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# A System for Vacuum Pouring of Epoxy Tensile and Impact Specimens with a Study of the Behavior of These Specimens at 77 K and 293 K

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Contract No. W-7405-eng-26

FUSION ENERGY DIVISION

A SYSTEM FOR VACUUM POURING OF EPOXY TENSILE  
AND IMPACT SPECIMENS WITH A STUDY OF  
THE BEHAVIOR OF THESE SPECIMENS AT 77 K AND 293 K

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Date Published: April 1978

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## ABSTRACT

The purpose of the investigation described in this report was to establish a suitable technique for vacuum pouring of epoxy test specimens and to study the behavior of these specimens at 77 K and 293 K. A series of tensile and impact tests was conducted using specimens made from the following resins: Epon 828, Epon 871, and Epon curing agent Z. These materials are of general interest to designers of magnets for cryogenic service. Some of the applications that may be considered are: structural support, spacing, electrical insulation, and thermal insulation. The epoxies mentioned above were selected for more extensive testing because they have been used successfully at room temperature in EBT and ORMAK programs in the Fusion Energy Division at Oak Ridge National Laboratory. Liquid nitrogen was chosen over liquid helium because it is less difficult to handle, less expensive, and in most instances physical properties of epoxies seem to change very little from 77 K to 4.2 K. The two main features of the vacuum pouring apparatus are (1) batches can be poured under near-identical conditions, and (2) samples can be handled free from air contamination.

Tests of the specimens were carried out at 77 K and 293 K. The 77 K data indicate that tensile strength increases proportionally with the increase of Epon 871 relative to Epon 828. When the mixture includes more than 90% Epon 871, impact testing at 293 K becomes practically impossible due to the rubbery condition of the material. However, when tested at 77 K, this same mixture evinces high tensile strength. When optimum data are sought over a wide range of temperatures, 77 K to 293 K, it appears that a mixture of 70% Epon 871, 30% Epon 828 with 13 pph of curing agent Z or 50% Epon 871, 50% Epon 828 with 15 pph curing agent Z offers the best compromise in tensile strength, modulus of elasticity, and impact resistance.



## 1. INTRODUCTION

The purpose of the investigation described in this report was to establish a suitable technique for vacuum pouring of epoxy test specimens and to study the behavior of these specimens at 77 K and 293 K. A series of tensile and impact tests was conducted using specimens made from the following resins: Epon 828, an epoxy resin with a molecular weight affording a moderate room temperature viscosity; Epon 871, an epoxy resin especially designed for increased flexibility; and Epon curing agent Z, a liquid of approximately 20 poise viscosity, which may be blended with liquid Epon resins at room temperature.<sup>2</sup>

## 2. MIXTURES

Five different mixtures of Epon 828, Epon 871, and curing agent Z were used in tensile and impact tests. The mixtures are as follows in Table 1.

Table 1. Resin mixtures

Mixture identification	Epon 828	Epon 871	Curing agent Z
A7135G-20	100%	0%	20 pph
A7135G-22	75%	25%	17.5 pph
A7135G-24	50%	50%	15 pph
A7135G-26	30%	70%	13 pph
A7135G-28	0%	100%	10 pph

## 3. SPECIMEN PREPARATION

Appropriate amounts of each epoxy and curing agent Z were weighed in separate containers. The individual containers were placed in a desiccator, outgassed until entrapped air was pumped out (down to 300-500 torr), and then brought up to atmospheric pressure using nitrogen gas. The components were then poured very slowly down the side

of a single container so as to avoid entrapping air. For the same reason, the container was stirred with round rather than overlapping movements. The resulting mixture was then poured into vacuum pouring funnels (see Item A, Fig. 1) and outgassed until bubbling stopped or became very slow, down to 300-400 torr. The specimen molds were prepared by cleaning (with a degreaser), drying, and then coating them with a very thin layer of mold release (Ram Chemical 225). The molds were then preheated to about 125°F in order to minimize shrinkage and surface blemishes on the specimen. This process also seems to minimize the seam marks which occur when a split mold is used. The molds were placed in desiccators directly beneath the vacuum pouring funnels and pumped down to approximately 500 torr (see Item B, Fig. 1). The pressure in the vacuum pouring funnel was slowly raised to near-atmospheric pressure by releasing nitrogen gas into the funnel (see Item C, Fig. 1). An arrangement of a ground glass valve, a gum rubber hose, and a copper tube (see Item D, Fig. 1) was employed for pouring the mixture from vacuum funnel to mold. The pouring was facilitated (see Item E, Fig. 1) by using an angle in the pouring tubes, a pouring trough screwed onto the top of the mold, and a lever clamped to the pouring tube to maneuver the tube into the proper position. After the pouring, the samples were outgassed in the desiccator for a short time, and then removed from the desiccator by valving in nitrogen gas to bring the pressure slowly up to atmospheric pressure. The molds were then placed in an oven to cure at 150°F for 24 hours. The specimens were removed from the molds and checked with a polariscope for residual stresses. If signs of stress were found, specimens were annealed by slowly raising the temperature to slightly above 150°F and then cooling very slowly at an approximate rate of 5°F/hr until room temperature was reached. With a fine emery cloth, specimens were smoothed after annealing. Burnishing tools were used to eliminate seam marks or any other blemishes that might cause premature failure during testing.

