

A Combined Cryostat/Oven and Specimen Transfer Device for Tensile Testing in the Range 83 to 473° K

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Received 29 September 1965

Design details and performance of a combined cryostat and oven for an Instron TT-CM universal testing machine are described. The cryostat is suitable for tensile testing in the range 83 to 473° K and has a test chamber 5.5 in. × 5.5 in. and 20 in. deep which could be used for testing specimens 7 in. wide. Special attention is drawn to the ease of specimen insertion which may be performed while the cryostat is cold. The temperature within the test chamber is controlled, by admitting dry air, to $\pm 1^\circ \text{C}$ of any desired test temperature for periods of at least 20 min.

A specimen transfer device is also described for transferring a pretreated specimen at -26°C to the cryostat with a temperature rise of less than 1°C . The transfer device provides a method of rapid specimen insertion once the cryostat is cold.

1. Introduction

The need to perform mechanical tests on materials at temperatures other than ambient is common to many branches of science. The tensile test is the most common mechanical test, and to obtain a complete range of test temperatures, provision must be made for the specimen to be enclosed and for a means of temperature control within the enclosure. Such an enclosure was required for an Instron TT-CM to test polymers. An arbitrary range of -200 to $+200^\circ \text{C}$ was selected to cover a wide range of polymer testing. The lower limit meant that some form of cryostat was required.

Several cryostats have been designed for tensile testing machines. Some of these have been reported in the literature although others probably exist. Those reported have been designed to cover a lower range (4 to 300° K) than that required for testing polymers. Chamberlain [1] describes a vacuum-insulated vessel capable of temperatures down to 4° K. Apparently no means of temperature control for any period is supplied other than at the cryogenic liquid temperatures. McClintock and Warren [2] describe apparatus to cover the range 4 to 300° K.

Temperature control at intermediate temperatures is obtained by thermal equilibrium between electrical heaters around the specimen and the surrounding cryogenic liquid.

The desire to test at 4° K necessitates a small testing chamber and a small specimen. Previous designs have required the cryostat to be partially dismantled for each new specimen insertion thus requiring a lengthy cooling-down process with each specimen.

The cryostat described here was designed to reach temperatures approaching that of liquid nitrogen (78° K) as the lower limit and 473° K as the upper limit, thus covering the useful range of polymer testing. The current interest in fracture mechanics in polymers [3] requires wide specimens. In addition, polymers are highly temperature-sensitive and any cryostat for testing them must be capable of control to within $\pm 1^\circ \text{C}$ at any desired test temperature. The advantages of the present design are versatility, the large test chamber, the ease of inserting a new specimen while the cryostat is cold, and the method of temperature control.

The resulting design uses a variety of coolants (liquid nitrogen, solid carbon dioxide, carbon

dioxide in methanol or other mixtures) to obtain a suitable temperature below the required test temperature. The final temperature adjustment is obtained by admitting dry air at ambient temperature to the test chamber through a small needle valve. The relatively warm air admitted provides a source of heat input which, when balanced by the heat flux to the surrounding cold liquid, establishes thermal equilibrium at any desired temperature. Using air instead of electrical heaters for the heat input has certain advantages. It provides forced mixing, instead of the convection currents obtained with electrical heaters, thus eliminating hot spots and establishing constant temperature throughout a large testing chamber. It also provides rapid control unlike electrical heaters which have some degree of thermal inertia depending on their size.

To minimise the cryogenic liquid consumption and the time taken to test a specimen, the cryostat was designed so that the chamber could

be opened and a new specimen inserted in about 30 sec without the chamber's temperature rising by more than 25°C when cooled to -190°C . This permitted a test every 20 min when operating at this temperature. The temperature rise and time to regain test temperature after insertion of a new specimen depend on the operating temperature.

2. The Cryostat

The cryostat is shown schematically in fig. 1. It is fixed underneath the beam of the Instron by screwing the top Duraluminium plate (A) to the beam. Stainless steel straining bars (B) are screwed into this plate and pass through the top insulation into the test chamber. The test chamber is 5.5 in. \times 5.5 in. and 20 in. deep permitting sheet specimens 7 in. wide to be used for fracture mechanics investigations or 1000% extensions on rubber specimens originally 1.5 in. long. The ends of the straining bars are con-

