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APPARATUS FOR LOW-TEMPERATURE TENSION TESTS OF METALS

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Apparatus for Low-Temperature Tension Tests of Metals*

By Robert J. Mosborg¹

SYNOPSIS

A brief description of the testing equipment and procedure developed at the University of Illinois for tension tests at low temperature is presented. With the use of a bath of liquid nitrogen for tests at -321 F and a bath of Freon 12 cooled with liquid nitrogen for tests between -90 and -230 F, the testing procedure proved to be both convenient and efficient. The use of a Dewar flask as part of the testing apparatus eliminated additional handling or transfer of the liquid nitrogen and resulted in considerable economy.

This paper describes apparatus developed in the Structural Research Laboratory of the Civil Engineering Department at the University of Illinois for the purpose of performing tension tests on various types of metal specimens at low temperatures. In the range between room temperature and the temperature obtainable with solid carbon dioxide (dry ice), no special difficulties are encountered, and relatively simple apparatus and procedures can be used.

Temperatures down to -90 F can be obtained with a bath of commercial solvent cooled with dry ice. For the tests at lower temperatures entirely different equipment is necessary. Testing temperatures within the range from -90 to -230 F can be reached with a liquid bath of Freon 12 cooled with liquid nitrogen. For tests at a temperature of -321 F the specimen can be immersed directly in a bath of liquid nitrogen.

The low-temperature tests for which the described apparatus was developed were conducted under slowly applied static loads. For the duration of these tests, it was necessary that the temperature of the specimen and the bath remain constant. Under these conditions, a method which would make it possible to control the temperature accurately

and to change it easily and promptly was required. Also, to conserve liquid nitrogen, the tests required apparatus with sufficient insulation to reduce the conduction losses to the testing machine and to the surrounding atmosphere to a minimum both while the specimen was cooling and during the test.

The apparatus was developed in connection with research programs sponsored by the Materials Branch of the Office of Naval Research and by the Copper and Brass Research Assn. For

the former, the program concerned tests of round, notched, steel specimens of the type shown in Fig. 1(a). The copper program included tension tests of copper coupon specimens of the dimensions indicated in Fig. 1(b). At present tests are being made of similar specimens having a welded joint.

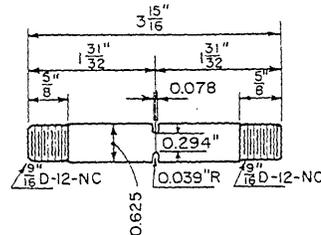
The apparatus was designed by the author working under the supervision of W. H. Munse, Research Assistant Professor of Civil Engineering and under the general direction of N. M. Newmark, Research Professor of Structural Engineering. The apparatus was built in the shops of the Civil Engineering Department.

GENERAL DESCRIPTION OF EQUIPMENT

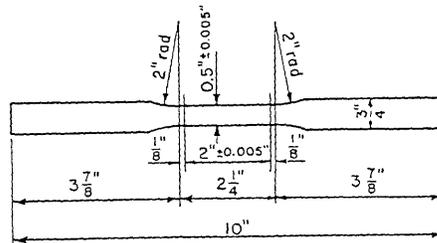
The tension tests were conducted in a 120,000-lb Baldwin-Southwark hydraulic testing machine equipped with spherical seats in both crossheads. The equipment was designed specifically for use with this machine, and a general view of the apparatus is shown in Fig. 2.

The testing chamber is provided by a double-walled sheet-iron container mounted on the lower crosshead of the testing machine. The container is made up of an inner and outer tank, and the space between the tanks is filled with a fine, light, powdery insulating material.² The bottom of the outer tank includes a pulling stud which connects to the testing machine crosshead.

Another stud, to which the lower pullhead for the specimen is connected, is located in the bottom of the inner tank. The load applied by the testing machine crosshead is transmitted through these pulling studs to the specimen. The two studs, however, are separated by plastic insulating rings which prevent direct contact of the metal in the inner and outer tanks. This reduces the absorption of heat from



(a) Round notched steel specimen



(b) Copper coupon specimen (1/16 and 1/16 in thick)

Fig. 1.—Details of Specimens Tested.

