SEMI-ANNUAL REPORT NO. 3

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FOR

OFFICE OF RESEARCH GRANTS AND CONTRACTS

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

WASHINGTON, D.C.

CONTRACT NO. NsG 619

CONTRACT PERIOD: MAY 1, 1965 - OCTOBER 31, 1965

SYRACUSE UNIVERSITY RESEARCH INSTITUTE

DEPARTMENT OF CHEMICAL ENGINEERING AND METALLURGY

MET. E. 1189-1165-SA

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OFFICE OF RESEARCH GRANTS AND CONTRACTS NASA

S.U.R.I. Report No.

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I. INTRODUCTION

The program has been expanded considerably since the second semi-annual report was submitted. A second vacuum system has been constructed, which enables us to achieve a better vacuum. Specimens can be exchanged more quickly, and the sample can be repeatedly loaded and unloaded at all temperatures. A titanium sublimation pump additionally to the Vac-Ion pump has been incorporated into the system. The glass sections have been reduced and the glass parts are easily removable and replaceable. Thus, delays due to glass breakage have been markedly reduced.

We achieved at room temperature pressures of $2 \cdot 10^{-10}$ mm Hg after a 2 hour outbaking at 180°C. The sample section has been heated to temperatures of 800°C. The heater should be powerful enough to obtain temperatures well above 1000°C in the test section. Length changes of the specimens are measured photographically using a 35 mm camera. These films are now analyzed on a microscope stage. This makes it possible to determine $\Delta L/L$ presently to about $1 \cdot 10^{-4}$. By using a fine grain film we should be able to increase the sensitivity to approximately $\Delta L/L \simeq 10^{-5}$.

Results on creep tests on polycrystalline copper show that the strength of a specimen changes noticeably if tested with either an argon bombarded "clean" surface or a reoxidized surface.

II. EXPERIMENTAL ARRANGEMENT

A. Test System I

The experimental data, as reported in section III of this report, were obtained in the vacuum system described in the Semi-Annual Report II[1]. Only the specimen support, the thermocouple position and the loading mechanism were altered. This vacuum creep test system will be referred to as Test System I.

Figure 1 gives a schematic diagram of the new specimen support and the system to measure length changes. The sample is looped at both ends around two nichrome wires A and A'. It was found that the sample did not break at the nichrome wire. Presumably the radius of the nichrome wire is large enough to reduce sufficiently stress concentrations at the sample end. Samples did break relatively frequently at the previously used "knot" (Ref. 1, p. 17).

The nichrome wire A extends from the top of the sample to point B, and nichrome wire A' from the lower end of the sample to point B'. The distance between B and B' indicates the length change of the sample. The tips of the nichrome wire, B and B', (see Fig. 1) are photographed. Their distance on the film is measured on the microscope stage of the metallograph.

A silica tube is slipped over the top detection wire and positioned at point C in order to insulate the specimen from the nichrome detection wire. This insulation is necessary to avoid cold-welding between sample and the top detection wire during argon bombardment. The thermocouple is inserted through the ceramic tube D and spot-welded to the nichrome wire. The thermocouple is placed through a two-hole ceramic tube for electrical insulation.

The unloading mechanism has been redesigned so that the sample can be repeatedly loaded and unloaded under vacuum. This design reduces the time consuming "bake-out cycle" in the testing procedure because the load can be released if the sample does not fracture during a test run. The sample can then be reannealed and the load can be applied with the sample at higher temperature. Figure 2 gives a schematic diagram of the new load release mechanism. This load-release mechanism, which is connected to the sample, consists of a flat stainless steel section of 1/8" thickness (A) and a movable nichrome wire (B) (Fig. 2a to b). Fig. 2a shows the system before the load (C) is connected to it. When the load C is to be applied, it and wire D are lifted by a solenoid. The horizontal section of wire D lifts wire B_2 and wire D slips past B_2. Then B_2 drops to its horizontal position, carrying wire D with the load as soon as the solenoid is lowered. When the load C is to be removed, C and D is lifted again with the solenoid. D lifts section B_1 of the wire B. B_1 is caught at E (Fig. 2b). Then wire D can slip past wire B_2 during lowering of the solenoid. With this the load is released. This mechanism allows repeated loading and unloading in vacuum.