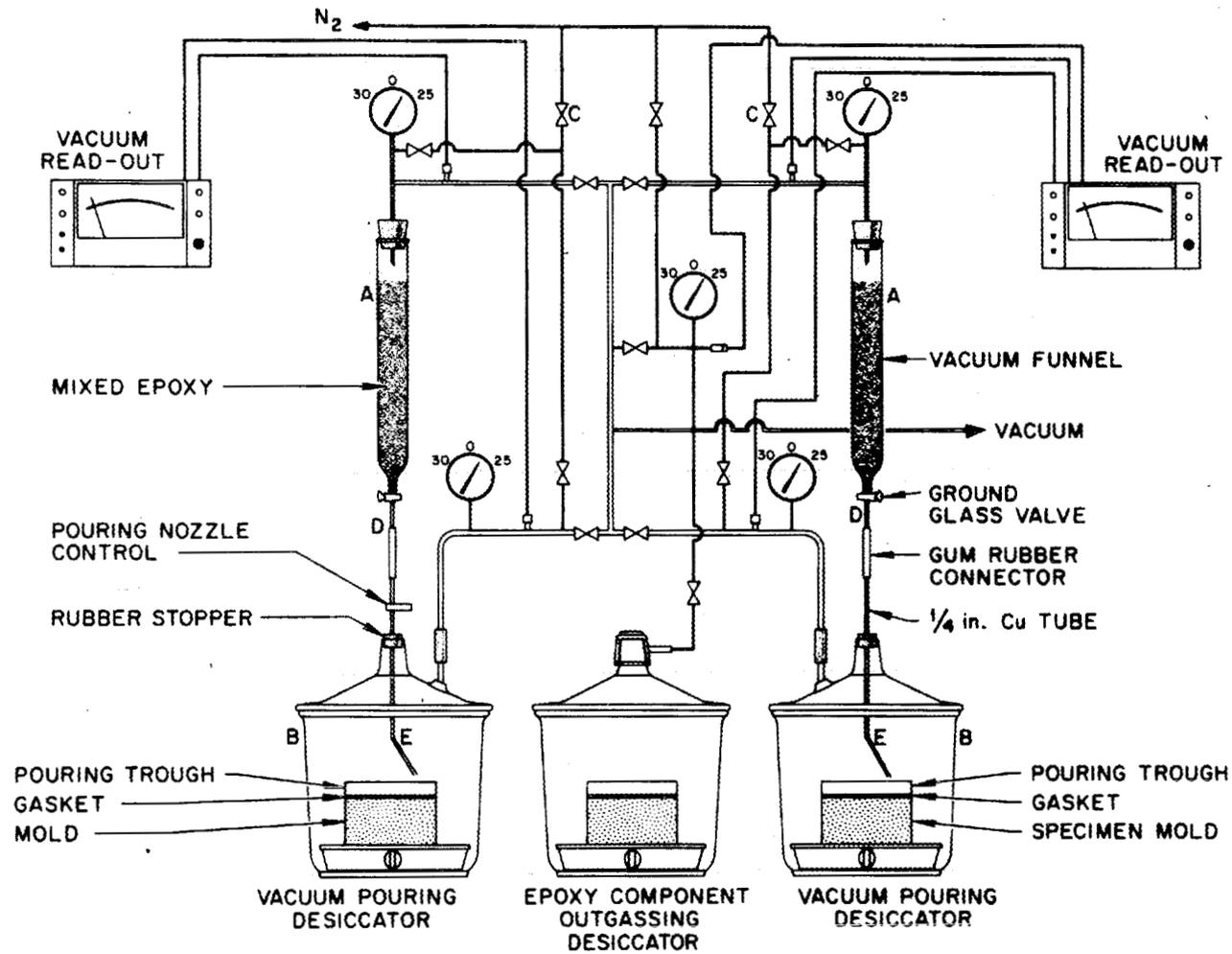


Fig. 1. System for vacuum pouring of epoxy tensile and impact specimens.

## 4. TESTING

Nine tensile specimens were poured from each mixture. The specimens were round, with a diameter of 0.250 in., and a gage length of 1.125 in. Five of the specimens were tested at 293 K and four were tested at 77 K (see Figs. 2 and 3). The tensile tests were carried out using a motor-driven Houndsfield Tensometer<sup>3</sup> with seven spring beams giving ranges with maximum loads of 2 tons,<sup>4</sup> 1 ton, 1000 lb, 500 lb, 125 lb, and 62.5 lb. Tensile strength vs epoxy mixture data may be noted in Fig. 4 (refer to Table 1 for amount of curing agent Z). Modulus of elasticity vs epoxy mixture may be noted in Fig. 5.