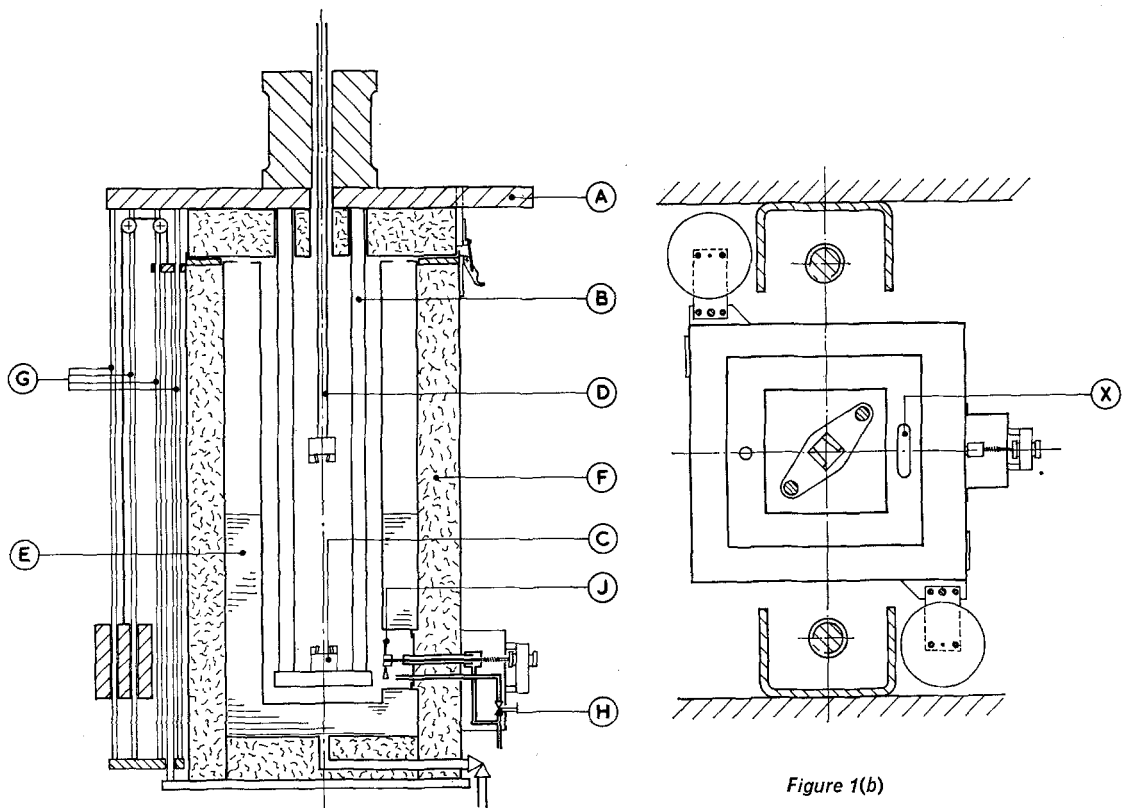


Figure 1(a)

Figure 1(b)

nected by a stainless steel block to which is fixed one half of a dovetail connexion (C) forming part of a specimen quick release system. The upper specimen connexion is another dovetailed block suspended from the appropriate load cell by a stainless steel rod (D). A range of these bars permit specimens of varying length to be used.

The test chamber is surrounded by a liquid bath (E) on all four sides and underneath, 1.5 in. wide and fabricated from aluminium sheet. A hole in the top of the bath allows the liquid nitrogen to be poured in through a funnel when the cryostat is open. An elongated hole (X) on the opposite side acts as a vent during pouring or alternatively allows crushed solid carbon dioxide to be placed in the bath should this be used as the coolant. The liquid bath is surrounded by a 2 in. layer of insulant (F) contained by aluminium cladding. The insulant used here and in the top insulation pad is Perlite (expanded aluminium silicate) but is not under vacuum. The heat loss with this insulant is much reduced when used under vacuum but creates the difficulties associated with vacuum equipment construction.

The liquid bath with surrounding insulant moves up and down around the straining bars and specimen on aluminium guide bars (G), is counterbalanced for ease of operation, and is locked to the top insulation pad in the raised position by four case clips. The counterbalance weights are each split into three, and either one, two, or three weights may be used depending on the quantity of liquid in the bath.

The temperature within the test chamber is controlled by admitting warm air. The air is initially taken from a 100 lbf/in.² air line, passed through a pressure regulating valve capable of supplying air from 0 to 30 lbf/in.², and is then dried by passing it through self-indicating silica gel. The dried air is passed by means of a flexible PVC tube to a small needle valve (H) mounted on the cryostat and into the bottom of the chamber.

The warm air is only used for temperature control purposes. On other occasions, to prevent the air becoming stratified within the chamber, a small fan (J) is used situated near the bottom of the test chamber and driven by a small electric motor mounted externally. The fan spindle runs through a copper tube which passes through the insulant and liquid bath as does the air supply tube. Bearings are provided at each end of the spindle tube. Initially these bearings

seized very rapidly with condensed water vapour from the air. This was overcome by using loose running bearings and a trickle of dry air (1 lbf/in.²) passed around the spindle and out through each bearing.

