International Standard 974

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Plastics — Determination of the brittleness temperature by impact

Plastiques — Détermination de la température de fragilité au choc

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 974 was developed by Technical Committee ISO/TC 61, *Plastics*.

It was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces ISO Recommendation R 974-1969, which had been approved by the member bodies of the following countries :

Australia	Finland	Netherlands
Austria	Greece	New Zealand
Belgium	Hungary	Romania
Canada	India	Spain
Chile	Ireland	Sweden
Colombia	Israel	Turkey
Czechoslovakia	Italy	United Kingdom
Egypt, Arab Rep. of	Japan	USA

The member bodies of the following countries had expressed disapproval of the document on technical grounds :

France
Germany, F. R.
Switzerland
USSR

Plastics — Determination of the brittleness temperature by impact

1 Scope and field of application

1.1 This International Standard specifies a method for the determination of the temperature at which plastics that are not rigid at normal ambient temperature exhibit brittle failure under specified conditions of deformation. A supplementary technique uses notched specimens and these show brittle failure at a much higher temperature. The method takes account of the statistical nature of brittle failure and makes provision for the testing of sufficient specimens to permit calculation of the brittleness temperature on a statistical basis.

1.2 The "brittleness temperature" test was originally developed to measure the temperature at which a polymer ceased to be flexible and became "glasslike". Because of the statistical nature of these failures, "brittleness temperature" is now defined as given in 3.1. The method specified in this International Standard establishes the temperatures at which there is a 50 % chance of failure in unnotched or notched specimens. It has been found useful for specification purposes, although it does not necessarily relate to the lowest temperature at which the material may be used, since the basic polymer brittleness will be modified by any orientation produced during fabrication, by thermal history, and by the stress system applied, especially by the rate of impact. The typical precision of ± 5 °C should be recognized in establishing values used in material specifications.

2 References

ISO 175, *Plastics — Determination of the effects of liquid chemicals including water.*

ISO 291, *Plastics — Standard atmospheres for conditioning and testing.*

3 Definitions

3.1 brittleness temperature : The temperature at which there is a 50 % probability of failure in a specimen when tested by the method specified. It may be designated t_{50}.

3.2 testing speed : The relative velocity between the striking edge of the test apparatus and a test specimen held in the specimen clamp.

4 Principle

Bending a cantilever specimen through 90° around a mandrel of specified radius, at a constant testing speed in an inert medium, the temperature of which is accurately known and precisely controlled.

5 Apparatus

5.1 Testing machine, consisting of a clamping device to hold the test specimens, a striking edge, and a mechanical arrangement appropriate to ensure that these are maintained in proper relation to each other and that the striking edge moves at a constant testing speed relative to the test specimens.

NOTES

1 Details of the striking edge and clamping device are shown in figures 1 and 2, and a photograph of the clamp with mounted specimens is shown in figure 3.

2 Commercial apparatus is available meeting the requirements of this sub-clause, in which the striking edge is driven by a motor, by a solenoid, by gravity or by a spring. In all cases it should satisfy the definition given in 3.2 under actual conditions of test.

The principal dimensions of the apparatus shall be as follows :

a) radius of striking edge : 1,6 ± 0,1 mm;

b) radius of lower jaw of clamping device : 4,0 ± 0,1 mm;

c) separation between point of impact of striking edge and clamping device : 3,6 ± 0,1 mm;

d) clearance between outside of striking edge and clamping device : 2,0 ± 0,1 mm.

The testing speed shall be 200 ± 20 cm/s at impact and during at least the next 0,5 cm of travel.

5.2 Temperature indicator, used with a thermocouple or an equivalent thermometer, capable of covering the temperature range being tested and accurate to within ± 0,5 °C.

The thermocouple, constructed of copper and constantan wires of diameter 0,2 to 0,5 mm, welded at their junction (or the thermometer bulb), shall be placed as near to the test specimens as possible.

5.3 Heat transfer medium. A liquid or gaseous heat transfer medium, preferably liquid, which remains fluid at the test temperature and which does not appreciably affect the material being tested shall be used. The medium shall be maintained at the test temperature to within ± 0,5 °C.

NOTE — Given that the time of contact between the liquid and the plastics specimens is short and the temperature low, the use of a methanol/solid CO_2 mixture has been found suitable for most plastics. This mixture can be used successfully down to −76 °C. Below this region other heat transfer media are needed, for example, silicone oils, dichlorodifluoromethane/liquid nitrogen, or an air bath.