Forty impact specimens were poured from each mixture at the same time and under the same conditions as the tensile specimens were poured. The impact specimens were 1/4 in. square and 1-1/2 in. long, notched to a root diameter of 0.144 in. Twenty of the specimens were tested at 77 K and 20 were tested at 293 K. The impact tests were carried out using a Houndsfield Plastic Testing Machine.<sup>3</sup> This machine is designed to measure the work done in breaking a notched test piece, which indicates the resistance a material can offer to stress concentration. In the impact test, the energy of fracture is equal to the difference in the energy stored in the tup before and after fracture. (See Fig. 6 for Houndsfield Plastic Impact Testing Machine.) If  $E$  = energy stored in the tup, I.S. = impact strength of the test piece, and  $e$  = residual energy, then  $I.S. = E - e$ . The stored energy is shown in numbers stamped on the tups and varies from 2 ft-lb down to 1/32 ft-lb. The residual energy is indicated by the height to which the tup ascends after breaking the test piece; for a given value of  $e$  to be subtracted each time, the dial is graduated backwards and gives a direct reading of the fracture of I.S. In each test, the dial reading must be multiplied by the figure stamped on the tup. (Tables are furnished with the equipment to enable the true energy of the fracture to be obtained without calculation.)

The impact test in liquid nitrogen was carried out in the following manner. The specimen was placed in a dewar of liquid nitrogen and allowed to reach ambient temperature. It was then removed from the liquid nitrogen