The method of argon bombarding is the same as that outlined in the second semi-annual report. A serious crack developed in the glass section of this System I. It is presently in the process of reconstruction. Improvements as found in Test System II will be incorporated into the rebuilt Test System I.

B. Test System II

Test System I has several disadvantages. Loading and unloading of the sample

takes a long time. Extensive use of glass makes it rather fragile, and the heater contains several non-metal components which increase the pressure due to prolonged outgassing.

It was therefore decided to build a second vacuum system (Test System II), which avoids most of these shortcomings. Glass sections are short. The load support is very rigid. The load can be attached and removed easily. Additionally to the 8 l/sec Vac-Ion pump, a 50 l/sec Ti sublimation pump is used. Both pumps are very close to the test section.

Fig. 3 gives a schematic picture of the vacuum system.

As shown, a Welch-Duo-seal mechanical pump is connected in series with a NRC Type 149 metal diffusion pump (pumping speed 60 l/second), a Kontes glass valve, a Kontes cold trap, a 1" Granville-Phillips ultra-high vacuum valve, and two Varian cross connections to the test cell. In addition, a Vac-Ion pump (pumping speed of 8 l/second), a Varian Titanium Sublimation pump (pumping speed of 50 l/sec.) and a Granville-Phillips variable leak valve are connected to the ultra high vacuum section by flanges of the Varian cross connections. The two vacuum gauges placed as shown and the Vac-Ion pump allow an accurate determination of the pressure in the system. Two Varian flexible couplings connected in series to a Varian cross connection allow a linear movement of about 1.5" inside the test cell. A 45 Amp electrical feed through closes the Vacuum system at the flexible coupling. The "feed through" is used for the electric current of the specimen heater. All connections, including the test cell connection, are made with Varian conflat flanges. High purity argon (10 ppm) is admitted through the Granville-Phillips variable leak valve into the ultra high vacuum section.

Figure 4 gives a schematic diagram of the test section. Two stainless steel rods (A) and two copper rods (B) form the frame for the specimen support (C). The rods carry also a set of 3 Mo-radiation shields (D) and a Ta-heating wire (E). The Ta wire has a diameter of about 0.030" and is self-supporting. Four Al-oxide rods separate it from the radiation shield. The specimen is attached at one end to a supporting nichrome wire (F), at the other end to the load (G). The copper rod (H) can be lifted to support the load (G). This copper rod, which is insulated, has also a flexible connection to the heater (E) on the top end. Its lower end is connected to the "high current vacuum feed through" attached to flexible bellows (See Fig. 3).

The temperature of the sample is measured with a thermocouple pair (I), which has a pressure contact to the electrical feed through (K). This allows one to remove the glass-envelope (L) easily.

The test procedure for Vacuum System II will be as follows. The mechanical pump is turned on and allowed to pump the whole system down to about 10^{-1} Torr. The oil diffusion pump is then activated to bring the system down to about 10^{-5} Torr. At this pressure the wire specimen is annealed with the tantalum wire coil resistance heater (E, Fig. 4) (resistance of about 1Ω). The system is then "baked out" at temperatures between 300° and 350°C for about 2 hours. During this process the pressure will rise. The system will be allowed to pump back down after "bake out" to about 10^{-6} Torr. The Granville-Phillip's ultra high vacuum valve is then closed. Argon will then be introduced to the system via the Granville-Phillips leak valve. The argon will be purified with the titanium sublimation pumps which will remove only contaminants from the system. The specimen is then "cleaned" by bombardment with argon ions.

The specimen is connected through the thermocouple (I, Fig. 4) to a negative potential (above 500v.) with respect to the heater coil. This figure for the required potential difference is determined from the appearance of the glow associated with argon ionization.