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² The material used was "Santocel," a product of the Monsanto Chemical Co.

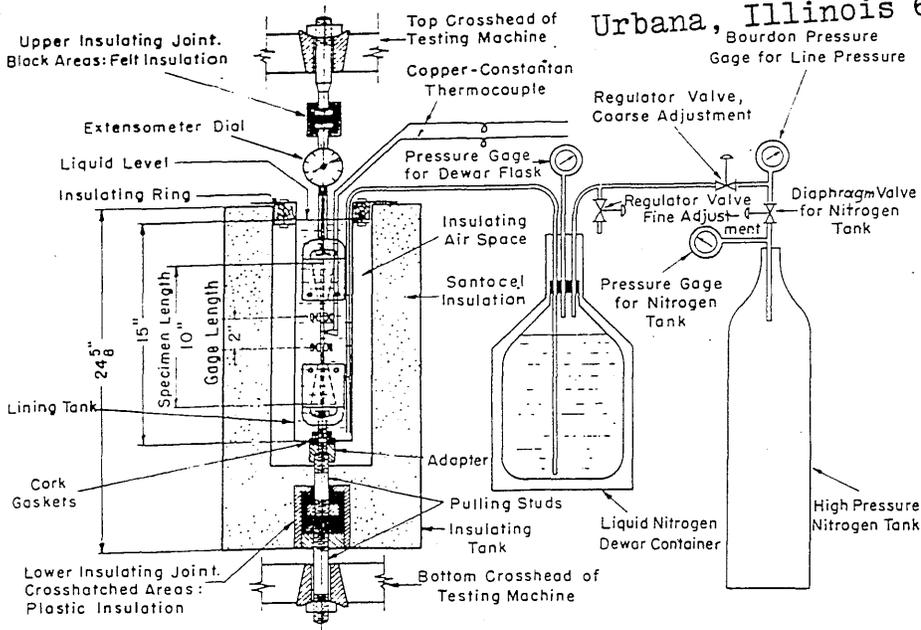


Fig. 2.—Test Assembly for Tests at -321 F.

the lower crosshead and the rest of the testing machine.

Between the testing apparatus and the top crosshead of the testing machine is another insulating joint employing felt insulation. This material proved to be highly satisfactory because it introduced a degree of flexibility which helped reduce eccentricity.

For the tests at -321 F, the pullheads, specimen and testing bath were placed in a lining tank inside the test chamber; the lining tank was surrounded by an insulating air space. Each of the pullheads contained a spherically seated connection so that an axial load from one pullhead to the other was assured. Details of these pullheads are shown in Fig. 3. In conducting tests at temperatures between -90 and -230 F, a helical cooling coil which surrounded the specimen and pullheads was placed in the testing chamber. Figure 4 shows the copper coupon test assembly surrounded by this helical cooling coil.

In the tests it was necessary to obtain a continuous record of the applied load and the accompanying elongation. An extensometer was required that would function satisfactorily when subjected to a variation in temperature from room temperature at its upper end to the testing temperature of the specimen at its lower end. Because of this extreme temperature gradient over a relatively short length, the development of this instrument was rather difficult. Several types of extensometers were tried. Best results were obtained with the double dial device shown in Fig. 3. With this arrangement two 0.0001-in. mechanical micrometer dials, mounted 180 deg apart, gave an average value of the

existing elongation. In constructing this extensometer, direct metal contact between the end immersed in the testing bath and the end exposed to room temperature was eliminated by the polystyrene insulating segments. These polystyrene segments were drilled and tapped to match the metal components from each end and effectively pre-

vented the formation of any frost on the dials of the extensometer. This eliminated the sluggish effect of frost action on the dials and enabled them to operate at a temperature only slightly below room temperature.

Liquid nitrogen was obtained in either a 15- or a 25-liter Dewar flask which served a dual purpose: that of a storage container between tests and a dispenser during the test. For use during the tests, a special fitting was developed which sealed the flask and also permitted connections to be made to the other apparatus within the system. This procedure eliminated any additional handling or transfer of the liquid nitrogen.