Should any doubt exist regarding the inertness of the plastics to the mixture used, selected physical properties should be measured before and after 15 minutes exposure at the highest temperature used (see ISO 175). The values obtained should not differ significantly.

Dimensions in millimetres

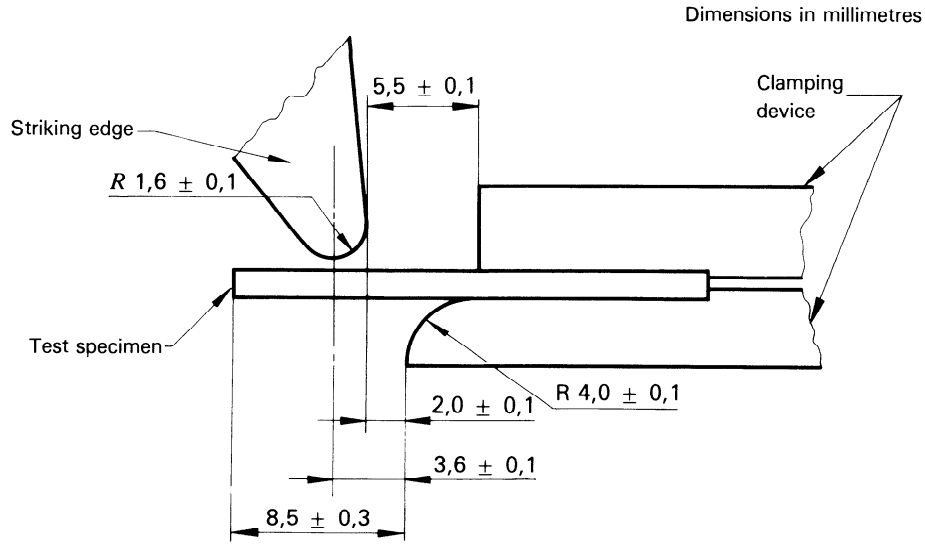

Figure 1 — Dimensional details of striking edge and clamping device
(Positioning of unnotched test specimen)