At this point there are two possible ways to conduct the experiment. One, the argon may be left in the test cell and assumed to have no effect on the test results. Two, the Granville-Phillips valve can be opened to the rest of the system (which is about 10^{-6} Torr) to allow the oil diffusion pump to remove the argon. The Granville-Phillips valve can then be closed again with the Vac-Ion pump operating to bring the test cell down to its ultimate vacuum. This method has the disadvantage that it may expose the freshly "cleaned" specimen to contaminants during the pump down of Argon. Both methods will be tried in an effort to determine the effect of the argon atmosphere.

The specimen and test cell should now be ready for the actual test. The load has not been applied but rests on rod H as shown in Fig. 4. The specimen is heated up to the test temperature. This temperature will be measured with the thermocouple (I) (see Fig. 4). The load will then be applied to the specimen by lowering the bellows assembly with the mandrel press. On Fig. 4 the system is shown in its initial, unloaded state with the copper rod H in position X. The load is applied to the specimen if rod H is in position Y.

35 mm photographs are taken of the detection wires, (M, Fig. 4), at about 10 second intervals to determine the elongation of the specimen. If the specimen does not fracture, the specimen is unloaded and the system is opened to the atmosphere at elevated temperatures to allow the specimen to oxidize.

The system is then closed and re-evacuated, the load reapplied, and photographs taken again. The results of the two sets of photographs are then compared and conclusions drawn. No actual test runs have been made as yet though the heater has been tested to temperature up to about 800°C.

III. PRELIMINARY RESULTS

As stated in the second semi-annual report (Ref. 1) "clean" samples of copper wire showed little deformation. However, the same sample broke rapidly when tested in the oxidized condition at the same temperature and a similar pressure. Further tests have been conducted in Test System I and the results are given below. It should be pointed out that the temperature given for each test is only nominal. The temperature reading depended noticably on the exact position of the thermocouple. However, the thermocouple position was fixed in each of the tests I to III. Therefore, we could keep the temperature in each run constant within a few degrees. The uncertainty in temperature means that these measurements are still of a preliminary nature. (Test System II, which is now ready for testing has a much better temperature control). Test I and II show therefore only that the present system is sensitive enough to measure creep rates. Only Test III gives information on the effect of the surface structure on creep rates.

Test I

A test was performed in order to obtain a creep curve for a sample tested without surface treatment. The sample was annealed at 600°C for an hour prior to the test. It was not argon bombarded. The pressure in the test chamber prior to the test was 10^{-5} mm Hg. A load of 185 gm was applied to this specimen which was at a nominal temperature of 375°C. The temperature remained constant to within 15°C. After 18 minutes the specimen had not fractured. The initial length of the specimen was 1.46 inches and the total strain was 1.8%. Figure 5 represents a creep curve for this sample. It can be seen that the scatter of the data is of the order of $\frac{\Delta l}{l} \simeq 0.2\%$.

Test II

The sample was annealed at 600°C for two hours. The vacuum system was subsequently baked out for a period of four hours at 300°C. The pressure in the high vacuum chamber prior to argon bombardment was 1×10^{-8} mm Hg. The sample was argon bombarded for two hours prior to the test. The load of 185 gm was applied to the specimen which was at a nominal temperature of 415°C. The sample fractured after 3 minutes. Only six pictures were taken. The initial length of the specimen was 1.43 inches and the total strain was 0.42%. Fig. 6 represents the creep curve for this sample.

Test III

A third test was performed in order to obtain a creep curve for a cleaned sample which is subsequently oxidized and retested. The wire sample was argon bombarded for 1 - 3/4 hours prior to testing. The pressure prior to argon bombarding was 7×10^{-8} mm Hg. The load of 185 gm was applied to the "clean" sample which was at a temperature of 465°C. The elongation was recorded for ten minute interval and the load was removed. The vacuum chamber was opened and the sample was oxidized for 45 minutes. The temperature in the chamber dropped to approximately 200°C during the oxidizing period although the voltage applied to the heater wire remained constant. The system was subsequently pumped down to a pressure of 5×10^{-6} mm Hg and the load was reapplied. The temperature in the test section had returned to 460°C. After a period of about five minutes this oxidized sample fractured. Fig. 7a and b gives the creep curves for this test. Although there is some scatter in the data points it appears as if the reoxidized sample fractured without

significant creep. Although the results are by no means conclusive, the shape of the two curves seems to indicate that oxidation of a sample decreases the creep rate and embrittles the sample. Once the oxidized sample begins to yield, it fractures rapidly.

