be taken with the preparation of specimens of adhesives that cure by a rapid exothermic reaction to prevent the temperature of the material from rising above the maximum value specified by the adhesive supplier. This is unlikely to be a problem if specimen thicknesses do not exceed 2 or 3 mm. This is a convenient specimen thickness for the measurement of properties with the possible exception of flexible adhesives where a slightly greater thickness may help with the application of extensometers.



### 3. Testing of Bulk Specimens

#### 3.1 Tensile Tests

##### 3.1.1 Test methods, properties and standards

The tensile properties of materials can be determined using a variety of tests. The most common is the constant rate test which involves applying deformation to the specimen at a constant speed. Test speeds are typically in the range 1 to 100 mm/minute which correspond to strain rates in the specimen of around  $1 \times 10^{-4} \text{ s}^{-1}$  to  $1 \times 10^{-2} \text{ s}^{-1}$ . A stress/strain curve to failure is usually measured, and a value for Young's modulus is determined from the initial (linear) part of the record. If the lateral strain in the specimen is also measured, then the Poisson's ratio can be determined.

This test is sufficient to characterise tensile behaviour if, for the purpose of the analysis, the adhesive is assumed to have properties that are independent of the time under load or the rate of loading or if the loading time in a particular application is comparable with the duration of the tensile test.

Alternative tests are needed for the generation of data for applications where the loading time is either very short, such as impact, or very long. The testing of materials for design under high rates of loading is still the subject of research. Materials characterisation for long-term performance however is relatively straightforward and is achieved using creep or stress relaxation tests. In a creep test, a constant load is applied to the specimen and the strain, which increases with time, is measured. The creep compliance of the specimen at any time is the ratio of the strain at that time to the applied stress. The inverse of this quantity is referred to as the creep modulus. In a stress relaxation test, a constant strain is applied to the specimen and the stress, which decreases with time, is measured. The stress relaxation modulus at any time is then the ratio of the stress at that time to the applied strain.

Modulus measurements on plastics, including adhesives, are also commonly made using dynamic mechanical thermal analysis (DMTA). A number of commercial instruments are available for this purpose. With these methods, the response of a specimen is measured to a displacement whose amplitude is varying sinusoidally with time. A dynamic modulus is determined which, for viscoelastic materials, is a complex quantity whose value depends on the temperature and the vibration frequency. Most commercial apparatus accommodates small test specimens which enables measurements over a wide temperature range to be made rapidly. However the small specimen size generally leads to errors in strain measurement, and accurate values for modulus are not readily achievable. Dynamic mechanical measurements over a wide temperature range will conveniently reveal the location of the softening temperature and other relaxation regions of an adhesive, and bulk specimens are needed for this purpose.

# A Guide to the Preparation and Testing of Bulk Specimens of Adhesives



## A Best Practice Guide

ISO standards for plastics can be consulted for guidance in performing some of these tests. ISO 572 part 2 (6) describes the measurement of tensile properties under constant deformation rate. ISO 899 part 1 (7) is concerned with the determination of tensile creep behaviour and ISO 6721 (8) parts 2 to 7 describe the measurement of dynamic modulus by forced non-resonance under different modes of deformation. None of these standards give guidelines on the measurement of tensile strain in the specimen. This can be the main source of error in the determination of tensile properties and more than a single device may need to be employed to measure strain with high accuracy over the range experienced by some materials in a tensile test. In section 3.1.3, various methods for strain measurement are described and their suitability discussed for a range of types of adhesive.

### 3.1.2 Specimen geometries

In section 2.7, reference was made to standard geometries for tensile specimens. These are waisted specimens that ensure that the maximum strain in the specimen is in, or close to, the region of uniform strain in the gauge section. Standard geometries for plastics will also be suitable for adhesives and are specified in ISO 3167 (9). These specimens need to be used if data on the failure of the specimen are to be recorded. If the only data requirements are the Young's modulus and Poisson's ratio for a linear elastic analysis, then a parallel-sided strip is satisfactory.

### 3.1.3 Methods for strain measurement

The measurement of stress in the gauge length of the specimen should be straightforward. It is necessary to employ a calibrated force transducer having an appropriate sensitivity. The dimensions of the specimen cross-section should also be uniform.

The measurement of strain is less routine, and a number of methods are available (10). Methods described in the following parts to this section include contacting extensometers, strain gauges, video extensometers and measurement of grip displacement.

#### 3.1.3.1 Contacting extensometers

These devices generally enable high accuracy to be achieved and are the preferred method of strain measurement for the determination of modulus. Designs based on the use of inductive or capacitive displacement transducers are shown in figure 4. The use of two extensometers attached to opposite surfaces of the specimen is recommended, especially for the measurement of small strains. By taking the average of both measurements, errors caused by bending of the specimen on application of a tensile load are minimised.

The strain range over which these devices operate is typically 0 to 10% although some designs will go higher. Furthermore, contact with the specimen surfaces is usually through knife edges and these can cut the surface and cause premature failure of the specimen. For measurements of tensile strain up to failure, non-contacting methods should be used.

Separate devices are needed for the measurement of the lateral strain for the determination of Poisson's ratio. An example of a contacting extensometer for lateral strain measurement is shown in figure 5.

# A Guide to the Preparation and Testing of Bulk Specimens of Adhesives

## A Best Practice Guide

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### 3.1.3.2 Video extensometers

A video extensometer is a non-contacting method for strain measurement. Using this device, it is generally not possible to achieve satisfactory accuracy in the measurement of strain below about 1%. There are however no restrictions on the upper strain limit, and this is a very convenient method for the determination of strains above 10%.

An extensometer consists of a camera that is mounted remotely from the specimen. The face of the specimen has two or more contrasting lines that mark the gauge length as shown in figure 5. Software scans the camera output and determines the separation of the edges of the lines before any load is applied. The change in the line separation is then recorded throughout the tensile test. Some video extensometers are able to simultaneously measure the lateral strain in the specimen.

Certain adhesives will stress-whiten at moderate strains and this can change the contrast between the gauge markers and the specimen. This can lead to uncertainty in the location of the reference edge. To maintain the contrast, the specimen surface should be coloured prior to testing, for example with spray paint (see figure 5). Since a video extensometer can be mounted on the outside of an environmental chamber, there is generally no restriction in the temperature range over which the device can be used.

### 3.1.3.3 Strain gauges

Significant errors are obtained in the measurement of tensile strain using strain gauges bonded to the surface of the adhesive specimen. Measured strains are smaller than true values, and the errors are greater for thinner specimens and more compliant adhesives. These errors are attributable to a local stiffening of the specimen by the gauge and, although corrections can be estimated, the use of contacting extensometers is more convenient. In common with contacting extensometers, the edges of strain gauges are sites for premature failure and, in general, are limited to the measurement of strains below 10%.

### 3.1.3.4 Measurement of grip displacement

An approximate value for the tensile strain in the specimen can be obtained from measurement of the displacement of the crosshead of the testing machine. A strain value is then derived from the ratio of this displacement and the initial grip separation. This value will differ from the actual strain in the central region of the specimen. The error arises because the strain in the specimen is not uniform along its length. In a waisted specimen, the strain at the ends will be less than in the gauge length, whilst in a parallel-sided specimen there will be regions of non-uniform strain close to, and within, the grips. Furthermore, the measured displacement will be greater than the change in the grip separation because of the compliance of the test arrangement. The application of corrections is feasible (11), although laborious, and will be specific to a particular test machine, a specimen size and geometry and the strain level in the specimen.