Additional pressure, to force the liquid nitrogen from the flask, was provided by a 220-cu ft cylinder of gaseous nitrogen. This cylinder was fitted with a pressure regulator which controlled, within 0.5 psi, the pressure of the gas taken from the cylinder.

The temperature of the test specimen was measured by a copper-constantan thermocouple clamped to the specimen. The temperature was recorded continuously with a recording self-balancing potentiometer. There was available also an accurately calibrated portable potentiometer which could be switched into the circuit occasionally to check the

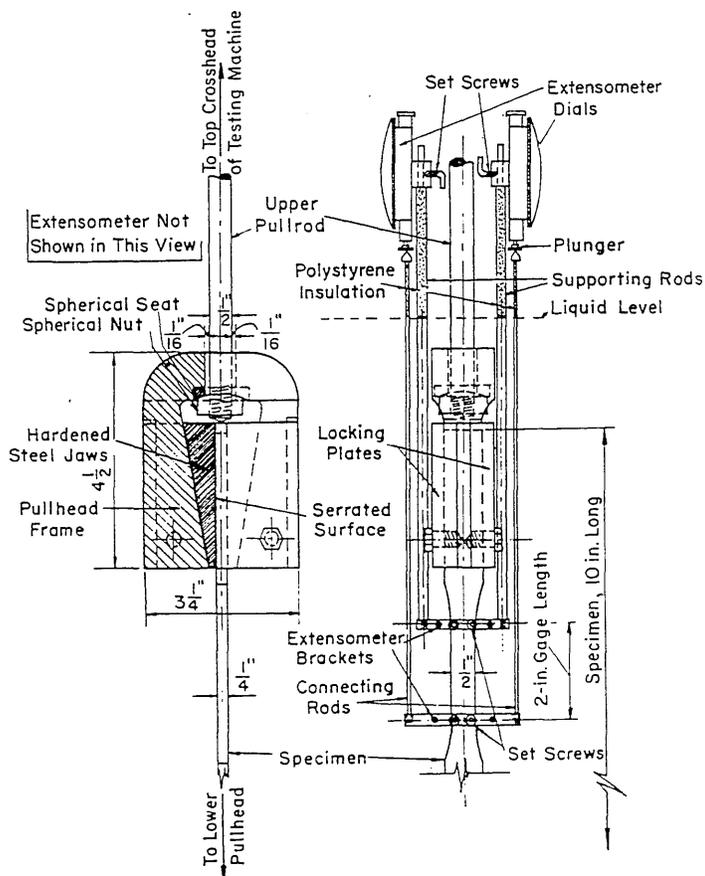


Fig. 3.—Pullhead, Specimen, and Extensometer Assembly for Tests at -321 F.

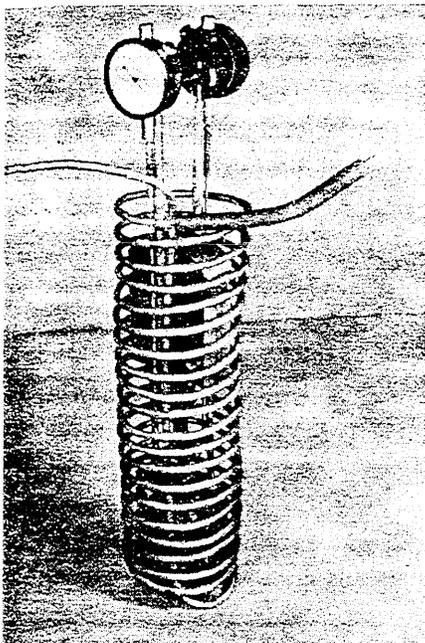


Fig. 4.—Helical Cooling Coil Surrounding Copper Coupon Test Assembly.

recorded temperature. These two instruments were found to be in good agreement at all times.

