(NASA-CR-163198) MANUFACTURE OF FIBER-EPOXY TEST SPECIMENS: INCLUDING ASSOCIATED JIGS AND INSTRUMENTATION (Michigan Univ.) 20 p
HC A02/MF A01 CSCL 11D
G3/24

N80-24368

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NGR 23-005-528

Manufacture of Fiber-Epoxy Test Specimens: Including Associated Jigs and Instrumentation

by

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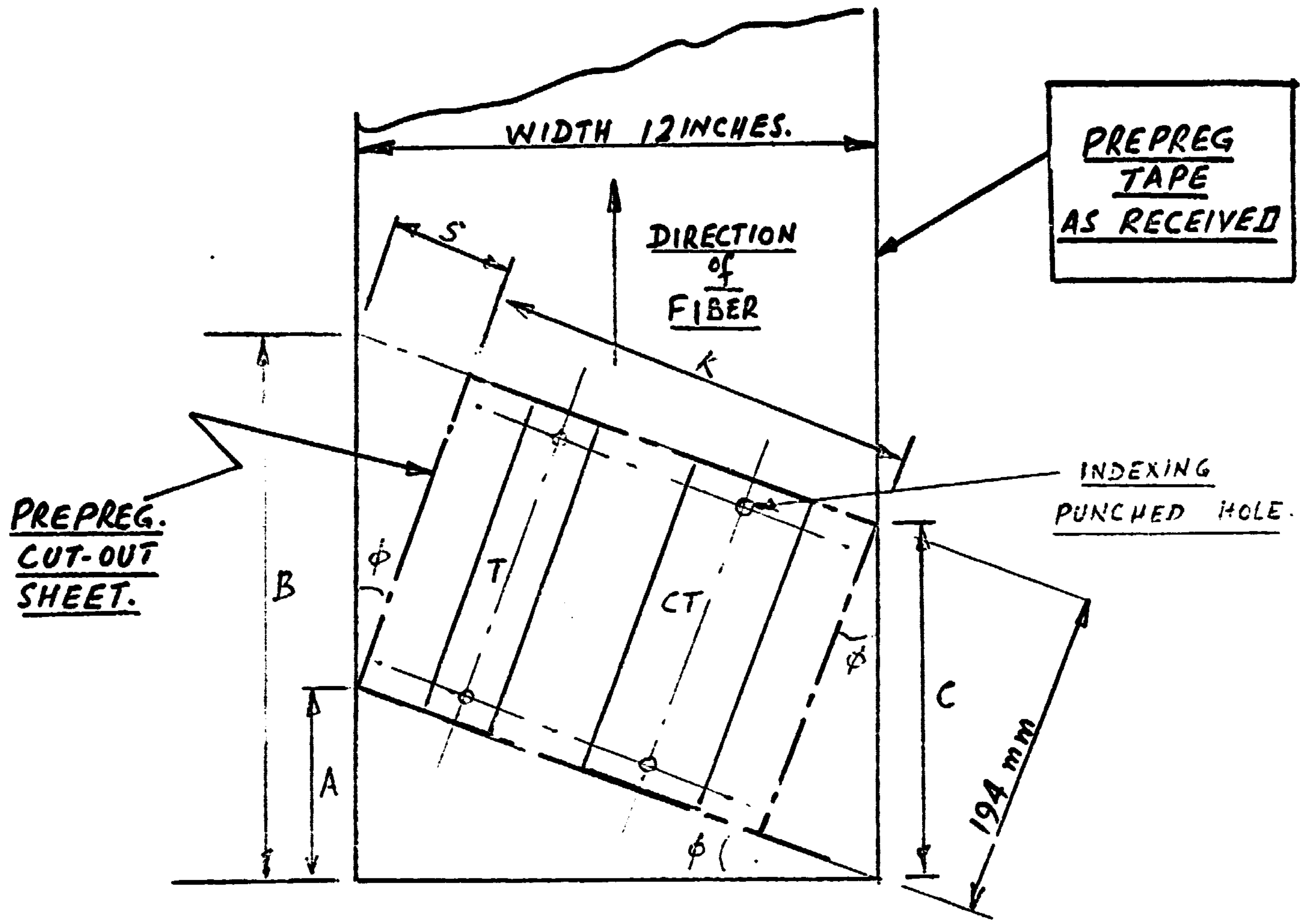
Introduction

A considerable amount of experimental work on the manufacture and strength of graphite-epoxy composites has been carried out in most European countries and the U.S. over the past decade. The correct data and thus a true assessment of the strength properties based on a proper and scientifically modelled test specimen with engineered design, construction and manufacture has led many a researcher to claim a very broad spread in optimized values. Such behavior is in the main due to inadequate control during manufacture of test specimen, improper curing and uneven scatter in the fiber orientation.

The graphite fibers are strong but brittle. Even with various epoxy matrices and volume fraction, the fracture toughness is still relatively low. At the University of Michigan various fibers and mixes have been constructed and tested over the past seven years. In recent years the work has continued with graphite-epoxy prepreg tape as a sandwich construction with intermittent interlaminar bonding between the laminates in order to produce high strength-high fracture toughness composites. Throughout the investigation an important factor has been, the quality and control of manufacture of the multilaminate test specimen blanks. The dimensions, orientation and cure have to be meticulous in order to produce the desired mix. The prepreg bought in quantity is stored in a freezer for a prolonged storage shelf life.



φ FIBRE ORIENTATION IN SPECIMEN BLANK

T TENSION SPECIMEN BLANK PROFILE. 41.5 x 194 mm

CT COMPACT-TENSION SPECIMEN BLANK PROFILE. 72 x 194 mm

FIG. 1. MARKING OUT PREPREG FOR APPROPRIATE ORIENTATION.

Dimensions A, B, C, S and K are dependent upon φ.

$A = 304.8 \tan\phi$ mm

$C = 194 / \cos\phi$ mm

$S = 194 \tan\phi$ mm

$S + K = 304.8 / \cos\phi$ mm

φ	A (mm)	B (mm)	C (mm)	S (mm)	K+S (mm)
10°	53.75	250.8	197.0	34.2	309.5
20°	110.9	317.4	206.5	70.6	324.4
30°	176.0	[illegible]	[illegible]	112.0	[illegible]

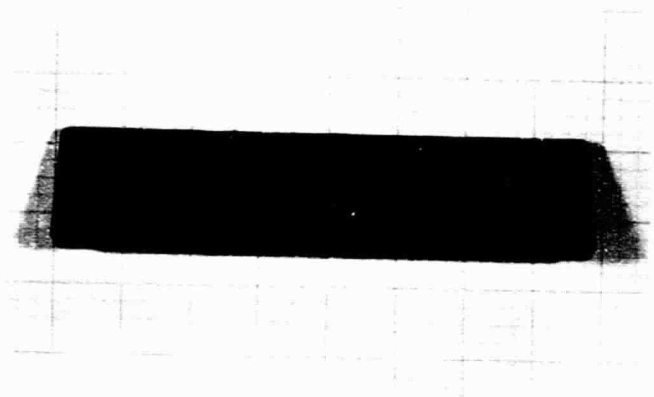


PLATE NO. 1. Base Indexing Plate Showing Indexing Dowels.