# A Guide to the Preparation and Testing of Bulk Specimens of Adhesives



## A Best Practice Guide

### 3.1.4 Summary of tensile strain measurement methods

The accurate measurement of small strains in a tensile test is needed for the determination of Young's modulus and Poisson's ratio. For this purpose, the use of contacting extensometers is recommended. At higher strains, high-elongation, contacting extensometers or non-contacting devices, such as video extensometers, should be used.

Contacting extensometers can cause premature failure so, if failure strains are to be determined, separate tests using non-contacting extensometers should be employed.

If a non-contacting extensometer is not available, an approximate measure of the strain can be obtained from measurement of grip separation. These errors can be reduced through the application of corrections.

The use of strain gauges is not recommended since they introduce an error in the measurement which depends upon the specimen thickness and the modulus of the material.

## 3.2 Shear Tests

### 3.2.1 Data requirements from shear tests

As explained in section 1, the purpose of carrying out shear tests on bulk specimens is to obtain additional data to the tensile results that are needed to allow different elastic-plastic materials models in FE packages to be used for the analysis of performance of the adhesives at large strains where the stress/strain behaviour is non-linear.

Interest in the results at low strains is therefore generally restricted to whether the shear modulus value is consistent with the value derived from measurements of Young's modulus and Poisson's ratio. The main objective of shear tests on bulk specimens is to obtain the shear stress/strain curve. Unlike the tensile test, different types of apparatus and specimen geometries are available for this purpose.

### 3.2.2 Notched Beam Shear (Iosipescu) Method

The specimen and loading device for this test method (12,13) is shown in figure 6. The specimen is typically 75 mm wide, 20 mm high and at least 4 mm thick. Thinner specimens can, in principle, be tested but metal end-pieces need to be carefully bonded on both faces between the jaws to maintain alignment of the specimen during loading. The specimen is loaded in tension or compression and a state of pure shear is established in a region between the notches. Strain gauges may be used to measure the shear strain in this region but a correction is needed for the local stiffening of the specimen by the gauge. The correction depends on the modulus of the adhesive and can be excessive for the more compliant materials. Strain gauges also impose a limit on the strain range over which measurements can be made. An extensometer design is described in section 3.2.3 which overcomes these problems and which should be applicable to this method.

# A Guide to the Preparation and Testing of Bulk Specimens of Adhesives



## A Best Practice Guide

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An average value for the shear stress in the centre of the specimen is obtained from measurements of the applied force and the cross-sectional area of the specimen between the notches. The local stress in the central region where the strain is measured differs from this average value by a factor that depends on the radius of the roots of the notches. An estimate of this correction can be derived by finite element analysis.

### 3.2.3 Notched Plate Shear (Arcan) Method

A modification of the method described in the previous section consists of a similar specimen geometry but the specimen is loaded as shown in figure 7 by grips that clamp the front and back faces, rather than the edges, of the specimen (13,14). This allows thinner specimens and more compliant materials to be tested. The grips are also easier to align. Specimens of thickness 2 mm can be tested with this method which makes it particularly suitable for those adhesives that have a rapid exotherm associated with their cure reaction and for which there are therefore problems with the moulding of bulk specimens of greater thickness.

An extensometer has been developed for use with this method. As shown in figure 8, this device is bolted to the lower grip and contacts the specimen at two points near the specimen centre. The shear displacement between these points is measured, and the shear strain is deduced from a knowledge of the point separation.

The main disadvantage of both this and the Iosipescu method is the presence of local regions of high tensile stress in the specimen near the tips of the notches. In brittle materials these can cause failure before the central area has reached the maximum stress, and the stress strain curve is truncated. More ductile materials can redistribute this tensile stress through plastic deformation, and shear stress/strain data can be obtained well beyond the shear stress maximum for the material.

### 3.2.4 Torsion Method

The torsion of a specimen in the shape of a rod or strip is one of the more accurate methods for determining shear properties, especially at low strains (15). Apparatus consists of a device for applying a torque to the specimen and transducers for measuring the torque and the angular rotation. An example of suitable apparatus is shown in figure 9. Bulk specimens are cast as a bar and the central region is machined to a circular section. For accurate measurements, the diameter should be typically 10 mm. Problems can exist with the moulding and machining of some adhesives to these dimensions, so there will be some limitation in the range of adhesives that can be tested by this method. Contacting extensometers, also shown in figure 9, are used to measure the angular rotation of a known gauge length of the specimen. Simultaneous measurements are made of the relative rotation of the clamps so that a value for the effective length of the specimen can be calculated. Measurements of clamp rotation are then used to determine shear strains in those tests where there is a risk that the contact between the extensometer and the specimen may induce premature failure. An unknown error is introduced with these strain measurements if the effective length varies with the strain in the specimen.

# A Guide to the Preparation and Testing of Bulk Specimens of Adhesives

## A Best Practice Guide



Since a non-uniform shear stress is established through the specimen cross-section, a correction is necessary for the determination of stress/strain behaviour at strains outside the linear region. The correction procedure developed by Nadai (16) for metal specimens is generally used.

### 3.2.5 Summary of shear test methods

Stress/strain data for adhesives under shear are needed in conjunction with tensile measurements to determine parameters for elastic-plastic materials models used for finite element analysis. Whilst joint test specimens can be used for this purpose, bulk specimen tests offer more scope for obtaining accurate measurements.

The torsion test is potentially the most accurate method for the measurement of shear properties but a torsional test machine and bulk specimens of relatively large thickness are required. The preparation of specimens of suitable thickness may present difficulties for some types of adhesive. Good accuracy can be achieved with the notched-plate shear method using bulk specimens of 2 to 3 mm thickness. The more brittle adhesives generally fail before the stress maximum is reached. Purpose-built extensometers are recommended for the measurement of shear strain using this method.

## 4. Rate and Temperature Dependent Behaviour

When a force is applied to a polymer, the resulting deformation is composed of an elastic component that comes predominantly from the stretching of intramolecular bonds and a time-dependent component that arises through relaxation processes that are characteristics of the material and involve changes in molecular conformations. The mobility of these relaxation processes is dependent upon the temperature. Adhesives, being polymeric materials, therefore have properties that vary with the time, rate or frequency of the applied load as well as the temperature. This viscoelastic behaviour gives rise to a number of features associated with mechanical property measurements that are not commonly experienced with other types of material.

The test methods introduced in section 3.1.1 that are commonly used to determine mechanical properties each have an effective loading time associated with a measurement. Properties from these various tests will therefore be different unless care is taken to compare results from tests at the same effective loading time. Reference (17) explains how measurements from these tests can be correlated.