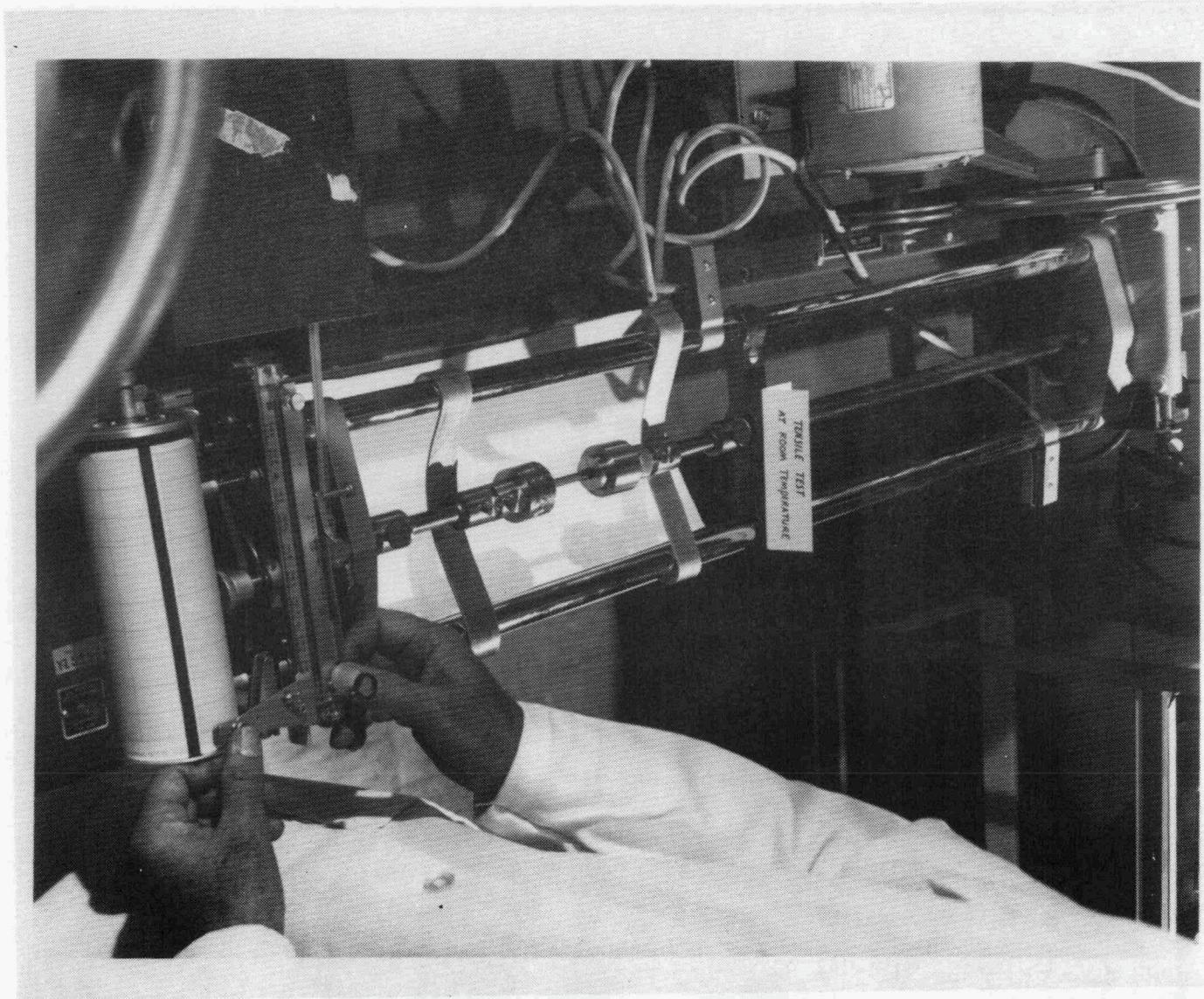


Fig. 2. Tensile testing 293 K.

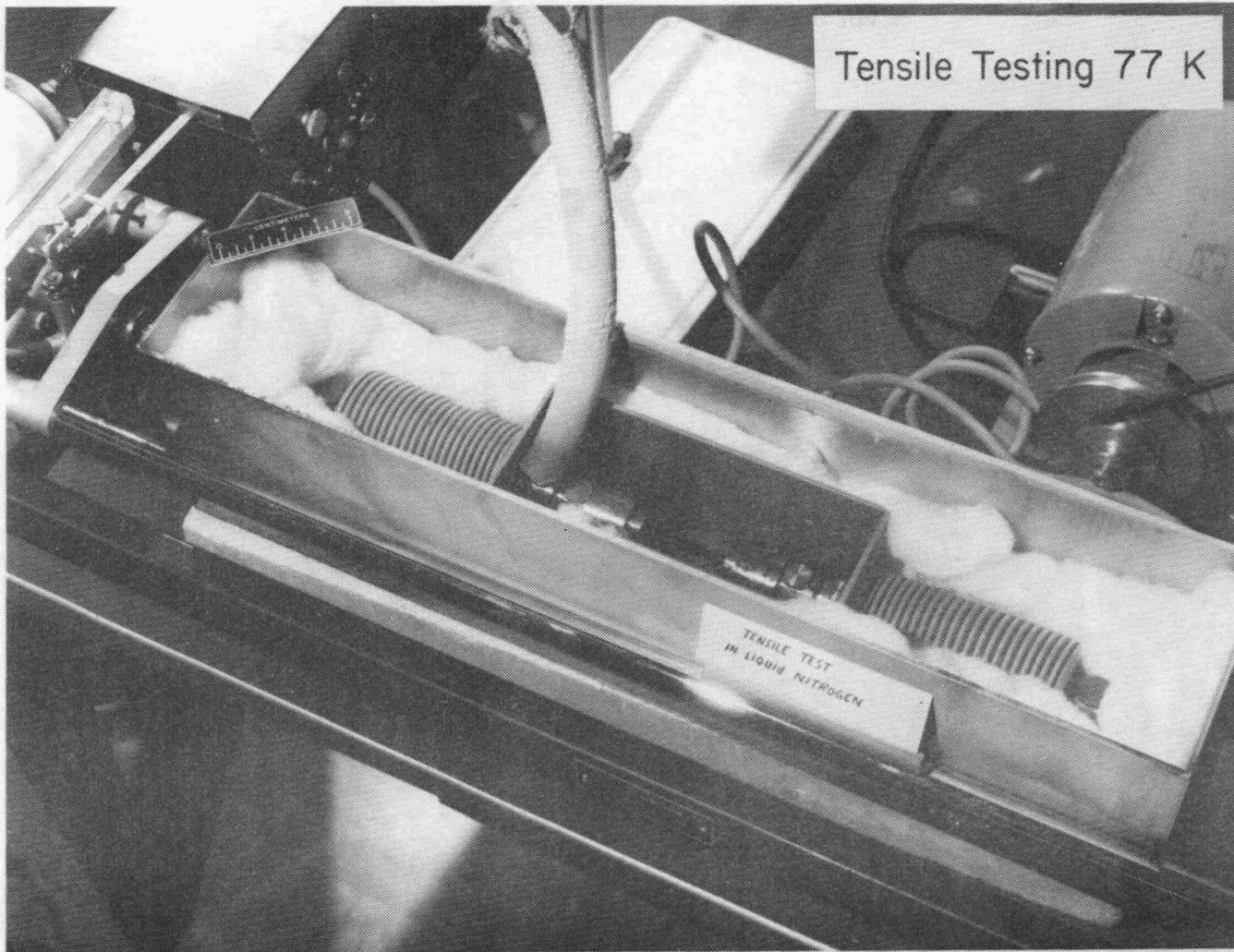


Fig. 3. Tensile testing 77 K.