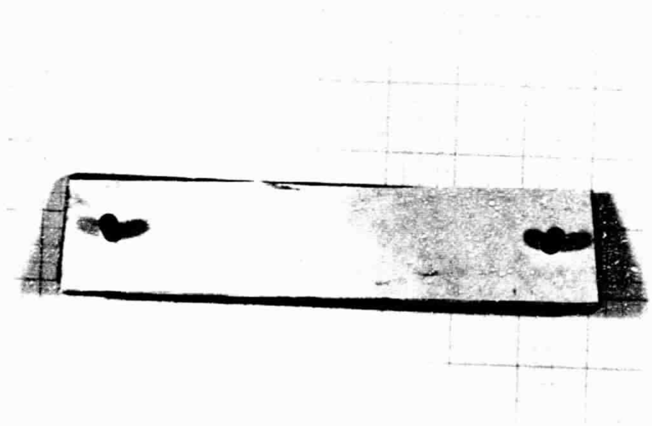


PLATE NO. 2. Base Indexing Plate Showing a Laminated Specimen Blank in Position.

Cutting and Orientation of Ply

The prepreg was bought from Narmco Industries, Incorporated, California, as a 12-in. wide tape under the commercial name 'Rigidite R 5213' with a 62-64% volume fraction. The tape has a peel-off waxed paper backing and the ply thickness is 0.004 in. approximately. The prepreg is laid flat on a Teflon sheet and the paper backing is marked off to suit the fiber orientation ϕ, see Fig. 1. The cut-out sheet is scissor cut to the required size. Two cut-out sheets are put together with the appropriate Mylar punched film. Now the double layered prepreg sandwich has paper backing on the outside. The tension specimen and the compact-tension specimen profiles are now scored on the paper back and are accordingly cut and punched for indexing holes. They are returned to the freezer and stored till the next manufacturing operation is due.

Double Layer Assembly

The base plate of the indexing and curing jig (described later) is placed with indexing dowels projecting upwards as shown in PLATE NOS. 1 & 2. The top paper backing of the double sandwich is removed and replaced by the appropriate Mylar film which with indexing holes duely punched, is stacked on to the indexing dowels. The procedure is repeated till the required number of layers have been built-up. (The present manufacture is of a 12 laminate or ply specimen.) The top backing paper is not disturbed so the completed blank stack has waxed paper backing both on top and bottom. This technique allows automatic staggering of fibers to $\pm\phi$ about the longitudinal axis of the specimen.

Four to six such stacks can be assembled on to the base plate. The pressure plate with locating holes is placed on to the stacks and compacted by a 2000-lb (8.9 kN) load for initial compaction. The stack is now ready for the thermal curing cycle.

<u>Alignment and Fixture Jig for Layered Stacks</u>

The technique described above provides the true alignment of fibers if they are not disturbed. The prepreg manufacturers recommend the following steps for thermal curing.

1.	Laminate cure pressure	10-15 psi (69-103 kPa)
2.	Initial vacuum	22 in. of Hg. (27 kPa)
3.	Initial temperature rise	RT to 285°F (141°C) in 40-120 minutes
4.	Cure temperature	Maintain 285 ± 15°F (141°C ± 8°C) for 60 to 90 minutes
5.	Cooling	Allow temperature to drop to 140°F (60°C) before removal of cured prepreg

At the temperatures employed, some of the epoxy is squeezed out and after cure would bond the metal surfaces. In order to avoid such adhesion all the metal surfaces, e.g., base plate, pressure plate, jig recess, etc., are sprayed with teflon prior to assembly and cure.

The fixture jigs are manufactured to accommodate two different sized prepreg stacks. See PLATE NOS. 3 & 4. The main body of the fixture is steel base 10-1/2" x 4-1/2" x 1-1/4" (267 x 114 x 32 mm). They have inset machined recesses of 195 x 45 (or 75) x 20 mm deep. The insert plates used as base plate and pressure plate are located into the recesses. A

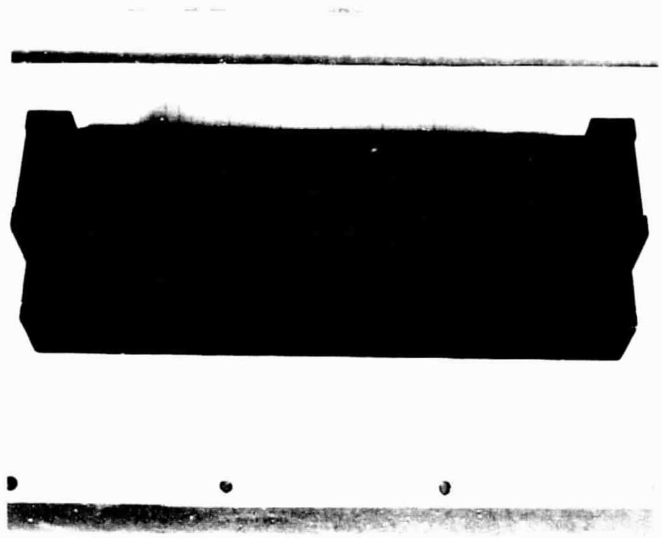


PLATE NO. 3. Fixture Jig for Manufacture of Tension Test Specimen Blanks.

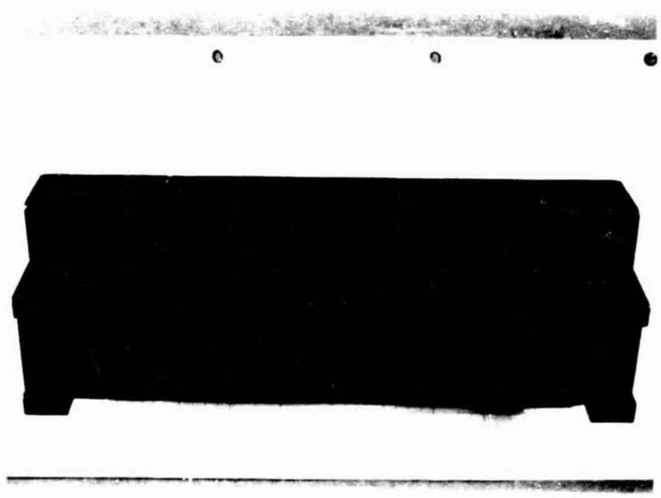


PLATE NO. 4. Fixture Jig for Manufacture of Compact-Tension Test Specimen Blanks.