IV. SUMMARY AND CONCLUSIONS

A presently constructed vacuum system is now capable to reach pressures of about 10^{-10} mm Hg. This should prevent noticable contamination during testing. It is now also possible to clean the argon before argon bombarding starts inside the ultra high vacuum section. The heater has been used to about 800°C. It should be capable to heat the sample well above 1000°C. The new specimen loading system makes it possible to load and unload the sample gently at all temperatures.

Measurements on the older vacuum system (Test System I) show that the creep rate of copper is noticably affected by its surface structure. The data indicate that the creep rate of a sample cleaned by argon bombarding is larger than after the same sample is reoxidized "in situ". The change of creep rates should be associated with changes in the surface structure of the sample.

V. REFERENCES

1. K. Schroder and A. Giannuzzi, Semi-Annual Report No. 2, May 1965

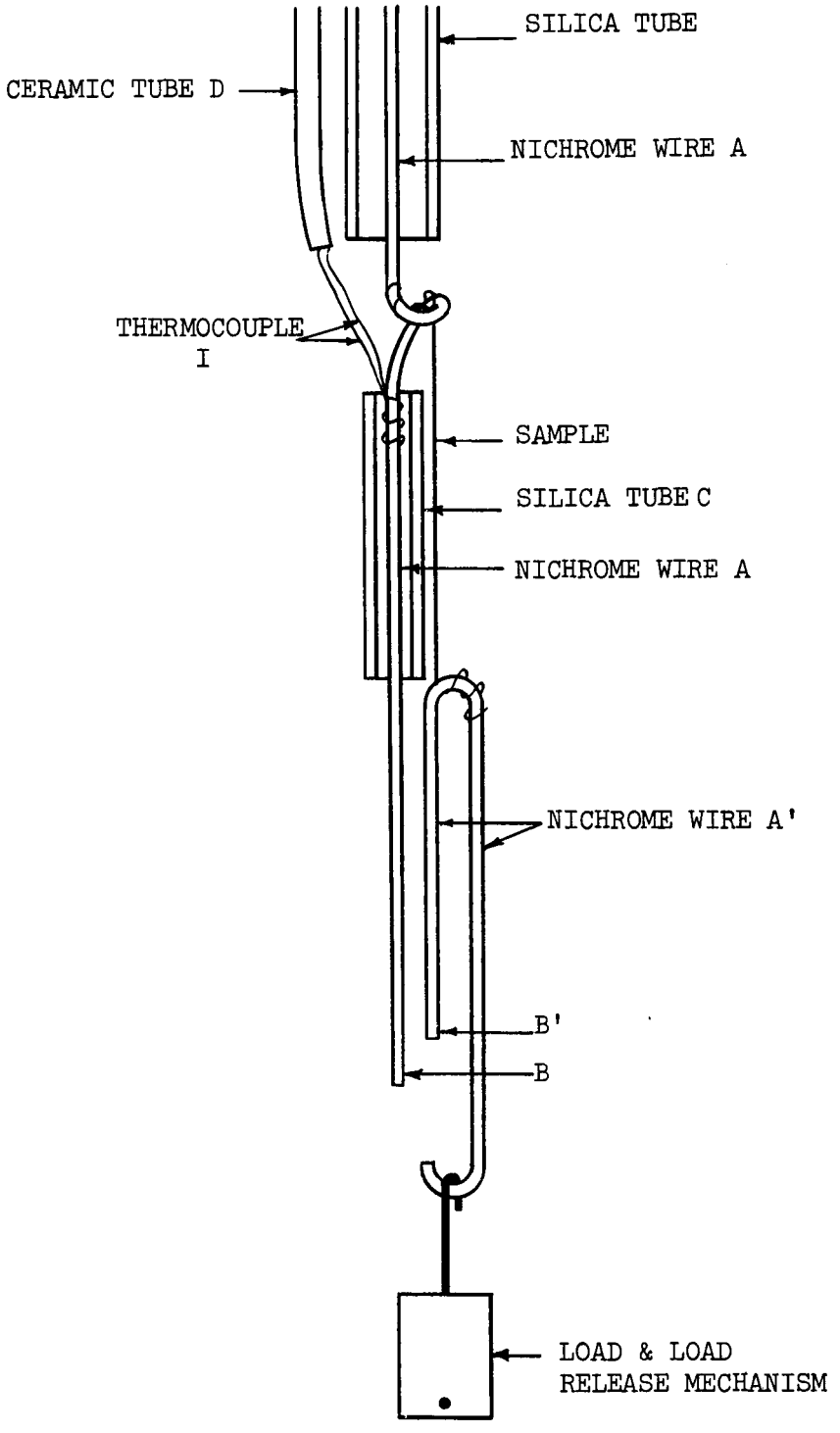


FIG. 1 SAMPLE AND SAMPLE SUPPORT (SCHEMATICALLY).

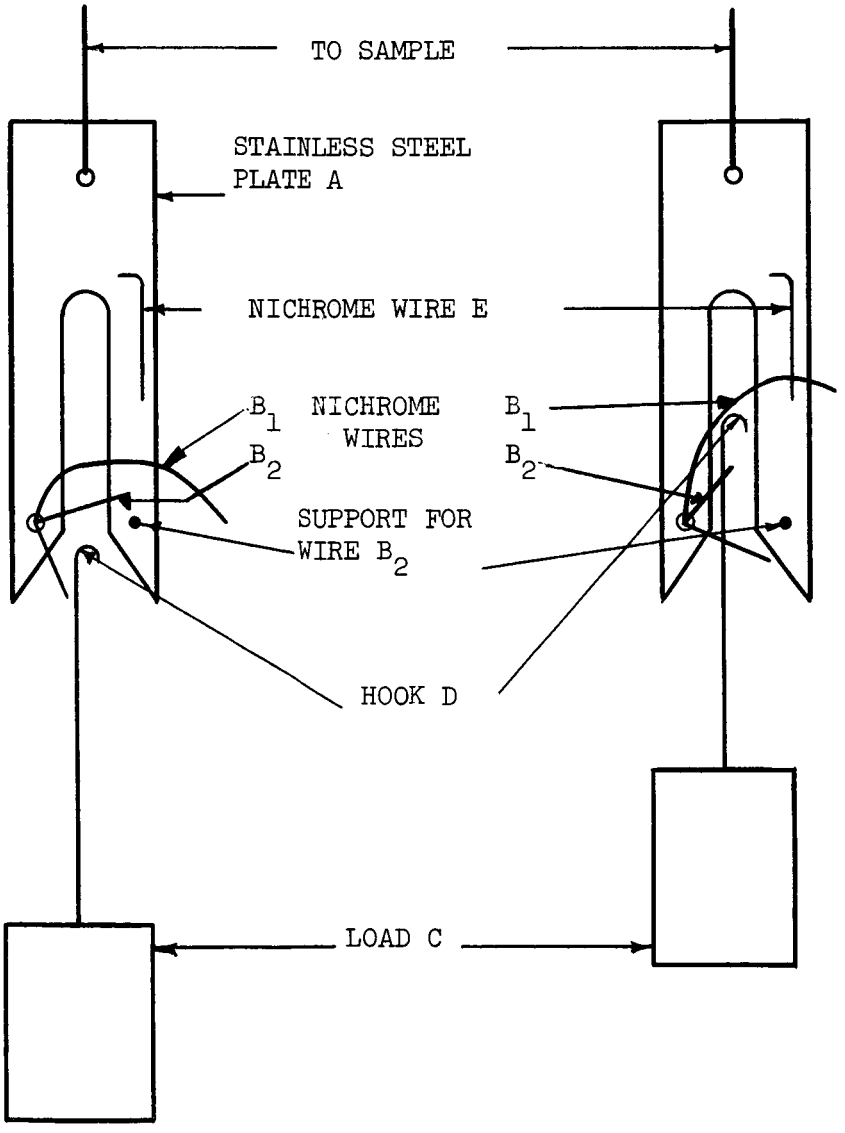


FIG. 2a FIG. 2b

FIG. 2 LOAD RELEASE MECHANISM (SCHEMATICALLY).