The time, rate or frequency dependence of properties will vary with the temperature. High dependence will be observed when the test temperature coincides with a relaxation region of the material. The greatest variation of properties is seen when the temperature lies close to the glass-to-rubber relaxation temperature  $T_g$  of the adhesive. This is illustrated in figure 10 for a polyurethane adhesive tested close to its  $T_g$ . The figure shows a series of stress/strain curves obtained from constant deformation rate tests carried out at different rates. The

# A Guide to the Preparation and Testing of Bulk Specimens of Adhesives



## A Best Practice Guide

stress/strain behaviour is seen to be sensitive to the rate. The behaviour is also highly sensitive to the temperature. These results emphasise the need for precise temperature control in order to achieve repeatable properties when testing adhesives close to their T<sub>g</sub>.

Also apparent in figure 10 is the curvature in the relationship between stress and strain. This is present despite the fact that strains are small and within the linear region. The curvature is caused, once again, by the viscoelastic behaviour of the adhesive and arises because of stress relaxation throughout the duration of the test. A simple application of linear viscoelasticity theory is used in reference (17) to correlate the rate dependence of properties observed in figure 10 with the results of a stress relaxation test on the polyurethane adhesive.

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The Centre for Adhesive Technology (CAT) for guidance and support.

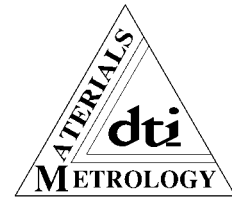
The Welding Institute (TWI) and the University of Bristol for their technical contributions to Project ADH 1.

The Industrial Advisory Group for the ADH Programme for advice and encouragement.

## 6. References

1. J. McCarthy. Industry Briefing: Failure Criteria for Adhesive Joints. AEA Technology (Harwell) Report, 1998.
2. M.N. Charalambides and G.D. Dean. Constitutive models and their data requirements for use in finite element analysis of adhesives under impact loading. NPL Report CMMT(A)59, April 1997.
3. ISO 15166:1998, Adhesives - Methods for preparing bulk specimens. Part 1: Two-part systems.
4. D.E. Lees and A.R. Hutchinson. Mechanical characteristics of some cold-cured structural adhesives. Int. J. Adhesion and Adhesives, vol 12, no 3, July 1992, pp 197-205.
5. ISO 2818:1994, Plastics - Preparation of test specimens by machining.

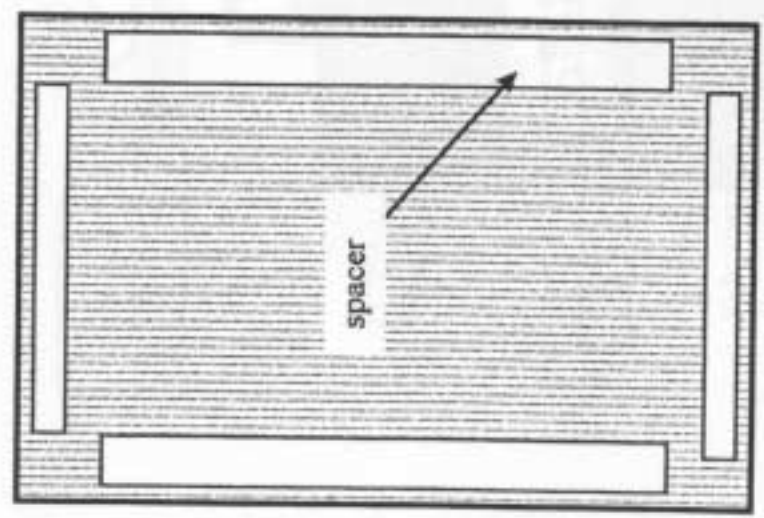
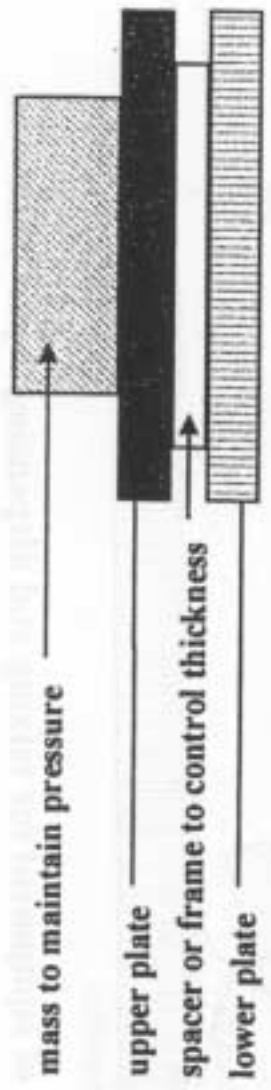
# A Guide to the Preparation and Testing of Bulk Specimens of Adhesives