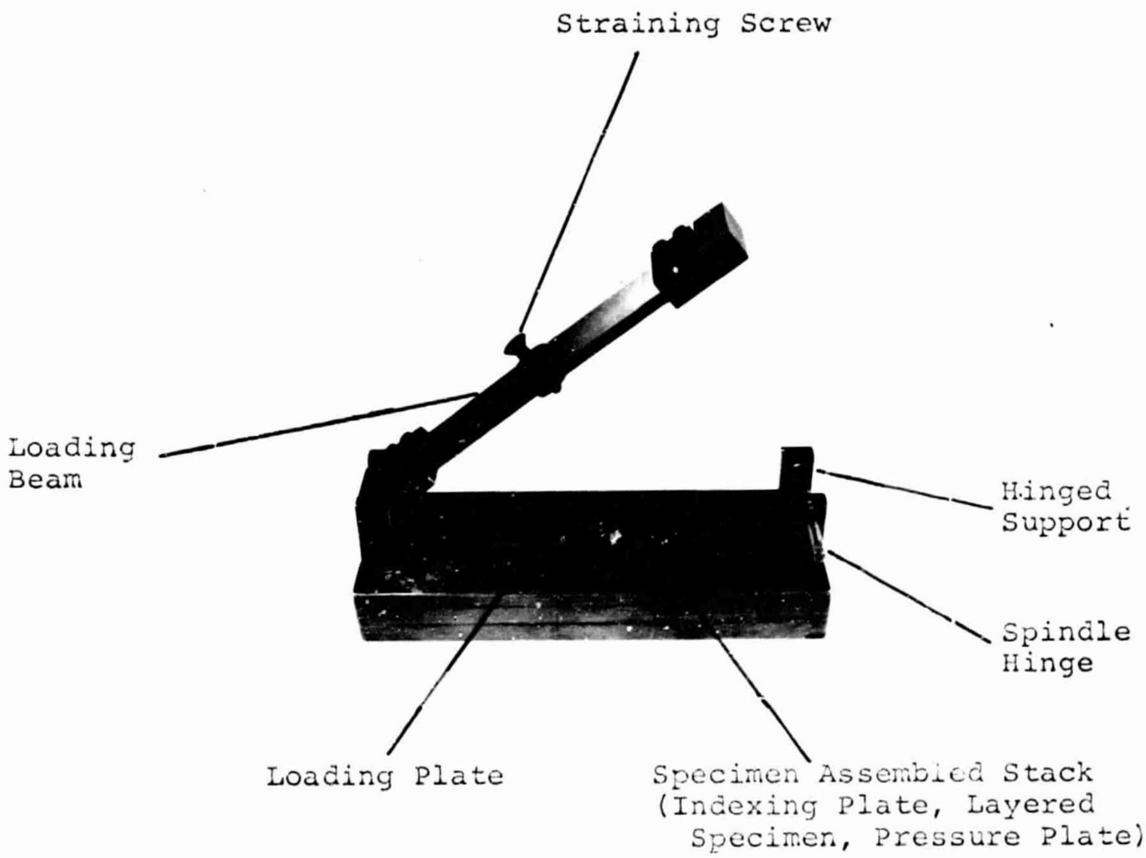
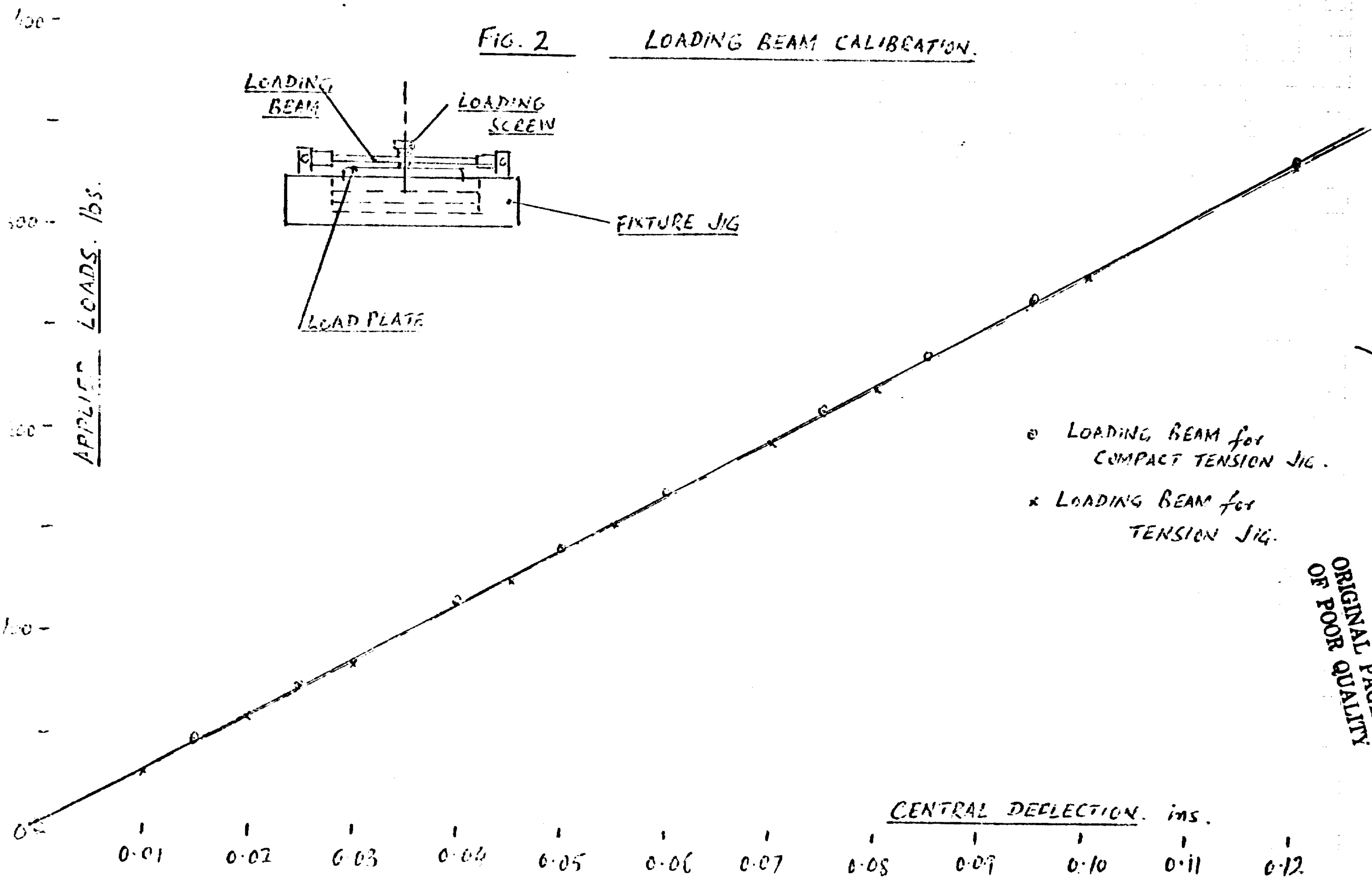