The cryostat has so far only been used below ambient temperature. For the range 300 to 473° K it is proposed to use the same equipment but add an electrical heater around the walls of the test chamber. Room has been left between the straining bars and the walls for this purpose, but the heater has been omitted to give as much room as possible in the chamber for the specimen transfer device currently used. When used as an oven, the air supply would be used to lower the temperature to the test value and mix the air already within the chamber to provide an even-heat oven.

3. Performance of the Cryostat

About 12 l of liquid nitrogen are required to cool the system down initially to -190°C which takes about 45 min. The difference between this temperature and that of liquid nitrogen (-196°C) is due mostly to the fan and to the warm air entering the chamber through the fan bearing. After the initial cooling-down, approximately 2 to 4 l are required per hour depending on the testing temperature. The choice of coolant depends on the test temperature required. Liquid nitrogen has been found useful for the range -120 to -190°C and a mixture of methanol and water with solid carbon dioxide for the range $+20$ to -40°C . For maximum economy it is best to have the coolant about 10 or 20°C below the desired test temperature.

The warm air provides a rapid method of temperature control. It has been found that -120°C can be reached and stabilised in about 30 sec from -190°C . By manual adjustment of the air valve a temperature of -120°C within $\pm 1^{\circ}\text{C}$ has been maintained for 20 min on 1 litre of liquid nitrogen. A temperature of $-26 \pm 1^{\circ}\text{C}$ has been maintained for 30 min.

Fig. 2 shows examples of the temperature variation within the test chamber. It can be seen that when the air is running, the mixing within the chamber is better than with the fan alone, giving 80% of the chamber with a variation of less than 1°C at -150°C . At -185°C , with the fan alone, 50% of the box is within 1°C of the test temperature. Since the specimen is generally near the bottom of the chamber during test unless large extensions occur, testing is

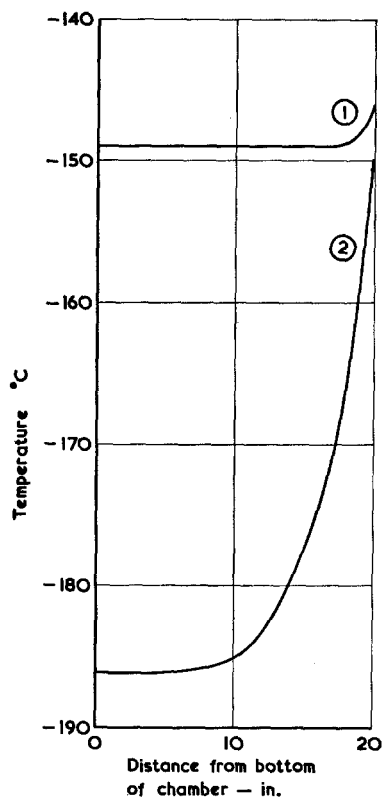


Figure 2

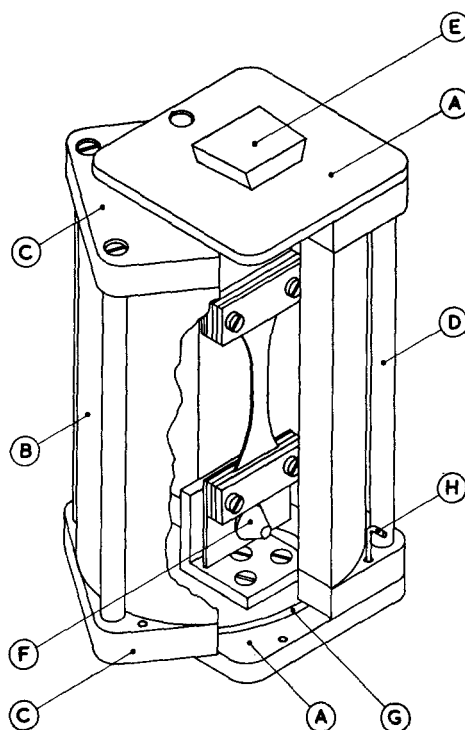


Figure 3

carried out at constant temperature over the whole length of the specimen.

When the cryostat is opened the cold air remains inside the chamber. A new specimen is placed in position in about 30 sec, with the device described later, using oven gloves to prevent burning. The hands do not actually enter the chamber during this process but may come into contact with the exposed straining bars or top of the liquid tank. Opening the cryostat when cooled to -185°C and inserting a warm specimen does not raise the temperature in the chamber by more than 25°C .