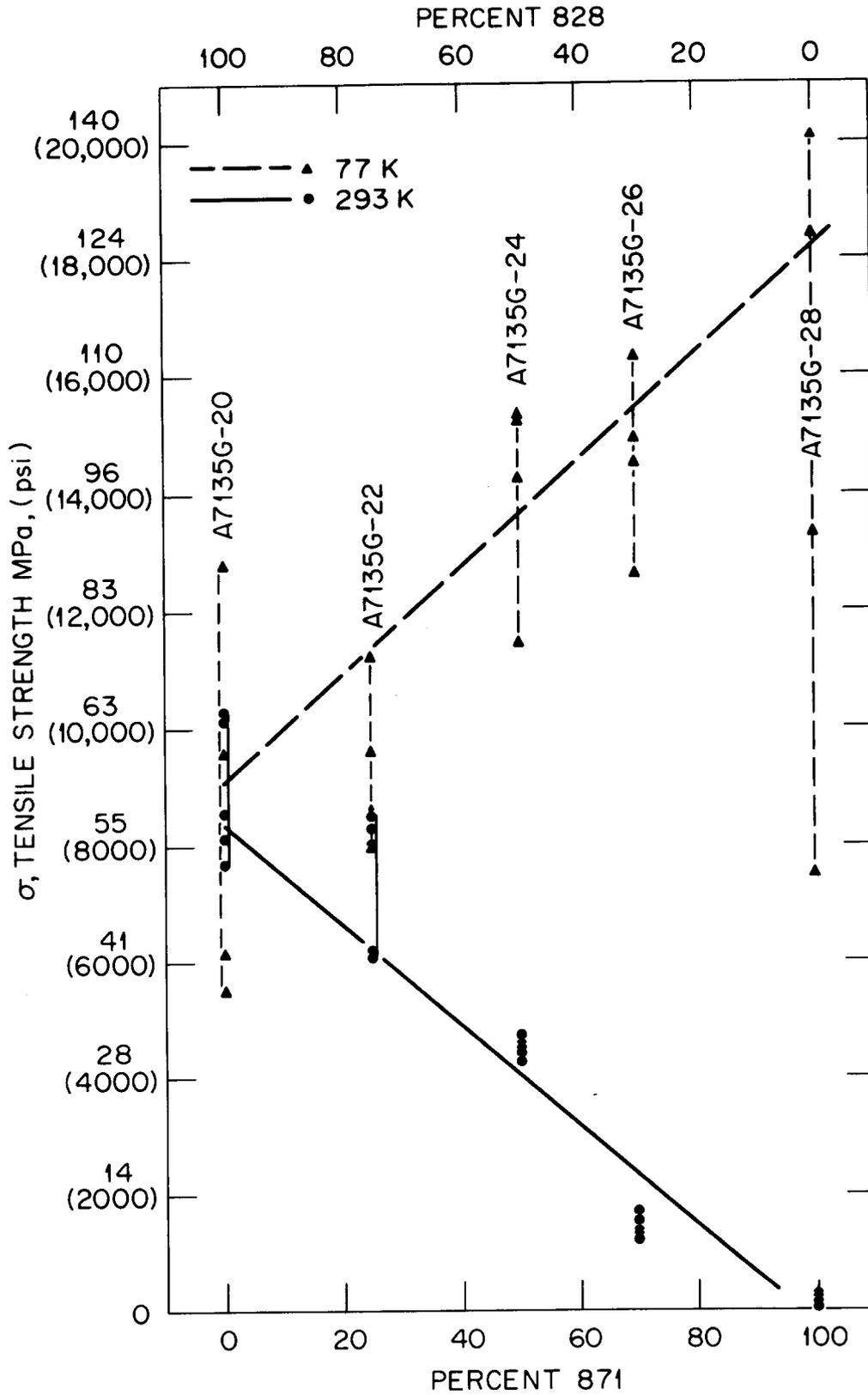


Fig. 4. Tensile strength vs epoxy mixture.