Dimensions in millimetres

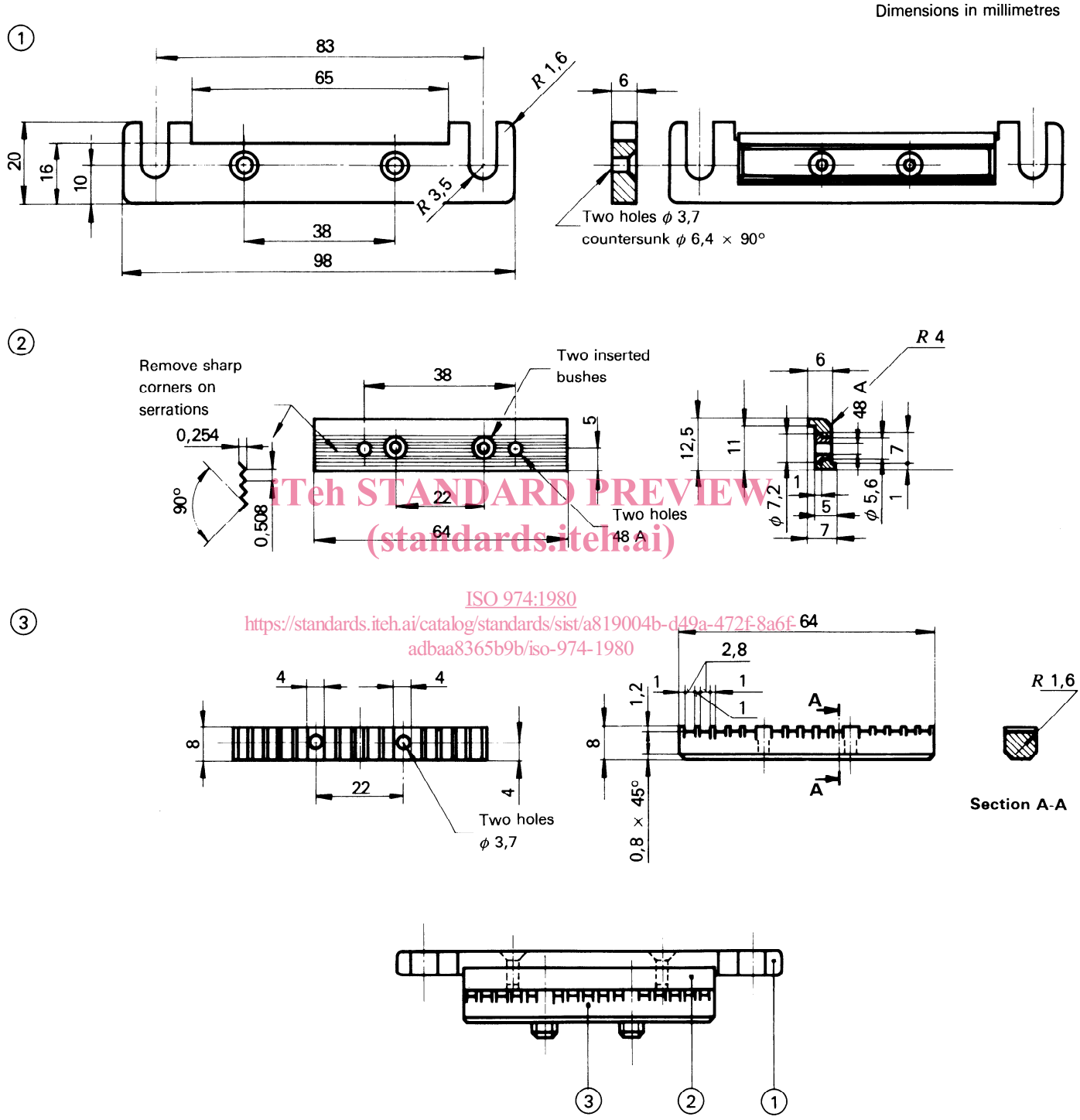

Figure 2 — Details of one form of clamp meeting the requirements of 5.1

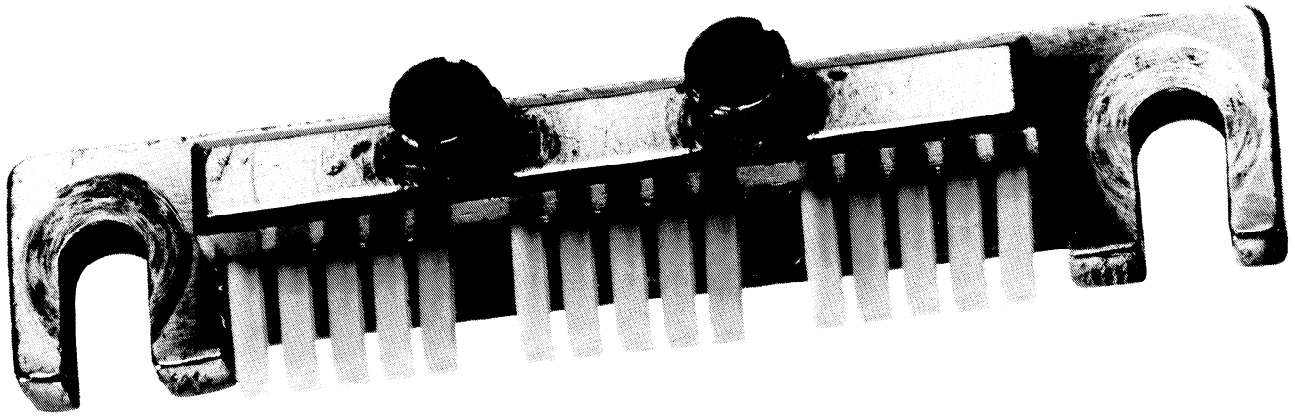

Figure 3 — Assembled clamp with test specimens

6 Test specimens

6.1 For many polymers, the results of this test depend to a large degree on the conditions used for sample moulding and on the mode of specimen preparation (three methods for moulding polyolefins are described in the annex); the cleaner the edges and the freer the specimens from accidental notches, the lower will be the observed brittleness temperature.

It is therefore essential that the specimens be prepared in a reproducible way. A razor blade or other sharp tool shall be used to cut the specimens, preferably in a single smooth stroke. Die-cut specimens are not recommended. Although it is possible to prepare satisfactory specimens by hand, it is strongly recommended that an automatic method be used, since this enables accurately reproducible specimens to the prepared, even by semi-skilled operators, from laboratory to laboratory. Whatever method is used, it is essential that the cutter be inspected frequently and maintained in the sharpest possible condition.

NOTE — For the use of an automatic cutter for specimen preparation see Bestelink, P.N., and Turner, S., *Low-temperature brittleness testing of polyethylene.* ASTM Bulletin No. 231, 68 (1958).