DISCUSSION OF TEST PROCEDURE

Operating Principles:

The operation of the apparatus shown in Fig. 2 may be explained as follows. The Dewar flask when placed in this system is sealed so that the liquid nitrogen, in its constantly boiling state, produces a pressure which cannot escape from the container and therefore acts on the surface of the liquid. This seal on the flask permits additional pressure from the cylinder of high-pressure nitrogen gas to be exerted on this liquid nitrogen surface. Also passing through this seal is a $\frac{1}{4}$ -in. copper tube which extends to the bottom of the Dewar flask. Because of the constantly boiling state, the liquid nitrogen builds up a pressure over its surface, and this, together with the supplementary pressure from the cylinder of gas, is sufficient to force the liquid nitrogen up through the copper tube and over to the testing chamber.

Since the Dewar flasks are nothing more than large vacuum bottles and are not constructed to withstand any appreciable internal pressures, a pressure gage is installed which indicates the pressure existing above the liquid nitrogen surface. This gage should be read frequently, not only as a safety measure, but also to indicate any change that might occur in the rate of flow.

In a test at a temperature of -321 F, the $\frac{1}{4}$ -in. copper tube from the Dewar flask conducts the liquid nitrogen directly into the lining tank, thus forming a

liquid bath of nitrogen for the specimen. The liquid nitrogen in this bath is boiling away into the atmosphere constantly. By proper adjustment of the pressure within the Dewar flask, a compensating incoming rate of flow is established and a constant level of liquid nitrogen is maintained in the tank.

For the tests within the temperature range from -90 to -230 F, a combination of liquid nitrogen and Freon 12 is used. For these tests, a helical coil of $\frac{1}{4}$ -in. copper tubing is placed in the testing tank. This coil just fits within the tank walls, extends from the top to the bottom of the tank and, with a vertical lead, vents to air. In the tests, liquid nitrogen from the Dewar flask is forced through this copper coil from the top to the bottom. This type of circulation provides the greatest cooling effect where it is needed most—at the surface of the bath. This cools the coil and also, somewhat, the inside of the testing tank. After the tank has been partially cooled, it is filled with liquid Freon 12.³ This procedure reduces the loss that occurs when Freon 12 is transferred to a comparatively warm container. The liquid nitrogen passing

³ Freon 12 has a boiling point of -18 F and a freezing point of -256 F. Therefore, when not used in a test, it is stored in a refrigerator at a temperature below -18 F.

through the coil now cools the Freon bath to the desired temperature.

In passing through this coil, the liquid nitrogen absorbs a considerable amount of heat and vents to the air as a gas. The fastest cooling rate for the bath occurs when this escaping gas changes to a spray. This indicates that the liquid nitrogen has not had sufficient time to absorb enough heat to change completely to a gas. This rate, or any greater rate, of flow is no longer efficient. No attempt is made to collect or retain the gaseous nitrogen which escapes to the air. A photograph of a typical test setup at -230 F is shown in Fig. 5.

Testing Procedure:

The specimen, pullheads, extensometer, thermocouple, and lining tank are carefully assembled, set in the testing chamber, and connected to the proper fittings. The liquid nitrogen is transferred to the testing tank, either directly or through the helical coil, depending upon the testing temperature desired. With a copper-constantan thermocouple clamped to the net section of the specimen, the temperature of the specimen is recorded from the time it is mounted in the machine until it is fractured.