PLATE NO. 5. Fixture Jig Showing Details of Structure.

FIG. 2 LOADING BEAM CALIBRATION.



packing plate and a loading plate are placed above the pressure plate in the recess as well.

The top face of the jig base has two hinge supports for locating the loading beam by end spindles. See PLATE NO. 5. The loading beam has an elastic load limit of 500 lb (2.22 kN). In order to obtain interply pressure of 15 psi, the applied loads, by turning the load screws of 24 T.P.I., are 205 lb and 338 lb for the tension and tension compact blanks respectively (see loading beam calibrations Fig. 2).

The fixture jigs so assembled are ready for autoclave cure in the 'Blue-M' vacuum furnace.

Furnace Calibration and Curing Cycle

The furnace has an upper limit steady temperature of 300°C. It has an evacuable cylindrical chamber of 15 in. (381 mm) diameter, 12 in. (305 mm) long. The chamber has an insulated thermocouple thermohieter probe housed in the upper third space so that the chamber space temperature can be continuously monitored. The heating elements surround the chamber walls externally and therefore when the chamber is evacuated, it only records radiation steady state within its space. Disparity between actual temperature of the component placed in the furnace chamber and the chamber space as monitored by the chamber probe is considerable.

It was necessary that the correct curing temperature for the prepreg be reached. It thus became necessary to calibrate the furnace settings for the correct component temperatures. An insulated sleeved copper-constantan thermocouple was passed

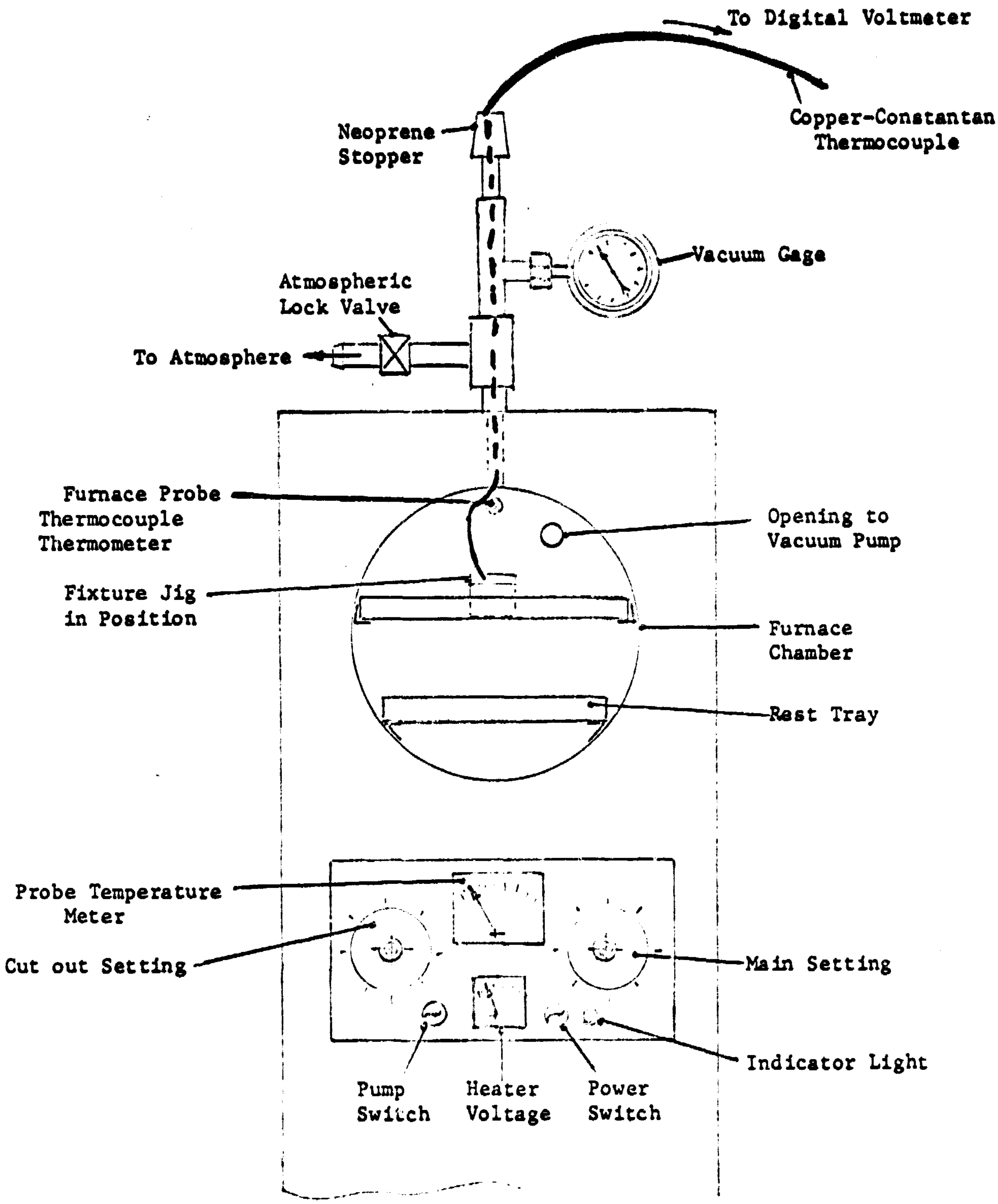
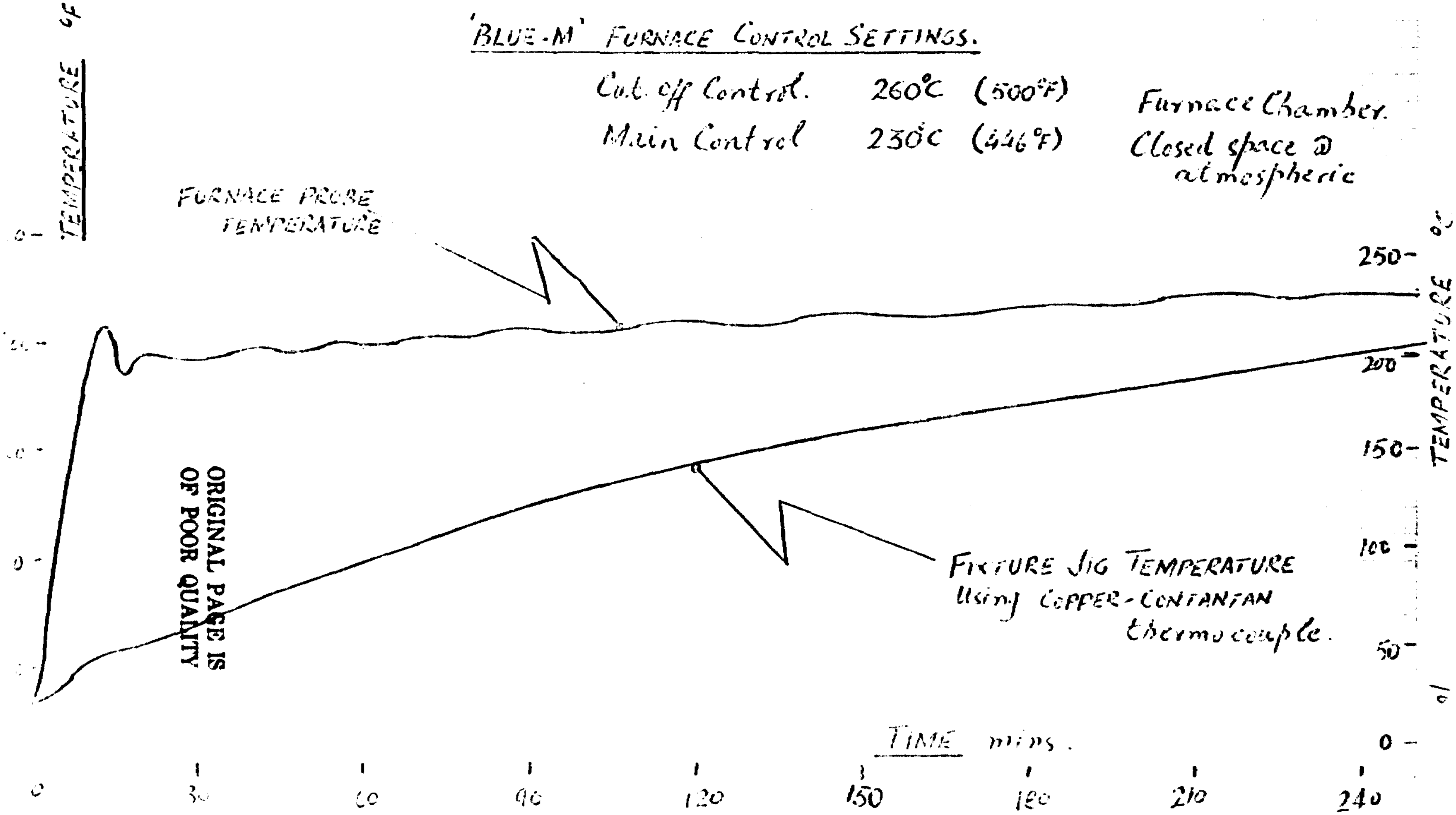