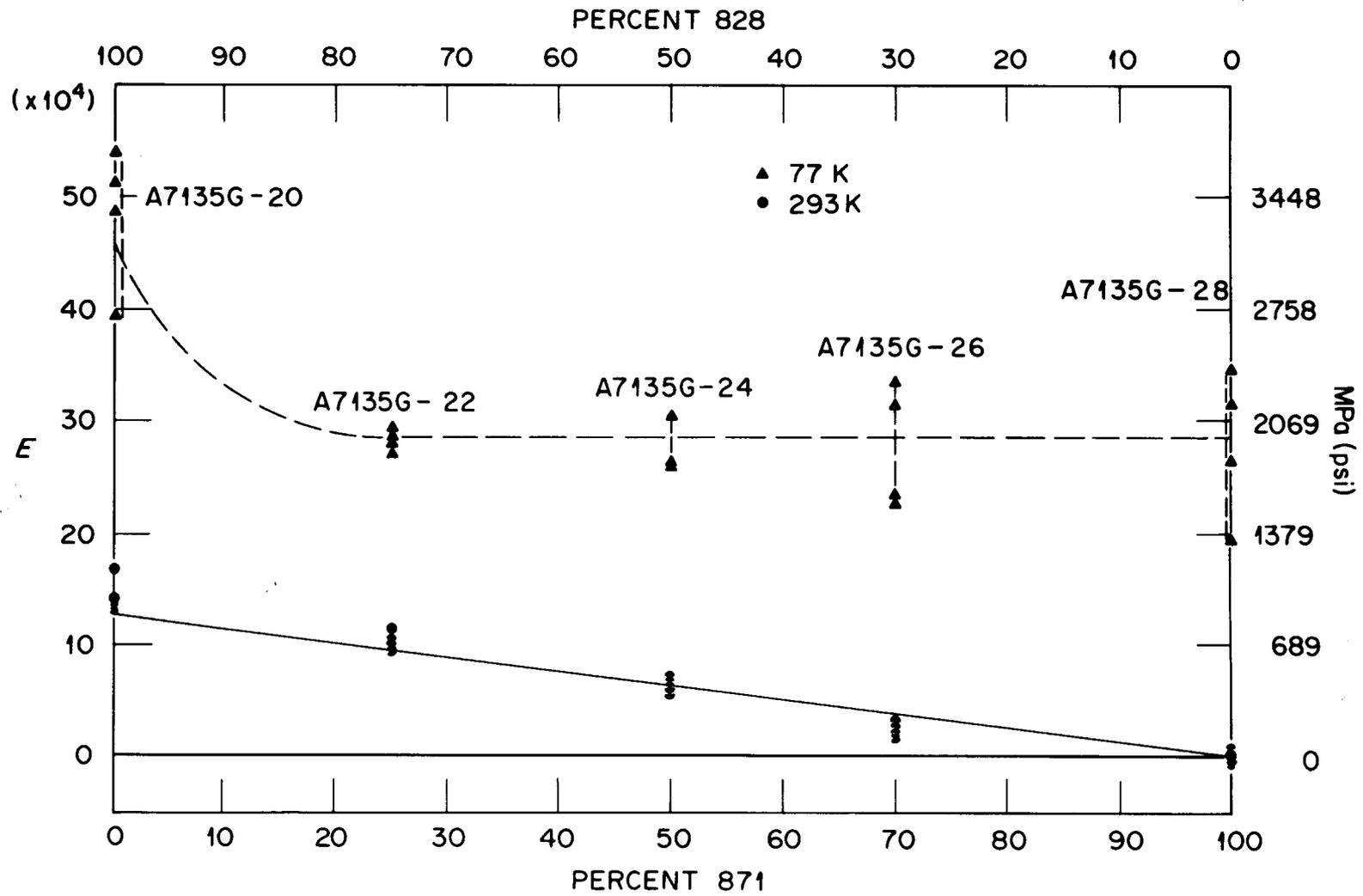


Fig. 5. Modulus of elasticity vs epoxy mixture.