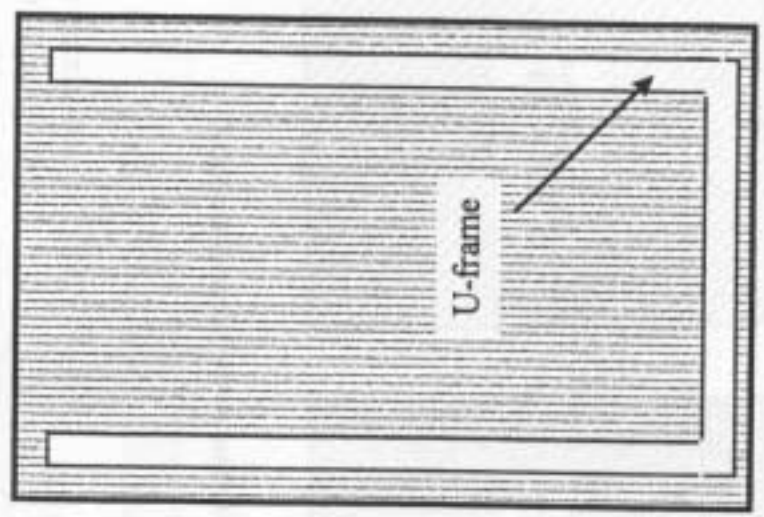
## A Best Practice Guide

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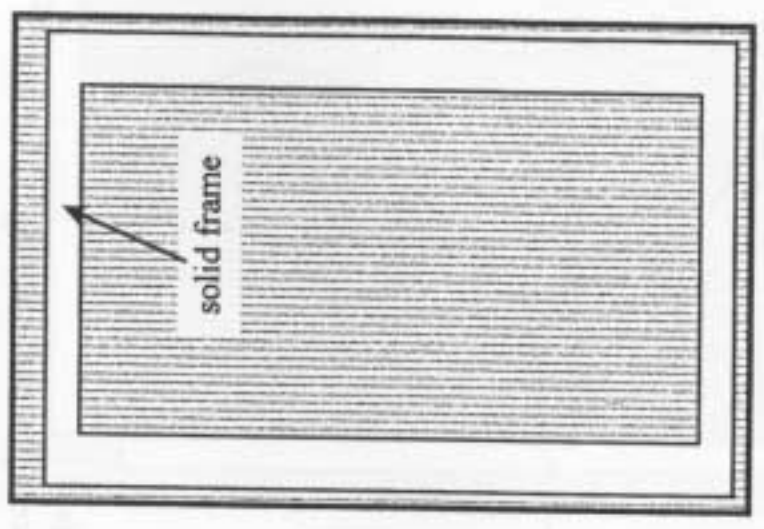
6. ISO 527-2:1993, Plastics - Determination of tensile properties - Part 2: Test conditions for moulding and extrusion plastics.
7. ISO 899-1:1993, Plastics - Determination of creep behaviour - Part 1: Tensile creep.
8. ISO 6721:1996, Plastics - Determination of dynamic mechanical properties.
9. ISO 3167:1993, Plastics - Multipurpose test specimens.
10. B.C. Duncan and P.E. Tomlins. Measurement of strain in bulk adhesive testpieces. NPL Report DMM(B)398, October 1994.
11. ISO 6721-4:1996, Plastics - Determination of dynamic mechanical properties. Part 4: Tensile vibration - non-resonance method.
12. D.F. Adams and D.E. Walrath. J. Composite Materials, vol 21, 1987, pp 494-507.
13. B.C. Duncan and G.D. Dean. Test methods for determining shear property data for adhesives suitable for design. Part 1: Notched-beam shear (Iosipescu) and notched-plate shear (Arcan) methods. NPL Report No CMMT(B)56, 1996.
14. M. Arcan, Z. Hashin and A. Voloshin. Experimental Mechanics, vol 18, 1978, pp 141-146.
15. R. Thomas and R.D. Adams. Test methods for determining shear property data for adhesives suitable for design. Part 2: The torsion method for bulk and joint test specimens. Report No 7 March 1996. (Work carried out in the Mechanical Engineering Department of Bristol University in connection with the Project Basic Mechanical Properties of Adhesives for Design).
16. A. Nadai. Plasticity, A mechanics of the plastic state of matter, McGraw-Hill, New York, 1931, pp 128-130.
17. G.D. Dean and B.C. Duncan. Correlation of modulus measurements on adhesives using dynamic mechanical and constant strain rate tests. NPL Report No CMMT(B)34, 1996.



1a) spacers  
Suitable for viscous adhesives.



1b) U - shaped frame  
Recommended for adhesives which are viscous when cast but which may flow when curing at an elevated temperature.



1c) closed frame  
Necessary for low viscosity adhesives

**Figure 1: Mould designs for preparing sheets of adhesive.**

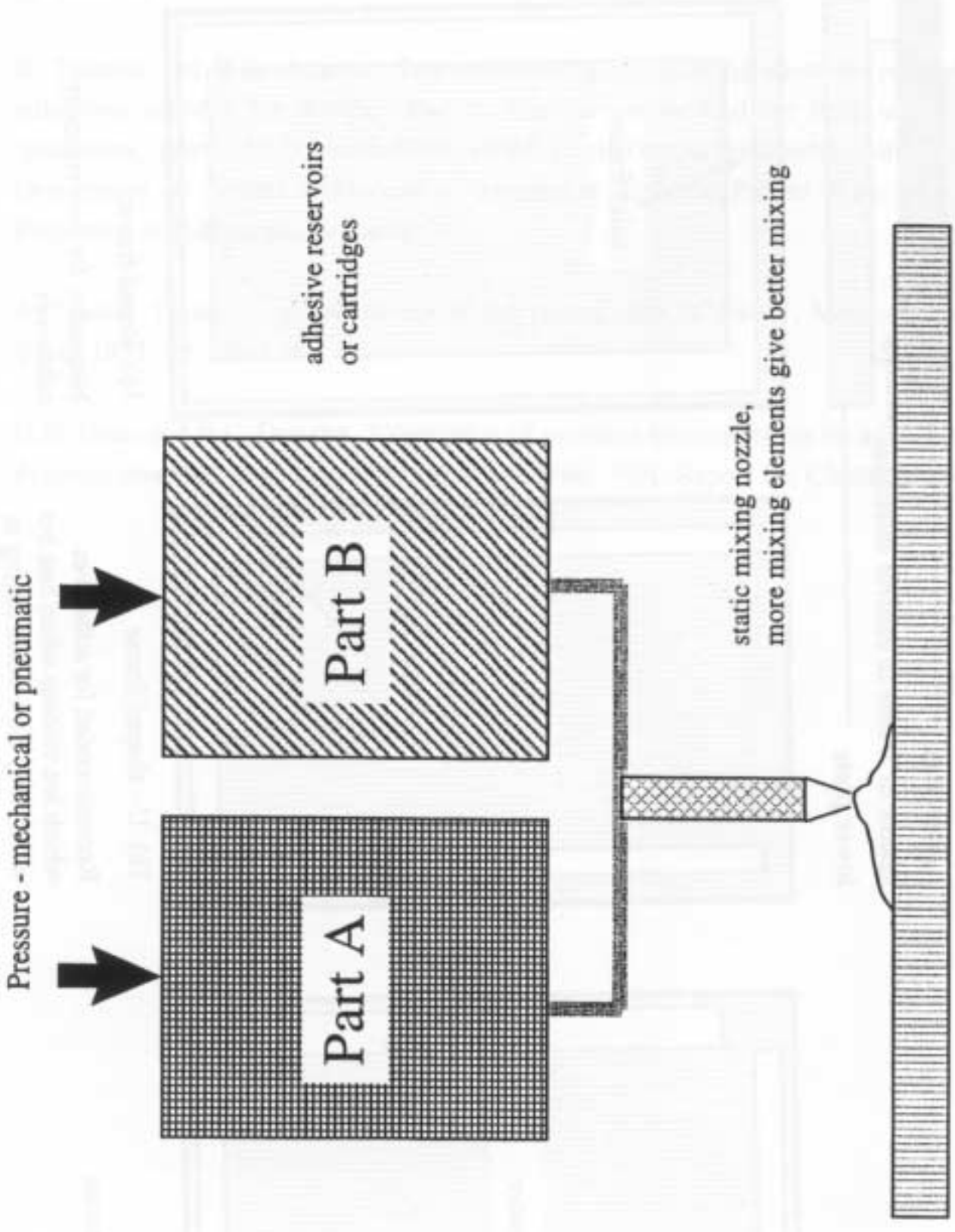


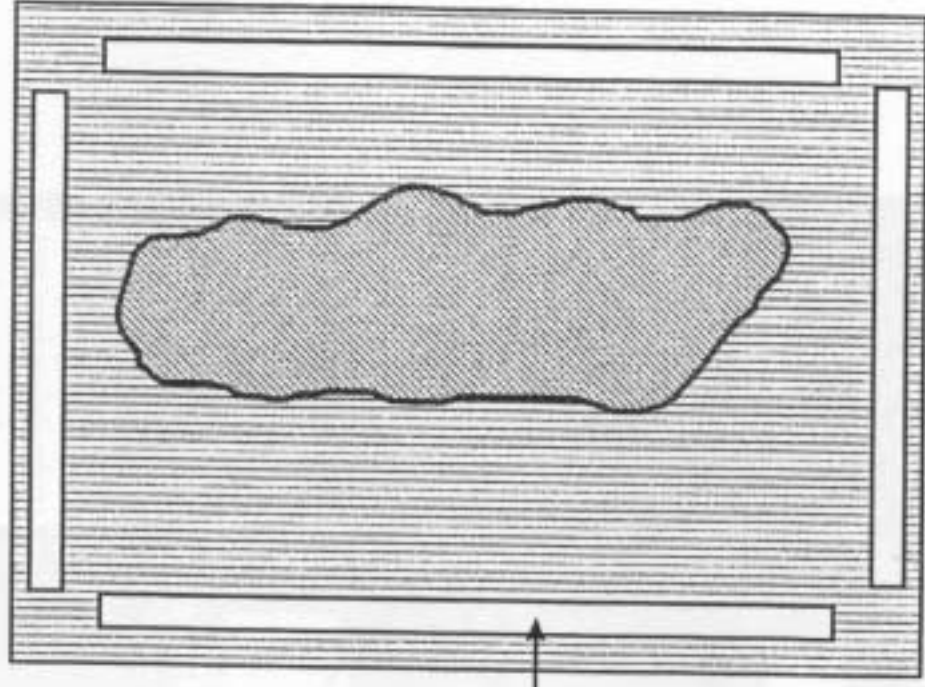
Figure 2: Schematic of equipment for mixing and dispensing 2-part adhesives.