PLATE NO. 6. 'Blue-M' Furnace Showing Probe, Thermocouple, Fixture Jig.

FIG. 3. FURNACE CALIBRATION. NO. 1.



through the vacuum gauge connector at the top of the furnace chamber. The thermocouple passes through a Neoprene stopper which caps the tee-piece used for thermocouple entry. See PLATE NO. 6. With this set-up and any leakage around the stopper a vacuum build-up of 28 in. of Hg (6.8 kPa) is obtained with the vacuum pump on.

Furnace Calibration No. 1

A fixture jig with the thermocouple located in its recess was placed at the center of the furnace chamber. The chamber door, the vent to atmosphere and valve to the vacuum pump were all closed. The furnace controls were set at:

Cutoff control (H) - 260PC (500°F)

Main control (L) - 230°C (445°F)

The power was switched on and the chamber probe temperature and the jig thermocouple temperature were recorded at suitable intervals. The chamber temperature was 400°F (204°C) within 12 minutes and stabilizes to 450°F (232°C) after 4 hours. The fixture jig heats up gradually and after 4 hours is only reaching a stable state. See Fig. 3.

Furnace Calibration No. 2

The above calibration was repeated with the furnace chamber evacuated to 28 ins. of Hg (6.8 kPa) with the vacuum pump running. The furnace heaters and the vacuum pump were switched on simultaneously. The chamber and the fixture both heat up more slowly. Both reach 344°F (173°C) after 4 hours. Whereas the probe temperature remains at 344°F (173°C) the fixture, which is

FIG. A. FURNACE CALIBRATION NO. 2.