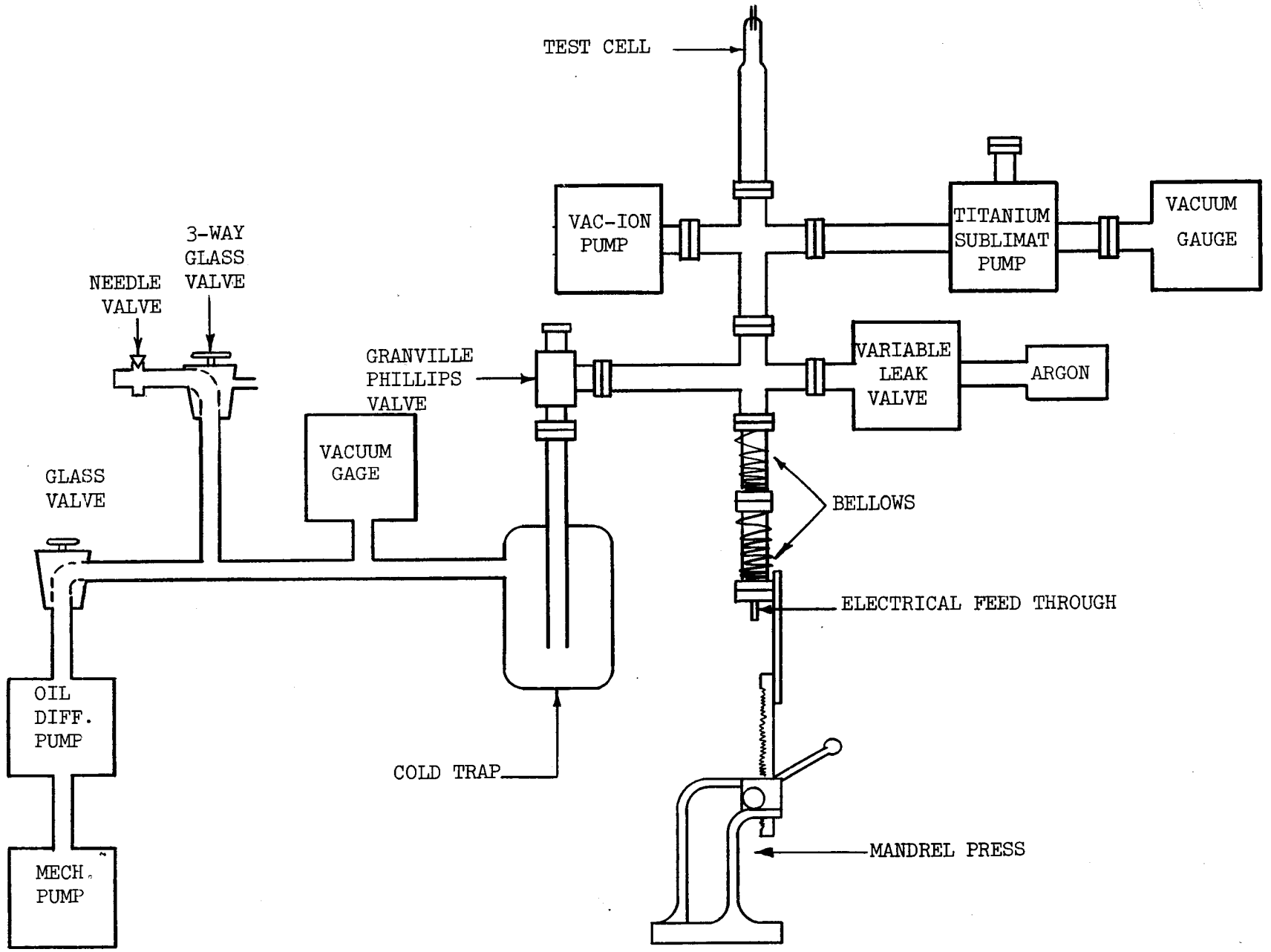


FIG. 3 SCHEMATIC DIAGRAM OF THE VACUUM SYSTEM II