3a) The nozzle is drawn along the length of the plate in a single motion without removing the tip from the adhesive. The height of the cast should be greater than the spacers.



3c) The top plate is lowered from one side thereby spreading the adhesive. Pressure is then applied to maintain a uniform thickness during cure.

3b) When sufficient adhesive has been dispensed, the nozzle is removed and the top plate is then applied.



**Figure 3: Filling the mould. The diagrams refer specifically to situations with a viscous adhesive.**

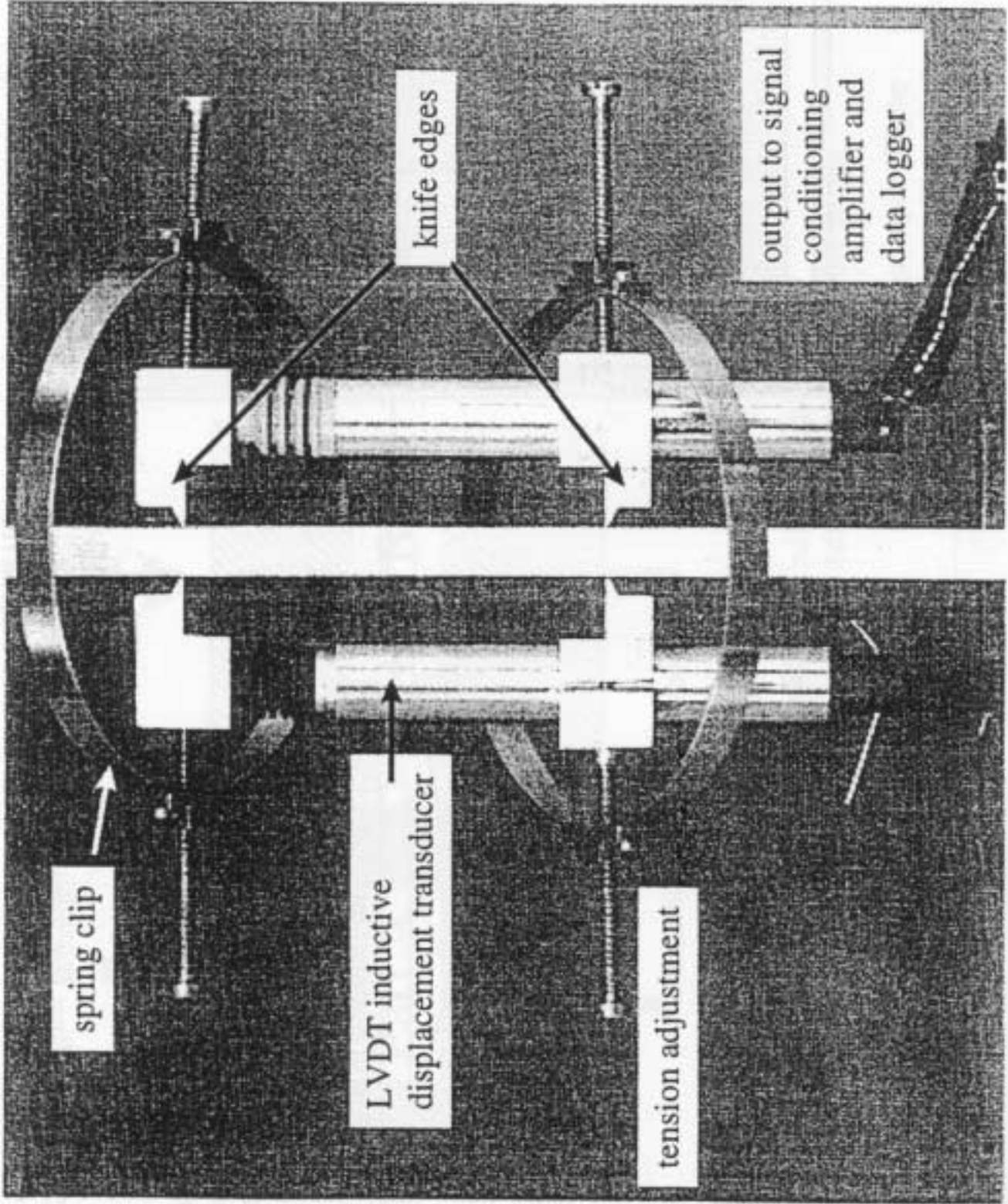


Figure 4: Typical contacting extensometers having good accuracy and precision.