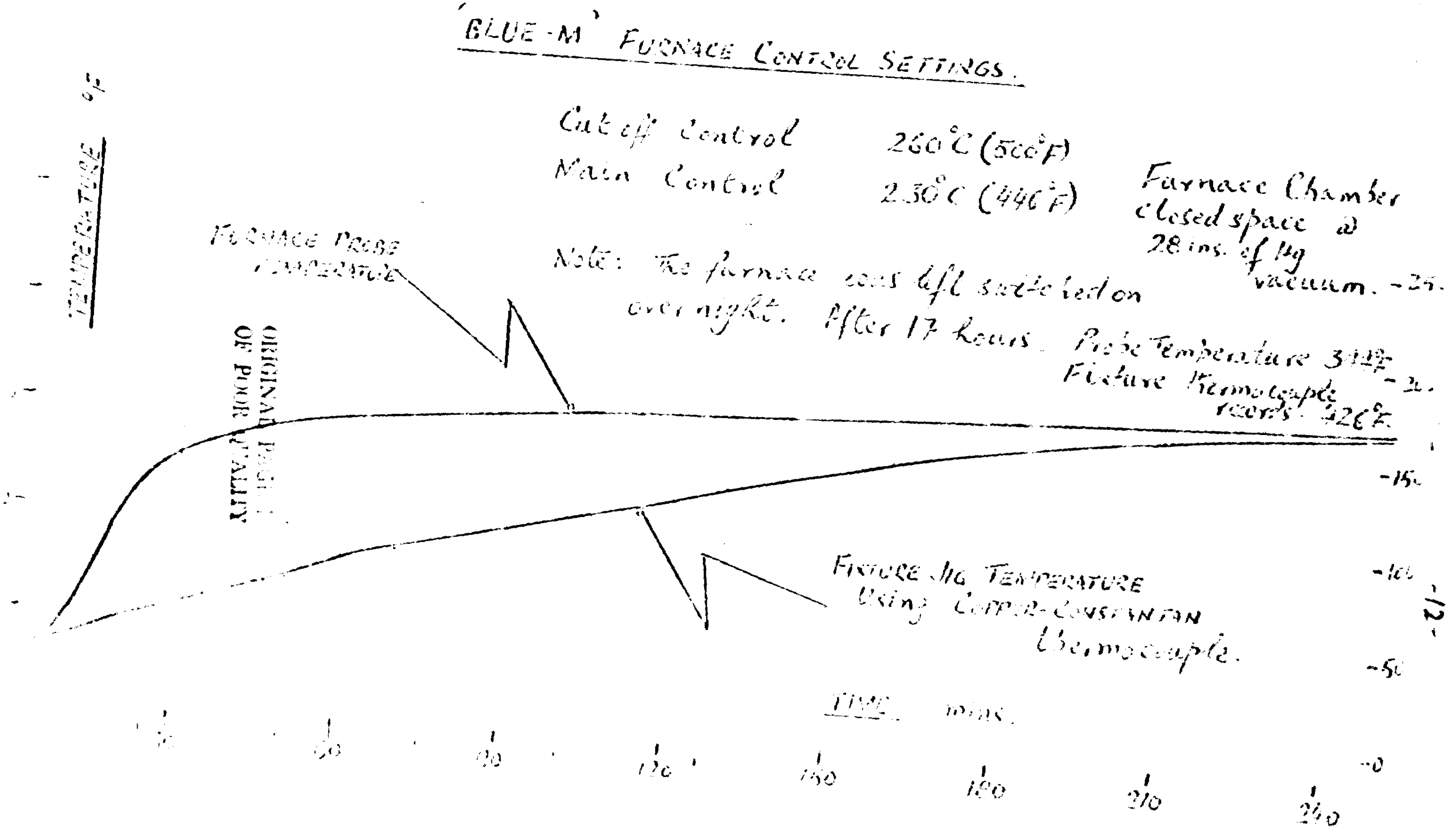
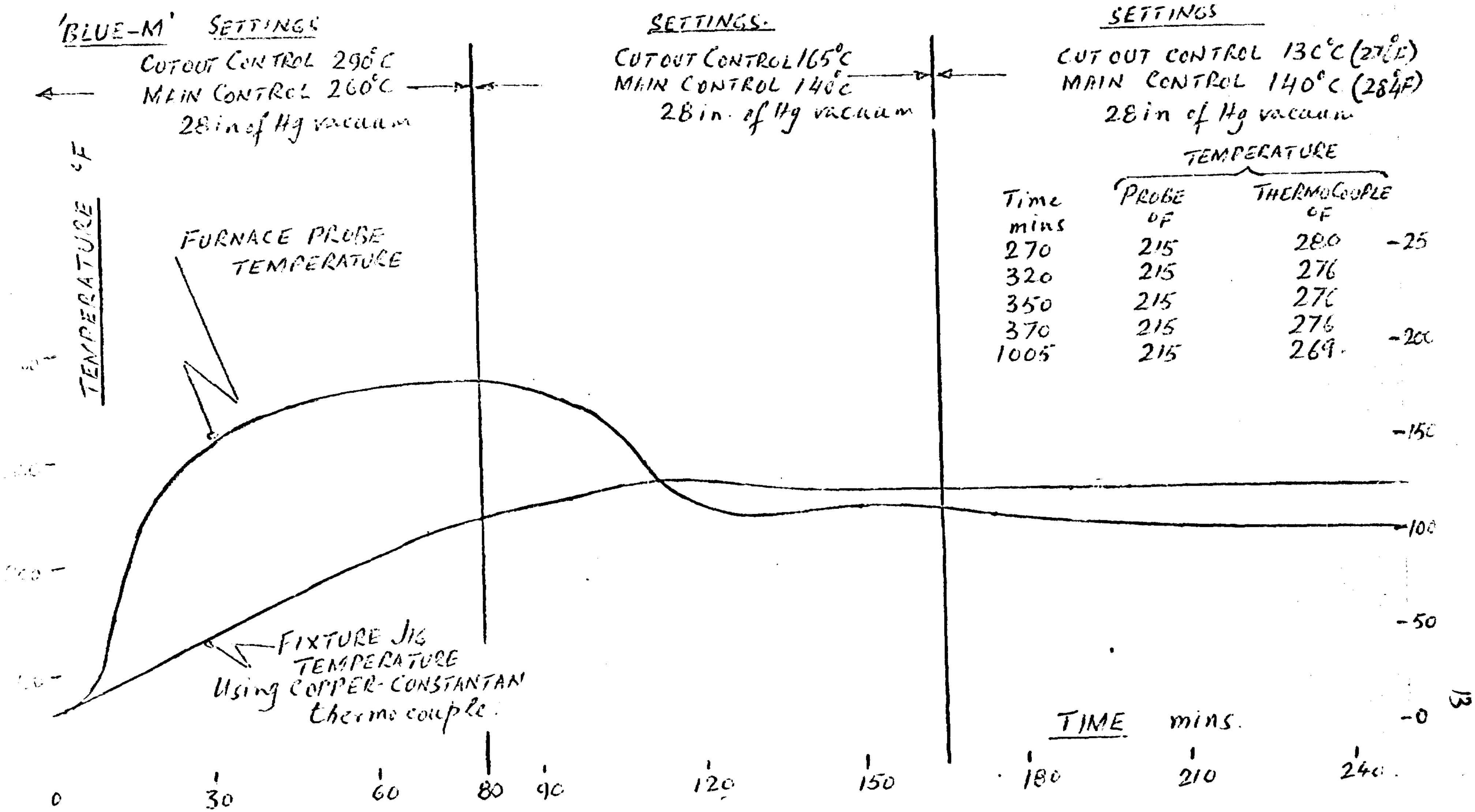


FIG. 5. FURNACE CALIBRATION NO. 3.



in contact with the furnace walls continues to heat up and after 17 hours the fixture had reached a stable temperature around 426°F (219°C).

The probe measuring the radiation stable state is confirmed while the fixture which receives heat from the furnace walls by conduction reaches a much higher temperature. See Fig. 4.