After a specimen reaches the desired testing temperature, it is maintained at

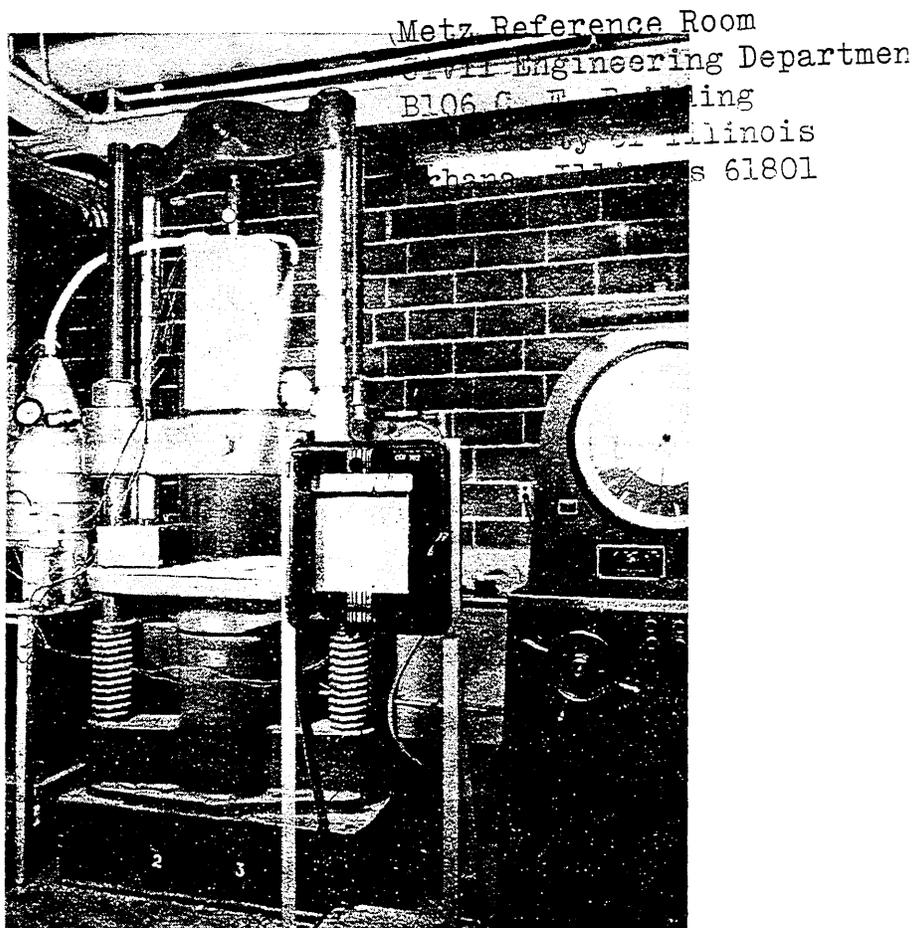


Fig. 5.—Testing Equipment Assembled for a Test at -230 F.

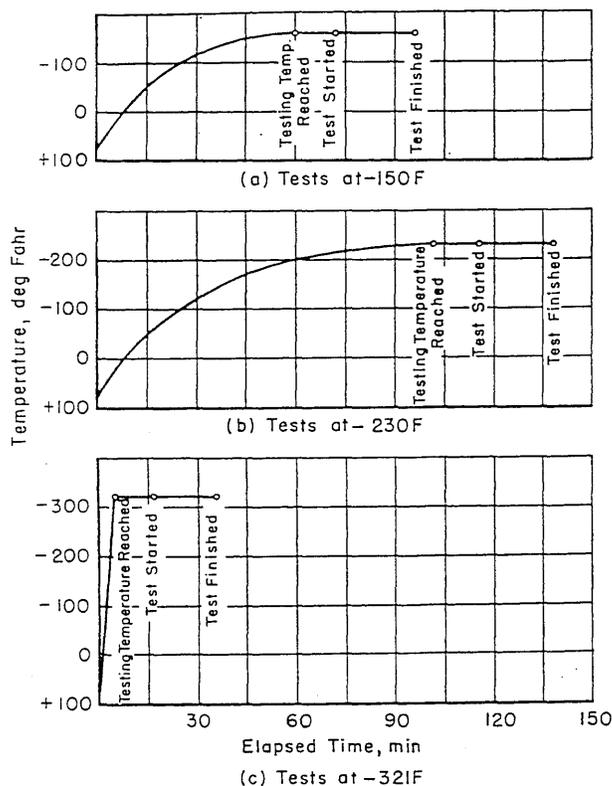


Fig. 6.—Typical Time-Temperature Curves for Low-Temperature Tests.

this temperature for 10 to 15 min before the actual test is started. This time interval reduces the temperature of the pullheads and connections to a value close to that of the test specimen. For tests in liquid nitrogen, the rate of boiling in the bath is greatly reduced when the temperature of the pullheads approaches that of the specimen. Once the test is started, simultaneous readings of load and elongation are recorded at regular intervals until fracture of the specimen occurs.