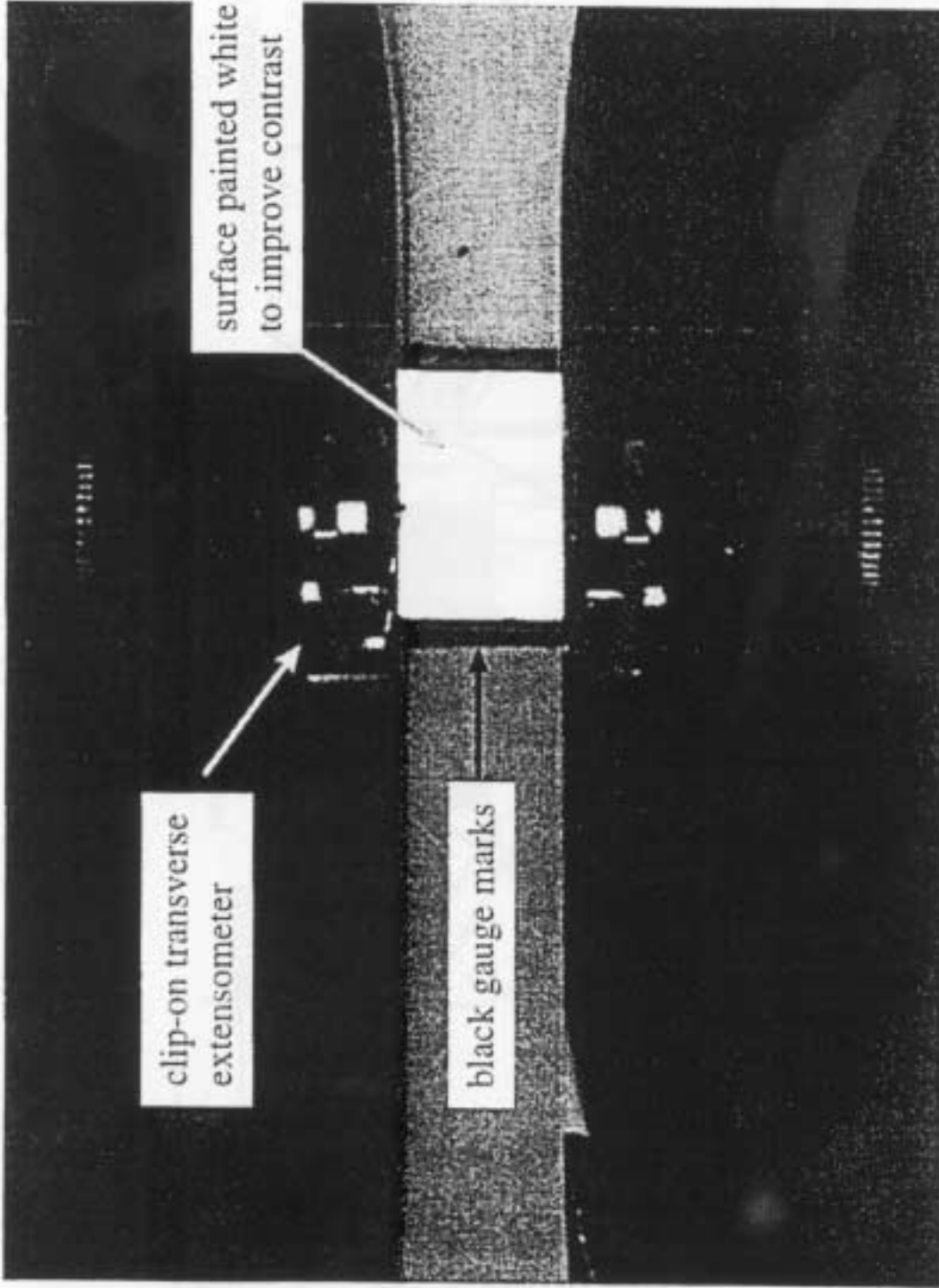


Figure 5: Specimen marked for video extensometry measurements. A clip on transverse extensometer can be used to make lateral contraction measurements.

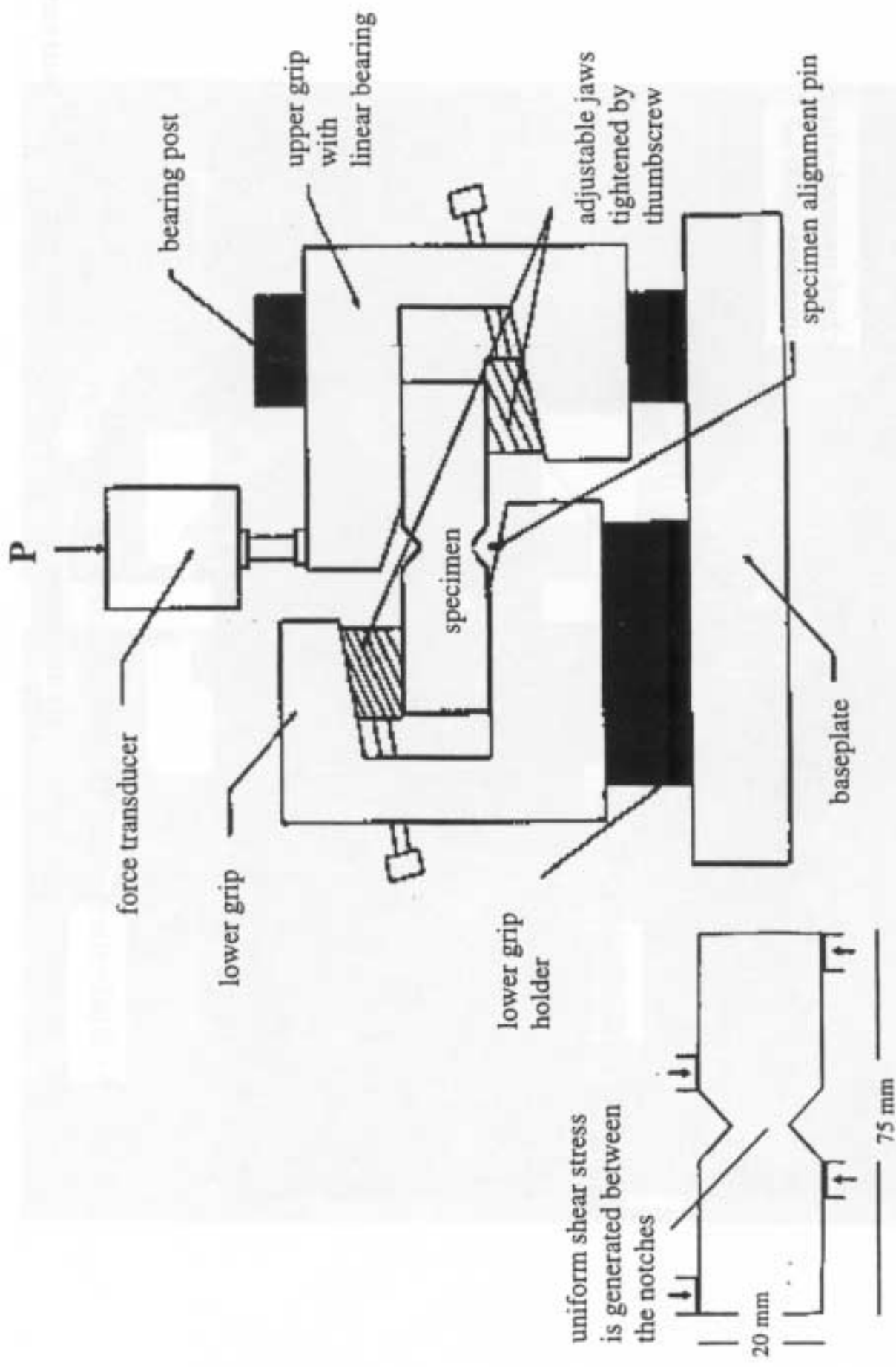


Figure 6: Notched beam shear (Iosipescu) method test fixture.