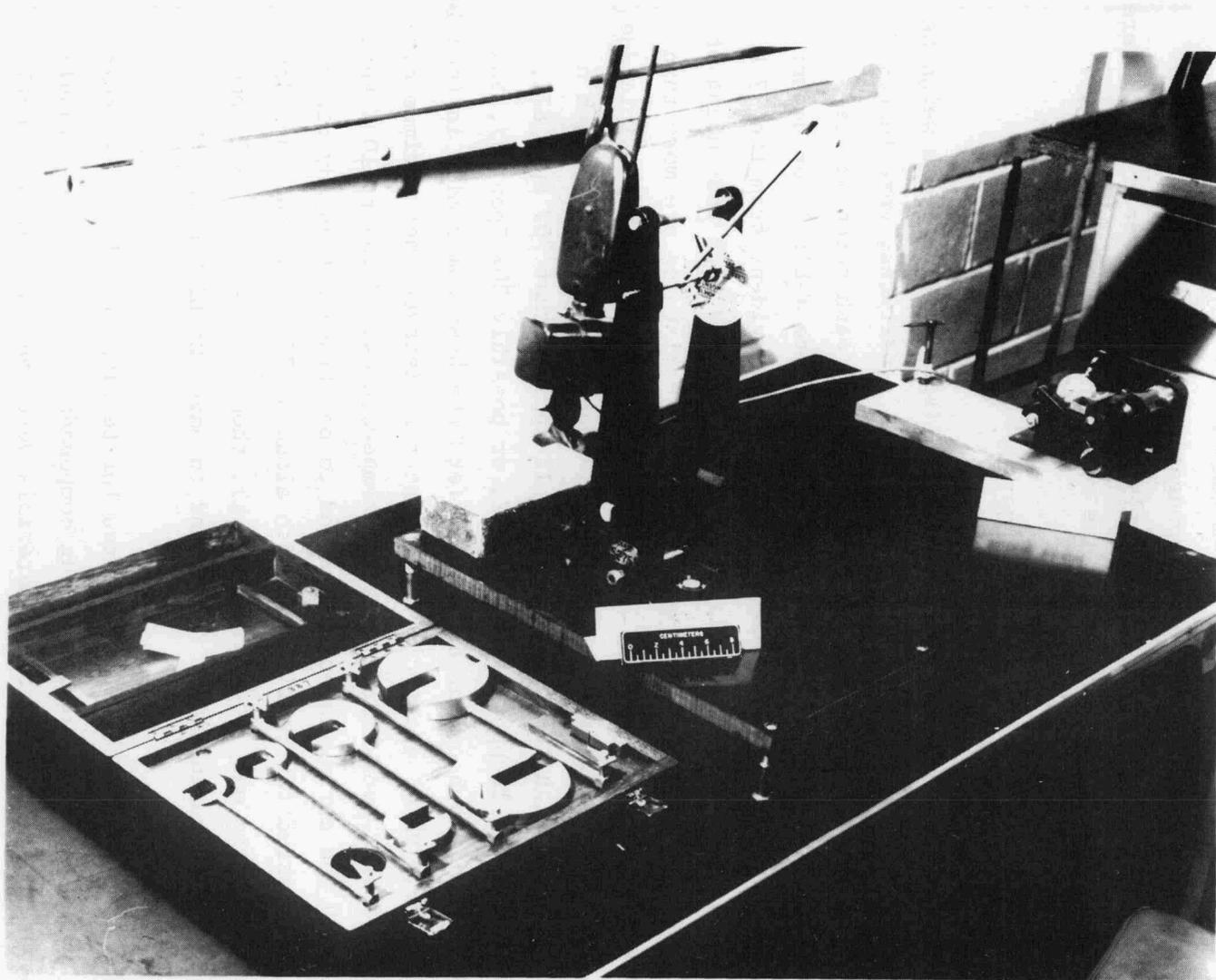


Fig. 6. Impact testing machine.

and placed on the impact testing machine, and the tup was released. After considerable practice, the entire test of transfer of one specimen from dewar to impact tester and release of tup required from 5 to 10 sec. The wide variation of impact data at room temperature may be attributed to the difficulty in developing a propagating fracture in a highly flexible medium. This effect is greatly reduced at cryogenic temperature due to the increase in stiffness. The results may be noted in Fig. 7.

## 5. CONCLUSIONS

Five different batches of epoxy mixture (see Table 1), each weighing 500 grams and prepared under nearly identical conditions, were tested. The identification, mixture, and test results of each batch may be deduced by the study of Figs. 4, 5, and 7 (refer to Table 1 for amount of curing agent Z in each batch). It is at once evident from the 77 K tests that tensile strength increases proportionally with the amount of Epon 871 in the mixture (see Fig. 4). When the mixtures are more than 90% Epon 871, impact testing at 273 K becomes practically impossible due to the rubbery condition of the material; when tested at 77 K, such a mixture evinces high tensile strength. It appears that 70% Epon 871, 30% Epon 828 with 13 pph curing agent Z, or possibly 50% Epon 871, 50% Epon 828 with 15 pph curing agent Z, offers the best compromise in tensile strength, modulus of elasticity, and impact resistance when optimum data are being sought over a wide range of temperatures. However, 100% Epon 871 with 10 pph curing agent Z is found to be stronger than any other batch when tested for tensile strength alone at 77 K. If one considers impact resistance only (tested at 273 K), then 70% Epon 871, 30% Epon 828 with 13 pph curing agent Z is found to have the highest shock resistance.

Some other materials were selected for testing in this series that were thought to be useful as potting compounds or as fabricated insulation separators. Some of these materials were available in a directly usable form, which could be purchased, while others had to be produced on sight due to the parameters which were to be investigated. The measured ultimate strengths of these materials are reported in Table 2.