4. Specimen transfer device

A further problem not directly connected with the cryostat presented itself in testing rubber. Specimens had to be crystallised for up to 3 days at temperatures of about -26°C before submitting them to test in the cryostat. The pretreatment was carried out in a carefully controlled refrigerator. The specimens had then to be

transferred to the cryostat and brought to the test temperature without their temperature rising more than 5°C . An ordinary insulated box would not suffice; since, if it prevented the temperature rising outside the cryostat, it would prevent the specimen reaching test temperature rapidly within the cryostat. Neither could an insulating box be removed while the cryostat was open, since the specimen would still be partially exposed to room temperature.

The device designed for the transfer is shown in fig. 3 with one half closed and the other open. The need to pretreat specimens frequently occurs and, although designed for rubber specimens, the device can be used for any material and specimens of any shape with only minor modifications. It also provides the quick release mechanism for fitting new specimens once the cryostat has been cooled.

The major components of the device are two hinged and sprung semi-circular insulated sides between end plates (A). For each side a suitable

length of semi-circular expanded polystyrene (B) (available for pipe insulation) is sandwiched between Tufnol end pads (C) and two external aluminium spacing bars (D). The two pairs of pads are hinged together and the lower pair permanently connected to the bottom end plate. To the Tufnol end plates are attached aluminium dovetails (E) externally, and angle brackets and pins (F) internally for the specimen clamps. The movement of the sides is limited by pins moving in a groove (G) in the lower end plate. The sides are held shut by two pins (H) through the lower pads into the end plate. In the shut position, the upper end plate is trapped, but when the pins are withdrawn the sides spring open and the upper end plate is then free to move vertically upwards, so straining the specimen.

To load a specimen, one side is closed to trap the upper end plate, automatically setting a gauge length for the specimen. The specimen, already mounted in clamps, is then dropped into position with the box lying on its side. The clamp pins have tapered heads which prevent the clamp falling off once it is in position. The remaining side is then closed and secured with a brass pin. The specimen is transferred to the cryostat and connected to the loading system by sliding the two dovetails into their respective housings. The cryostat is closed and the test temperature established. When this has been reached outside the transfer device, the two fixing pins are withdrawn externally by two attached cords and the device springs open. A short time is allowed for the specimen to come to the test temperature before the test is performed. The device can be modified for any length of specimen by merely changing the polystyrene and spacing bars.

This technique has been used at various temperatures down to -185°C . Contraction of the device occurs after insertion in the cryostat, placing the load cell under tension. As this takes place, the contraction is backed off by moving the Instron beam slowly upwards.

It has been found that with a polystyrene thickness of 1/2 in., the temperature rise from an initial temperature of -26°C does not exceed 5°C until 10 min has elapsed, when the device is brought into the room at $+20^{\circ}\text{C}$. This gives ample time for a specimen transfer which takes about 1/2 min.

Fig. 4 shows stress/strain curves for crystallised natural rubber obtained at various temperatures using the transfer device and cryostat.

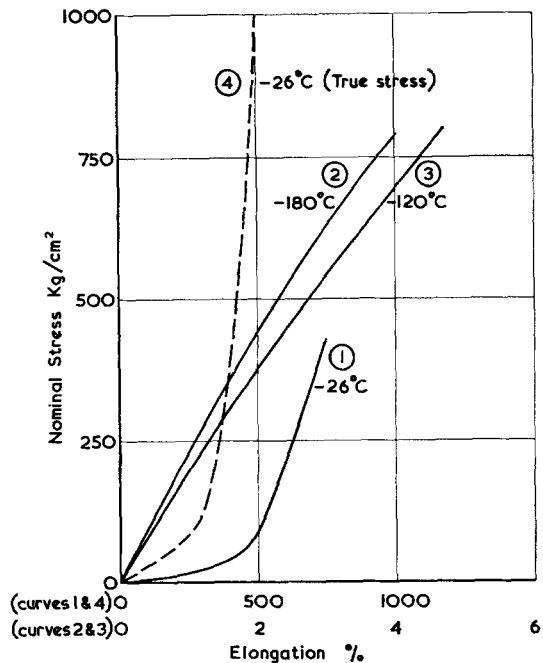


Figure 4

5. Conclusion

Using air instead of electrical heaters for temperature control provides rapid control especially at low temperatures and gives constant temperature through a very large volume. The method of specimen transfer and insertion devised achieves economy of testing time and of cryogenic liquid requirements. The cryostat and transfer device have provided a solution to the testing of polymers in the range 83 to 300°K where fine and rapid temperature control is required. The resulting designs are of a very general nature and have potential applications in many similar and related tensile testing problems.

Acknowledgement

The author wishes to thank Mr D. Middleton and Dr E. H. Andrews for valuable discussions on the mechanical design.

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