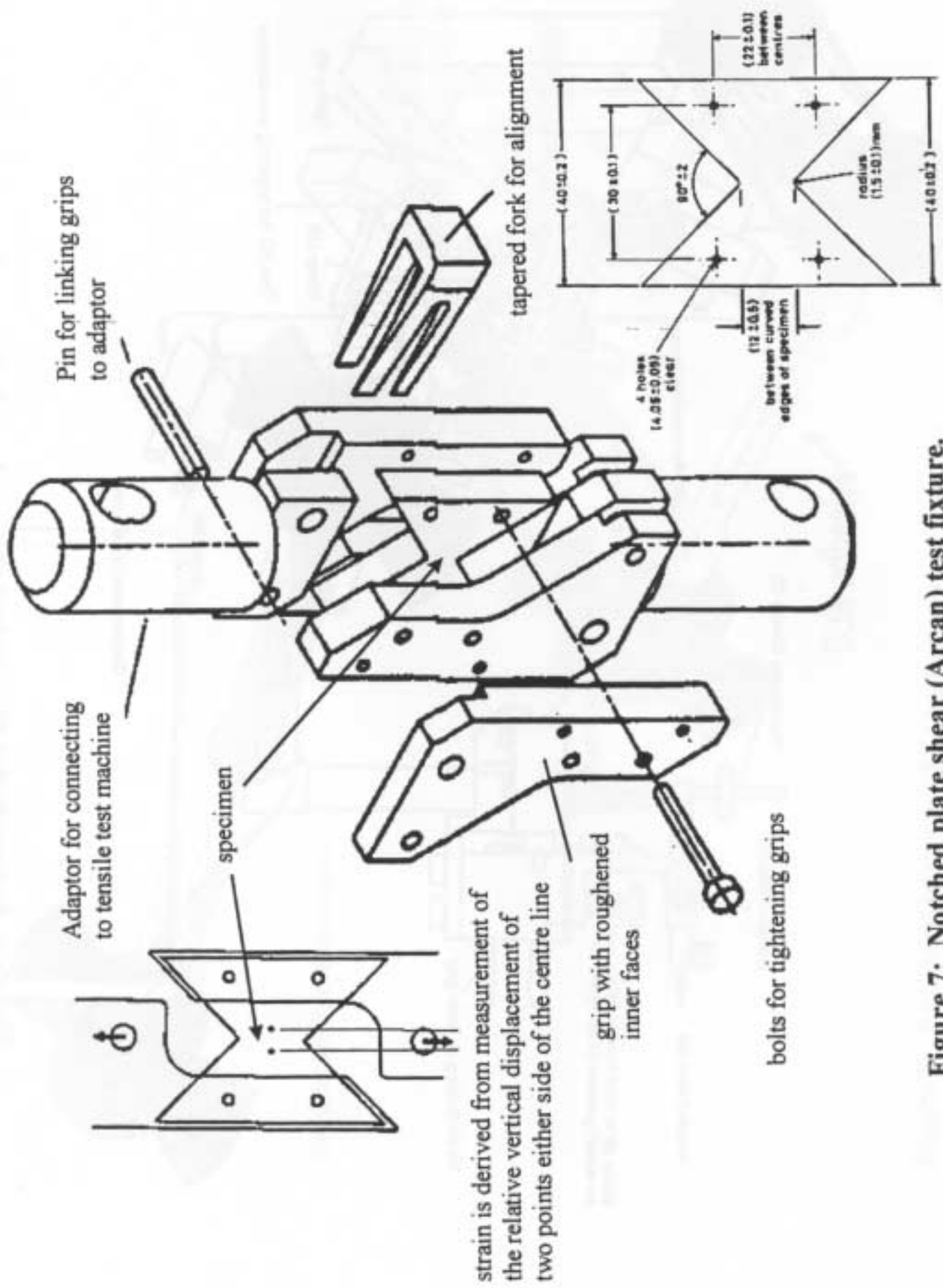


Figure 7: Notched plate shear (Arcan) test fixture.

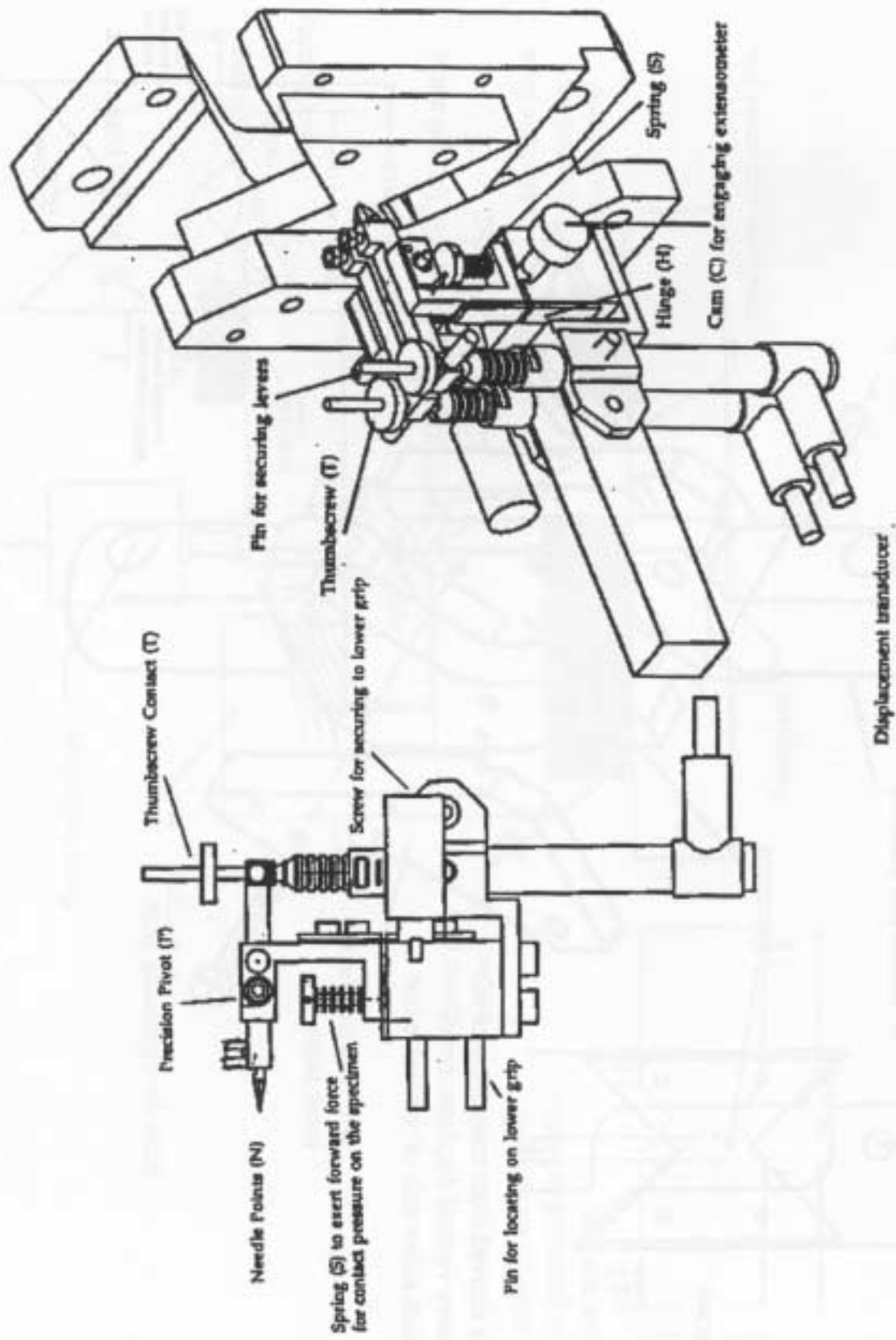


Figure 8: Extensometer for notched plate and notched beam shear test methods.