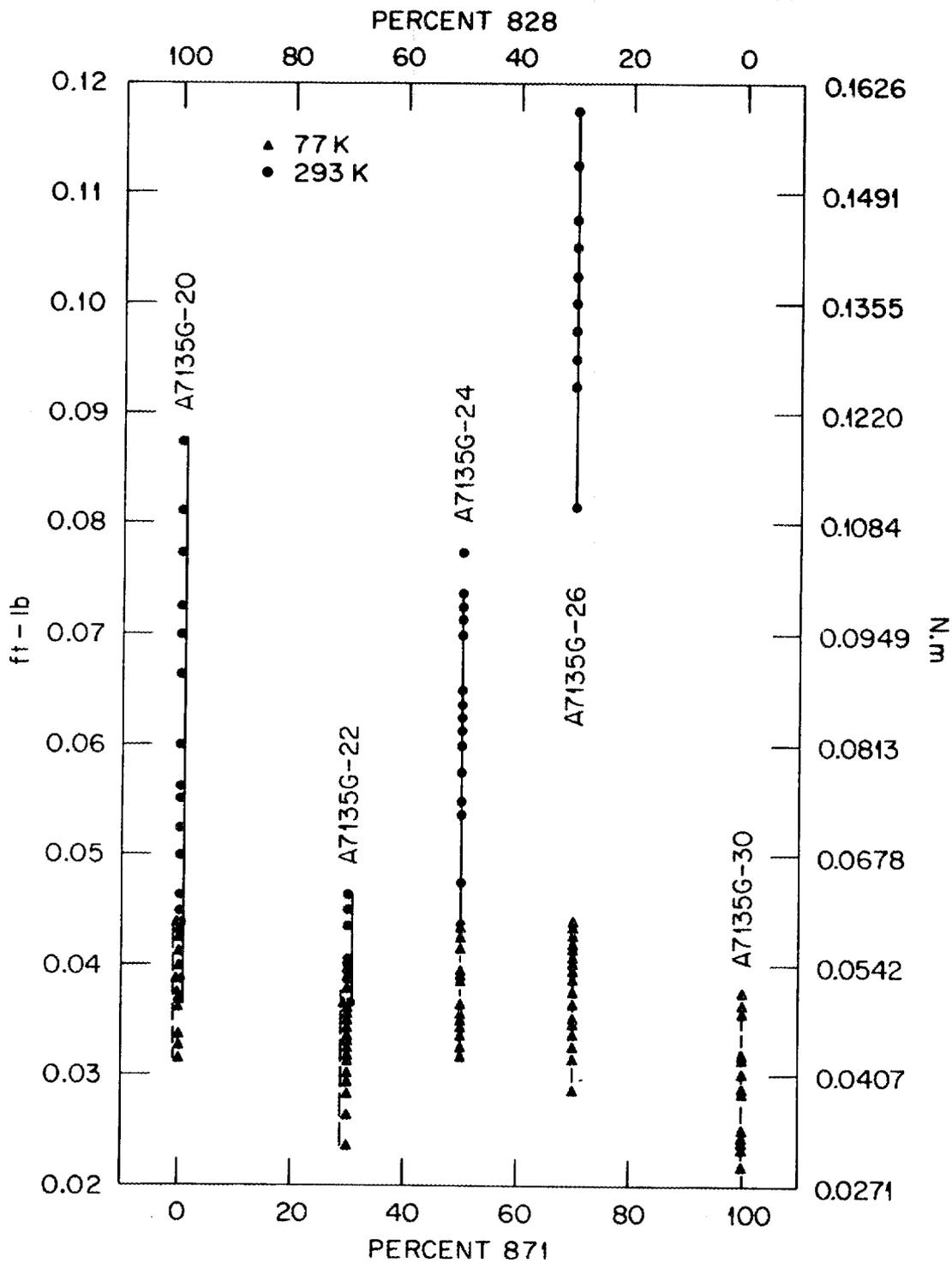


Fig. 7. Impact strength vs epoxy mixture.

All of the materials in this table were fabricated from bulk stock. Stycast 2850 FT was the only material mentioned in the table that had to be mixed, and it was of such high viscosity that it was not vacuum poured, but outgassed in the molds after it was poured. The specimens did not show any signs of voids.

Table 2. Ultimate tensile strength of bulk materials

	Ultimate tensile strength			
	MPa	Psi	MPa	Psi
	293 K		77 K	
Stycast 2850 FT	14	(2,000)	105	(15,000)
G-30	340	(50,000)	105	(15,000)
G-50	310	(45,000)	550	(80,000)
DuPont 101	48	(7,000)	130	(18,500)
Delrin	69	(10,000)	140	(21,000)
Vespel 211	62	(9,000)	100	(14,500)
Vespel 92Y77	86	(12,500)	105	(15,000)
Tefzel	32	(4,700)	83	(12,000)
Insulation Papers	19	(2,800)	45	(6,500)

Some experiments have recently been performed to test the 70% 871, 30% 828, 1.3 pph curing agent Z with the addition of 0.5% Silane 6020 and 6040, two coupling agents manufactured by the Dow Corning Corporation. The few tests performed with these agents have shown favorable results and seem to warrant further investigation.

## REFERENCES

1. Tradenames of materials are used in this report for clarity. In no case does such selection imply recommendations or endorsement by the authors, nor does it imply that the material is necessarily the best available for the purpose.
2. Epon resins are manufactured by the Plastic and Resin Division of Shell Chemical Company.
3. Houndsfield Tensometer manufactured by Tensometer Limited, 81 Moorland Road, Croydon, Surrey, England.
4. British ton (2240 lbs).



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