6.2 Test specimens 20,00 ± 0,25 mm long by 2,50 ± 0,05 mm wide and 1,6 ± 0,1 mm thick shall be cut from a test sheet. They can be cut conveniently from a strip of the required thickness and 20,00 ± 0,25 mm wide by slicing off the required widths, preferably automatically.

6.3 Where a notch is desired, a clean cut shall be made at about the middle of one of the 20 mm × 1,6 mm sides of the specimen at right angles to its long axis and 0,40 ± 0,02 mm deep using a sharp razor blade or, preferably, an automatic cutter machine (see note to 6.1).

NOTE — For some materials, particularly polyethylene, it may be desirable to use notched specimens, except when the test is being used to assess the effects of ageing. The presence of the notch has the double effect of reducing the scatter of results and of raising the brittleness temperature in polyethylene from the vicinity of about − 100 °C into a temperature region which is more easily reached experimentally, i.e. above − 70 °C. [See Hoff, E.A.W., and Turner, S., *A study of the low-temperature brittleness testing of polyethylene,* ASTM Bulletin No. 225, 58 (1957).]

7 Conditioning

The test specimens shall be conditioned prior to testing, but (if notched) after notching, in accordance with ISO 291.

8 Procedure

8.1 Mount the test specimens firmly in the clamping device and secure the latter in the testing machine. When notched specimens are used, the notch shall be in the side of the specimen, not in the top or bottom faces, and shall be positioned on a line tangent with the bottom of the curve of the mandrel (the lower edge of the clamping device).

8.2 Bring the specimens to equilibrium at the test temperature. With liquid heat-transfer media, this requires 3 min, with gaseous media, allow 20 min.

8.3 Actuate the testing machine, bending the specimens around the mandrel.

8.4 Remove the specimens from the cold bath and note the number which have failed. Failure is indicated by separation into two or more pieces.

8.5 Carry out tests at four or more temperatures in the range including 10 % to 90 % failure. (0 % and 100 % failure are not useful in determining t_{50} by the graphical method given in 9.1).

8.6 Test at least 100 specimens. If four temperatures are used, test at least 25 specimens at each temperature. If more temperatures are used, fewer specimens may be tested at each temperature but never fewer than 10 at any one temperature.

9 Expression of results

The temperature t_{50} may be determined by either of the following methods.

9.1 Graphical method

Plot the percentage failure at any temperature against the test temperature on arithmetic probability paper and draw the best straight line through the results. Read the brittleness temperature from the graph where this line intersects that for 50 % probability.

9.2 Method by calculation

The temperature t_{50} may be calculated by using the following formula :

$$t_b = t_h + \Delta t \left(\frac{S}{100} - \frac{1}{2} \right)$$

where

t_b is the brittleness temperature, in degrees Celsius;

t_h is the highest temperature, in degrees Celsius, at which failure of all the test specimens occurs (correct algebraic signs shall be used);

Δt is the uniform temperature increment, in kelvins, between successive tests;

S is the sum of the percentage of failures at each temperature (from a temperature corresponding to no failures down to and including the temperature t_h).

10 Test report

The test report shall include the following particulars :

a) reference to this International Standard;

b) complete identification of the material tested, including type, source manufacturer's code designation, form in which supplied, and previous history;

c) brittleness temperature to the nearest 1 °C;

d) whether notched or unnotched test specimens were used;

e) the method of preparation used for the test sheets and test specimens;

f) the conditioning procedure used, including time elapsed since moulding or annealing;

g) the heat transfer medium used.

Annex

Polyolefin sample preparation

A.1 General

The test sheet is compression moulded using one of the three procedures described in this annex, the choice of procedure depending upon the material :

Polymer	Procedure
Low-or medium-density polyethylene	A or C
High-density polyethylene Polypropylene	B or C

A.2 Procedure A (oven annealing)

The required sheets shall be formed in a simple three-part mould. This shall consist of a chase having an opening of suitable size for the desired test sheet dimensions and sufficient depth to produce sheets 1,6 ± 0,1 mm thick, plus two smooth metal backing plates at least 1 mm thick and large enough to cover the chase. Two sheets of clean aluminium foil 0,05 to 0,2 mm thick and of sufficient size to cover the chase are also required.

Clean the backing plates and the aluminium foil with solvent and dry them thoroughly. Liquid release agents or waxes shall not be used. Cover one plate with a sheet of foil and place the chase on tops of this assembly, thus forming the cavity.