Furnace Calibration No. 3

It was observed that at atmospheric condition the temperature rise is rapid. Hence the following sequential steps were chosen.

The furnace chamber and the chamber atmospheric vent were closed. The vacuum pump was switched on. The cut out control was set at 290°C and the main control was set at 260°C. The furnace was switched on with the fixture in the chamber space with the thermocouple duely attached. After 80 minutes had elapsed the controls were adjusted to

Cut out control @ 165°C

Main control @ 140°C

The temperature variation is shown in Fig. 5. After 165 minutes the controls were further trimmed to:

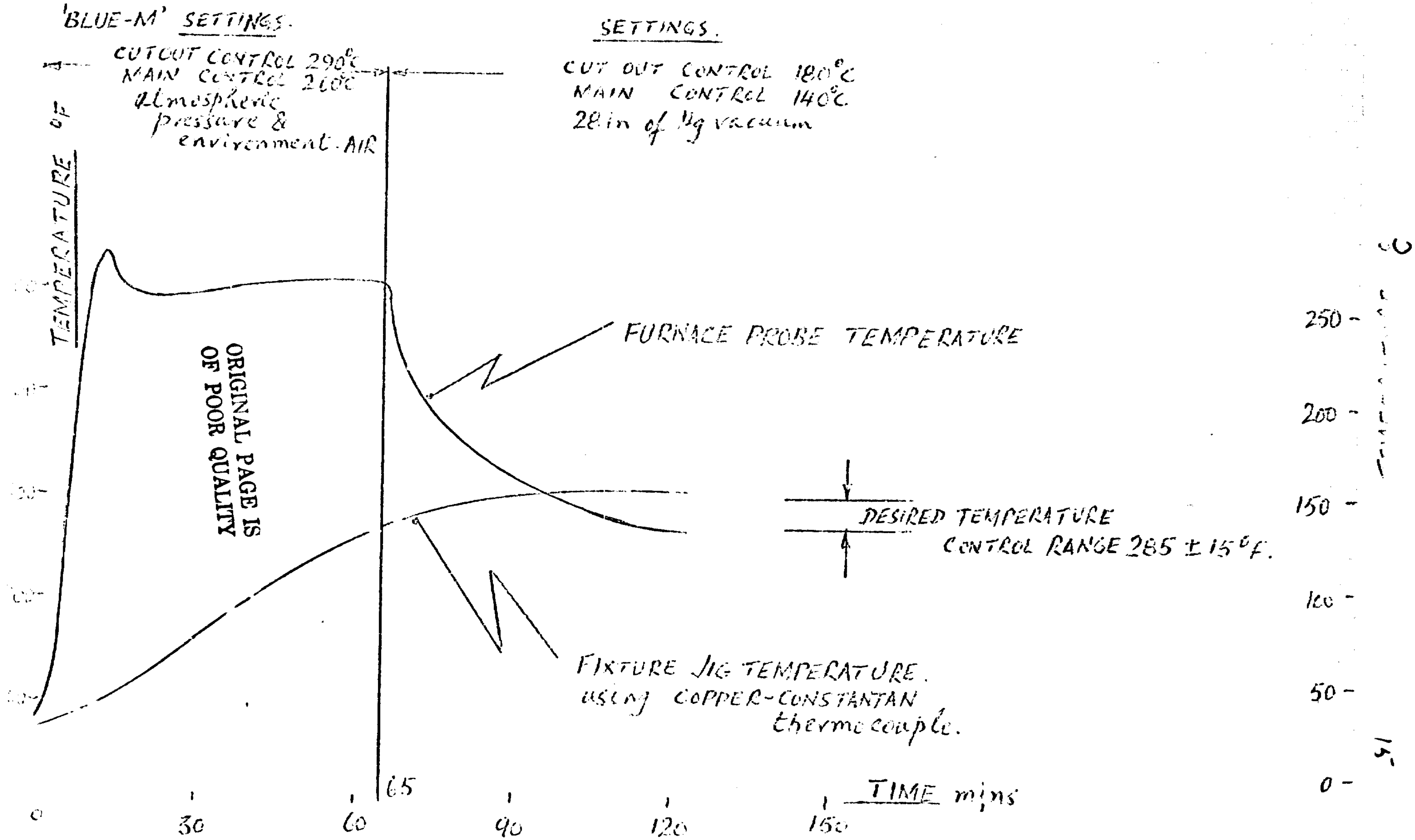
Cut out control @ 140°C

Main control @ 130°C

The final variations in temperature settlement are shown and the two temperatures are also quoted for time lapses of 270, 320, 350, 370 and 1005 minutes.

It can be surmised that the optimum difference for least thermal cycling variation occurs when the cut out and the main

FIG. 6. FURNACE CALIBRATION No. 4



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control differ by at least 30°C. Also that the initial heat build-up time should be in the presence of atmospheric pressure. And the vacuum pump should be started at the end of this heat build-up time of 70 minutes.

Furnace Calibration No. 4

From the conclusions above the furnace was finally calibrated. The fixture jig was placed in the center of the heating chamber with the thermocouple appropriately connected. The controls were set as cut out @ 290°C and the main at 260°C. With the furnace door and the atmospheric vent closed the power was switched on. At the conclusion of 65 minutes of heat build-up time, the fixture temperature was 270°F (132°C). The controls were now trimmed to the following settings:

Cut out control @ 180°C

Main control @ 140°C.

The chamber was evacuated to 28 in. of Hg (6.8 kPa). The stabilization and the temperature variation are shown in Fig. 6.

From these furnace calibrations the following sequential procedure was adopted.

1. Cut and assemble prepreg-mylar laminate blanks into fixture jigs. Apply loads by loading screw to give interlaminar pressure of 15 psi.
2. Place both jigs at the center of the furnace chamber with copper-constantan thermocouple tip sandwiched between the jigs.
3. Close furnace door, atmospheric vent and the vacuum valve.

4. Set the 'BLUE-M' furnace controls at

   Cut out @ 290°C

   Main @ 260°C

5. Switch power on and allow a heat build-up time of 70 to 75 minutes.

6. Reset cut out @ 177°C and the main @ 137°C and switch the vacuum pump on.

7. Allow a steady state curing time of 90 minutes.

8. Switch off in the following sequence:

   a. close vacuum valve.

   b. open atmospheric vent.

   c. switch off vacuum pump.

   d. switch off power.

   e. open furnace chamber door and allow to cool for 10 minutes.

   f. remove fixture jig on to a bench till the temperature drops below 140°F (60°C) and then remove the cured specimen blank.

Finish Machining Test Specimen Blanks

For profiling the finished specimen two template jigs were manufactured.