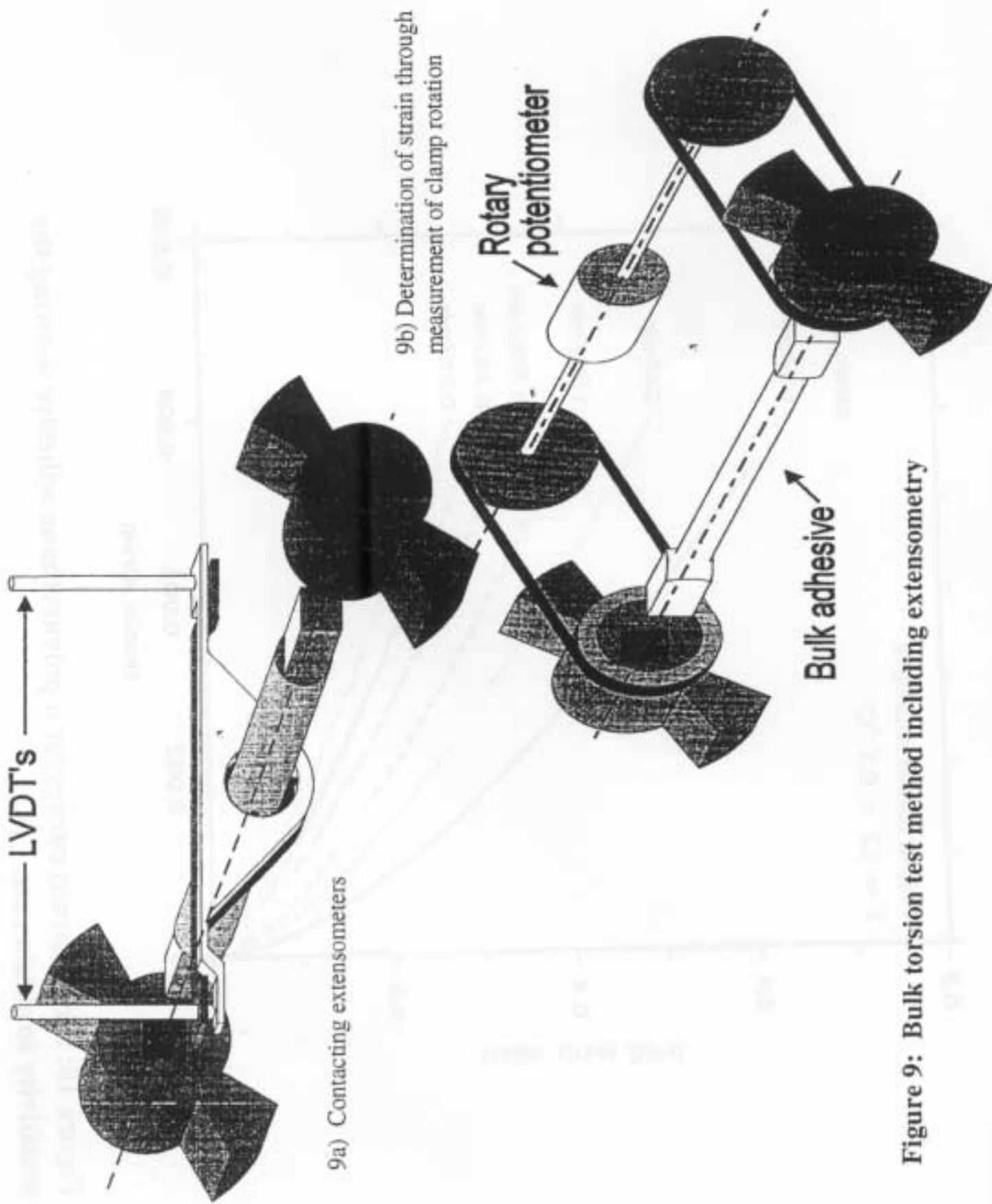


Figure 9: Bulk torsion test method including extensometry

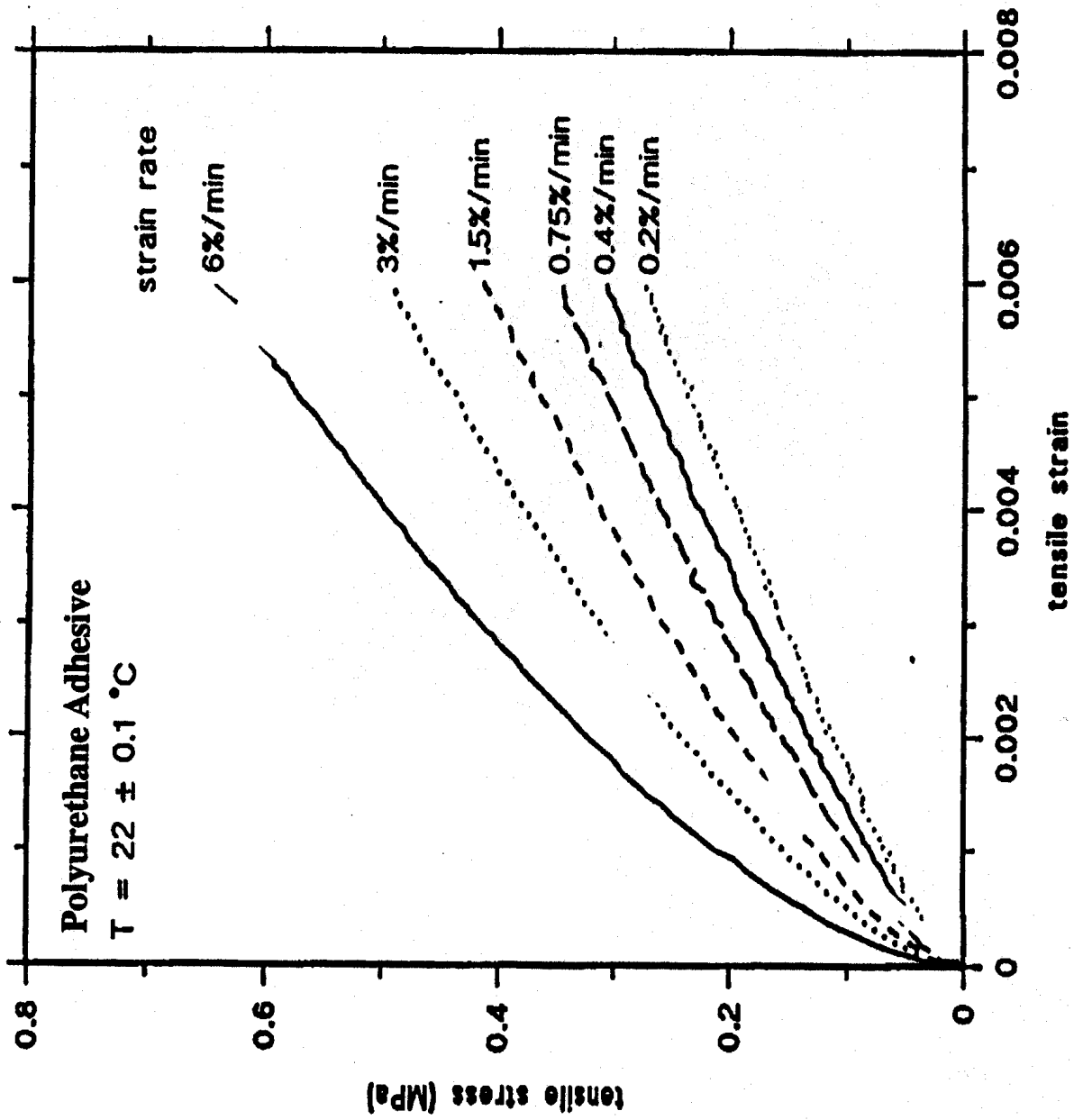


Figure 10: Stress-strain curves for a polyurethane adhesive showing the sensitivity to strain rate.