Although pellets or granules may be charged directly into a cavity, milled crêpe is recommended since milling serves to erase pre-existent crystalline structure. To prepare the crêpe, the mill rolls shall be hot enough to flux the plastic but not so hot as to cause it to drip. Slash or turn the crêpe frequently to promote mixing. Subsequent compression moulding is facilitated if the rolls are adjusted to produce a crêpe which is as thin as possible. Ethylene plastics ordinarily shall not be milled for more than 5 min to minimize oxidative and thermal changes.

Load the mould with enough material to form a sheet which fills the cavity completely and add an excess of 2 % to 10 % for flash. Level the charge and cover it first with a sheet of clean dry foil and then with the second backing plate. Place the assembly in a press between platens preheated and maintained at a temperature high enough to flux the material and to ensure that it adheres well to the aluminium foil. Platen temperatures of 150 to 180 °C may be required depending upon the characteristics of the material being moulded.

Using low pressure, close the press enough to establish good contact between platens, plates and material and hold in that position for about 5 min to flux the material. Then apply sufficient pressure to form a smooth, void-free sheet and maintain this pressure for 5 min. After the moulding has been formed, cool the platens at any convenient rate. When the mould has been cooled to 50 °C or less, take it out of the press and pry the backing plates off without disturbing the aluminium foil, which shall be adhering tightly to the chase and sheet and shall appear smooth and free of dimples or sink marks.

If the sheet is not smooth and free of voids, it shall be pressed over again. In that event, cut the sheet into at least four pieces and load them into the mould as previously described together with enough additional material, preferably crêpe, to make up for flash lost in the first moulding. Repeat pressing as described in the preceding paragraph.

Place the assembly consisting of chase, sheet and adhering foil on a thin plate on a rack in an oven maintained at an appropriate temperature (140 to 145 °C for low-density, 150 to 155 °C for medium-density, and 155 to 175 °C for high-density polyolefins). Assemblies may be stacked with spacer plates between them, in which event the ability of the oven to heat all of them to the required temperature within 1 h shall be checked by means of suitably placed thermocouples.

After the assemblies have been stacked in the oven, heat them at the specified temperature for at least 1 h. Then cool them in the oven at constant rate of 5 °C per hour until they attain a temperature not greater than 50 °C, at which point they may be removed and cooled to standard laboratory temperature (20 to 27 °C) on the bench. After cooling strip the foil off and press the sheet out of the chase. Then cut the required test specimens from the sheet

Slow cooling may embrittle some ethylene plastics, rendering them unsuitable for most tests with the exception of density.

A.3 Procedure B (quench cooling)

Prepare the sheets as in Procedure A, but after the assembly has been heated for 1 h in the oven at the specified temperature, remove it while hot, place it in a wire tray, and quickly lower it into a bath containing water maintained at a temperature between 15 and 20 °C. Quenching shall be accomplished within 30 s after the assembly is removed from the oven, so the cooling tank should be located near the oven. Also, only one assembly at a time shall be immersed in the tank unless it has been established that test results are not affected by the presence of more than one assembly.

After being immersed for at least 15 min, remove the cooled assembly from the tank, strip the foil off, and press the sheet out of the chase.

Cut the test specimens from the sheets. Since polyolefin plastics quenched in this manner may be unstable, they shall be tested only after a standardized conditioning period at room temperature. 18 h is recommended.

A.4 Procedure C (press cooling)

Except as modified by the following instructions mould the sheet with the apparatus described and in accordance with the directions given in Procedure A.

In preparing the assembly, uncoated cellophane or polyester film may be used instead of aluminium foil if suitable and preferred. Before the assembled mould is inserted in the press, heat the platens to a temperature suitable for the plastic involved (for example 180 °C for high-density polyethylene or 160 °C for polyethylenes of low and medium density). Then mould the sheet according to the instructions of Procedure A. After the sheet has been formed under heat and pressure for 5 min, circulate cool water through the platens to cool them and the mould assembly at a rate of 16 ± 2 °C per minute. Measure the rate of cooling either continuously or at intervals not exceeding 30 s by means of thermocouples or thermometers inserted in the platens. If a thermometer is used, its bulb shall be wrapped with metallic wool to ensure good contact. Record the measured temperatures and plot them against time. Report the slope of the best straight line drawn through the plotted points as the cooling rate. Control the cooling rate within the specified limits by regulating the flow of water through the platens.

When the platens have been cooled to a temperature not higher than 40 °C, remove the mould assembly from the press then, if necessary further cool it to standard laboratory temperature (20 to 27 °C) on the bench. Remove the backing plates and strip adhering foil or film from the sheets. Following that press the sheet out of the chase and cut the required test specimens.