Tensile Template

This is a two part sandwich system produced from a 1/4 inch (6.35 mm) thick sheet steel. One half has clearance holes and the other has the holes suitably threaded. The holes are in the same relative position as the indexing punched holes in the laminated composite specimen blank. The specimen blanks are

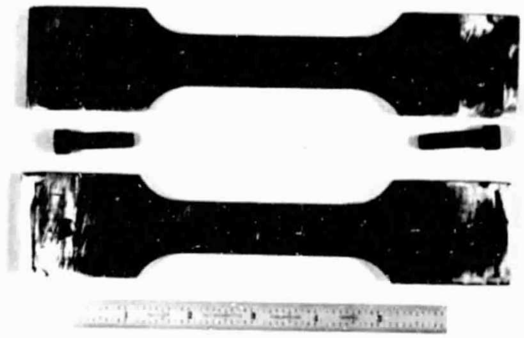


PLATE NO. 7. Templates - Tension Test Specimen.

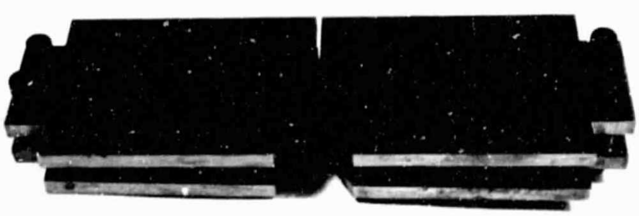
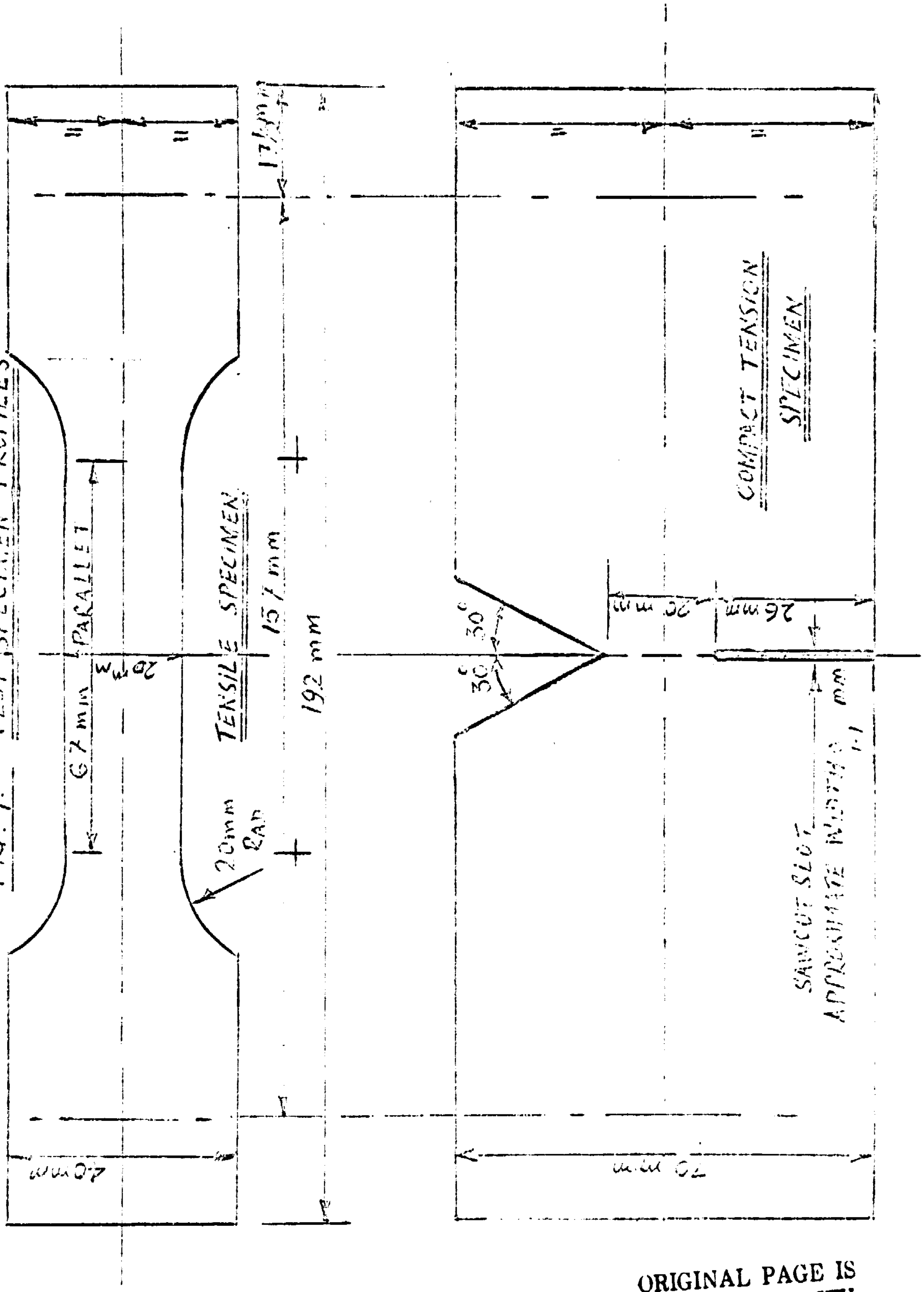


PLATE NO. 8. Templates - Compact-Tension Test Specimen.

FIG. 7. TEST SPECIMEN PROFILES



thus located and clamped between the templates. The excess material that projects is first machined off on the band saw and then rough filed. Now the backing waxed paper is removed from the test specimens which are reassembled into the templates and finish fine filed to the exact template dimensions. The templates are shown in PLATE NO. 7 and the tensile test specimen profile is in Fig. 7.

Compact Tension Template

This is also a two piece system produced from 1/4 in. (6.35 mm) steel sheet. One half has clearance holes while the other has corresponding threaded holes. The two plates sandwich the specimen blanks by securing screws, see PLATE NO. 8. The projecting edges of the cured blanks are first machined by a band saw and then rough filed to approximate size. The waxed backing paper outer covers are removed and the blanks are reassembled into the template. The specimens are now finished using a fine file. The slot in the test specimen is first cut with a hack saw and the roots of the slot and the vee notch are finished off with a fine tooth jeweler's saw. The dimensions of the finished compact tension specimen are in Fig. 7.

Finally it can be stated that with the procedures so adopted and the techniques described we are able to reproduce geometrical similarity between individual specimens to within ± 0.03 mm. Such control has given us repeatable tensile and compact strength data to within ± 5% for similar test specimens.

Ann Arbor
May 1980