Upon completion of a test, the specimen, pullheads, extensometer, and fittings are removed from the bath. On exposure to the atmosphere, these extremely cold parts become covered with frost and have a tendency to rust. This rusting is prevented by quickly drying these parts with a warm air blower.

Performance of Tests at -321 F:

The average amount of liquid nitrogen and time required to complete a test at -321 F in each of the two series of tests is given in the following table:

	Time, hr	Amt. of Liquid Nitrogen, lb
Steel specimens...	1/2	7
Copper specimens	3/4	11

In the above tabulation the time required includes that which was necessary to bring the specimen and apparatus down to the testing temperature plus that needed to complete the test. Be-

cause of the shorter length of reduced section in the specimen, the tests of steel specimens required less time than the tests of copper specimens. Also, the specimens and apparatus for the steel tests were smaller than those for the copper tests and less liquid nitrogen was consumed.

For tests at -321 F, a slight pressure in the Dewar flask produced a sufficient flow of liquid nitrogen into the testing chamber to maintain the desired liquid level. In these tests the bath was boiling liquid nitrogen; hence the specimen was maintained at a constant temperature of -321 F throughout the entire test.

Performance of Tests Between -90 and -230 F:

The average amount of liquid nitrogen and time required to complete a test at a temperature within the range of -90 to -230 F for each of the two series is given in the following table:

	Time, hr	Amt. of Liquid Nitrogen, lb
Steel specimens....	1	6
Copper specimens..	2	14

Again in these tests the requirements for the steel specimens were considerably less than those for the copper specimens. When the additional losses due to storage, evaporation, and transfer were considered and averaged for all the tests, about 4 lb of liquid nitrogen were added to the consumption of each

test. Typical time-temperature curves for tests at -150, -230, and -321 F are shown in Fig. 6.

To cool the liquid Freon bath in the steel specimen tests, a pressure of 4 psi in the Dewar flask was great enough to provide a flow of liquid nitrogen such that all of the liquid did not change to a gas in the cooling coil. This resulted in the emission of a spray of liquid and gaseous nitrogen from the cooling coil. However, for the tests of the copper specimens a pressure of 7 psi (the maximum pressure used in the Dewar flasks) was such that only gaseous nitrogen was emitted from the 1/4-in. copper coil. In this latter series, the cooling coil was considerably longer than the coil used in the steel specimen tests and allowed additional time for the liquid to change into a gas.

In the latter tests, a coil of smaller diameter was tried. However, it was found in experimenting with these coils that the larger diameter coil transferred more liquid nitrogen for a given pressure and consequently produced a faster cooling rate within the bath. This resulted in less liquid nitrogen consumed per test and a shorter period of time necessary to complete a test. Probably the consumption of liquid nitrogen and the long testing period could be reduced somewhat by using a 3/8-in. coil.

By proper adjustment of the pressure within the Dewar flask, the deviation of the temperature during a test could be held within ±2 F of the required test temperature.

SUMMARY

The equipment and method outlined in this paper proved to be quite satisfactory for static tension tests at the temperatures indicated and for the types of specimens shown. A sufficient amount of insulation prevented excessive conduction losses and permitted satisfactory operation.

In tests between -90 and -230 F, Freon 12 was used as the cooling bath. Freon 12 was a convenient substance to use since it is inert, nontoxic, does not become gummy or sticky at low temperatures, and freezes suddenly. However, it boils at -18 F, and consequently the liquid had to be stored in a refrigerator when not in use.

This procedure does not furnish testing bath temperatures between -230 and -321 F. Therefore a liquid with a higher boiling point and a freezing point below that of Freon 12 would be desirable and more convenient to use. However, such a material was not readily available.

The use of a liquid nitrogen bath was found to provide an efficient testing medium for tests conducted at a temperature of -321 F.