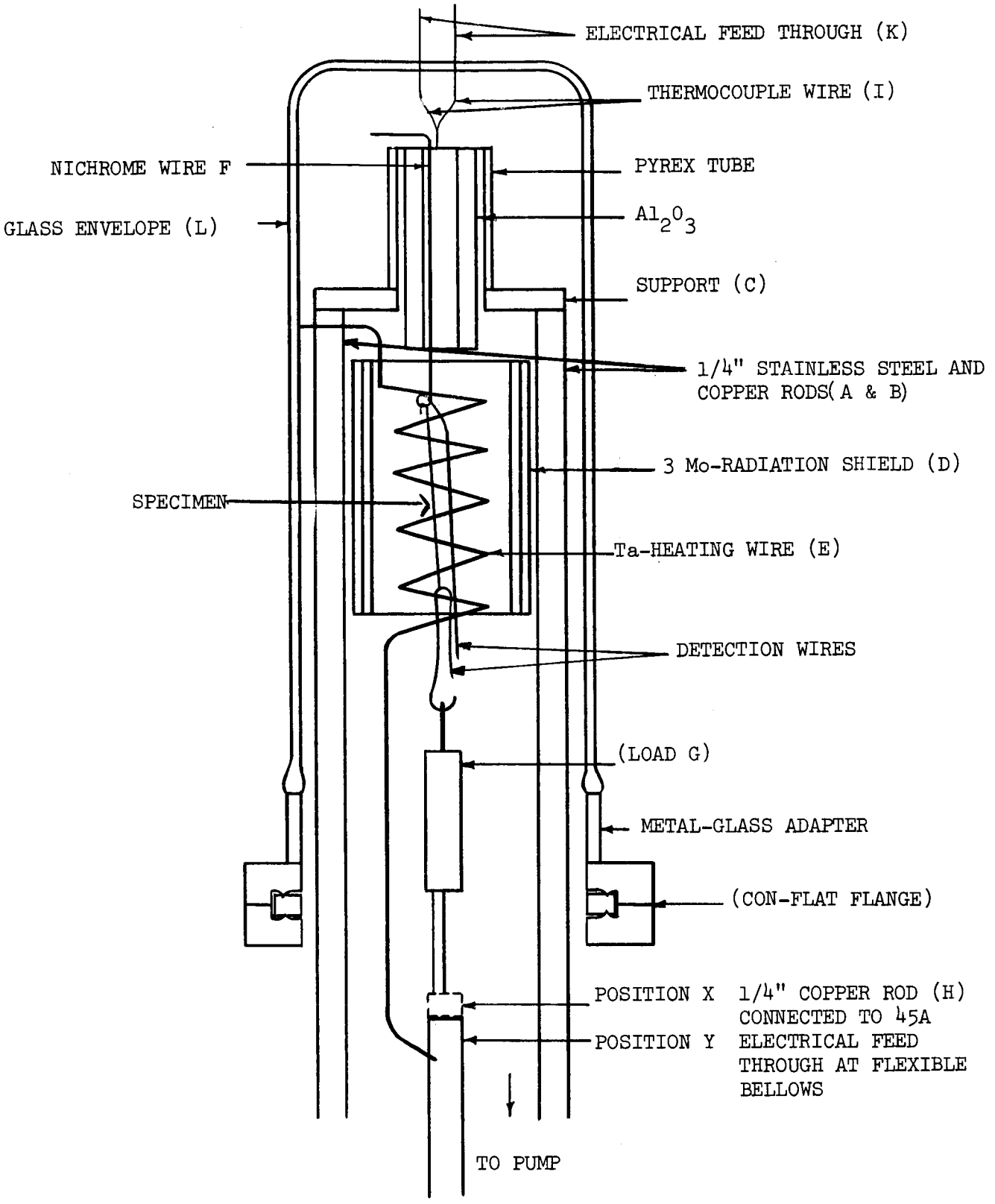


FIG. 4 TEST SECTION IN TEST SYSTEM II.(SCHEMATICALLY)

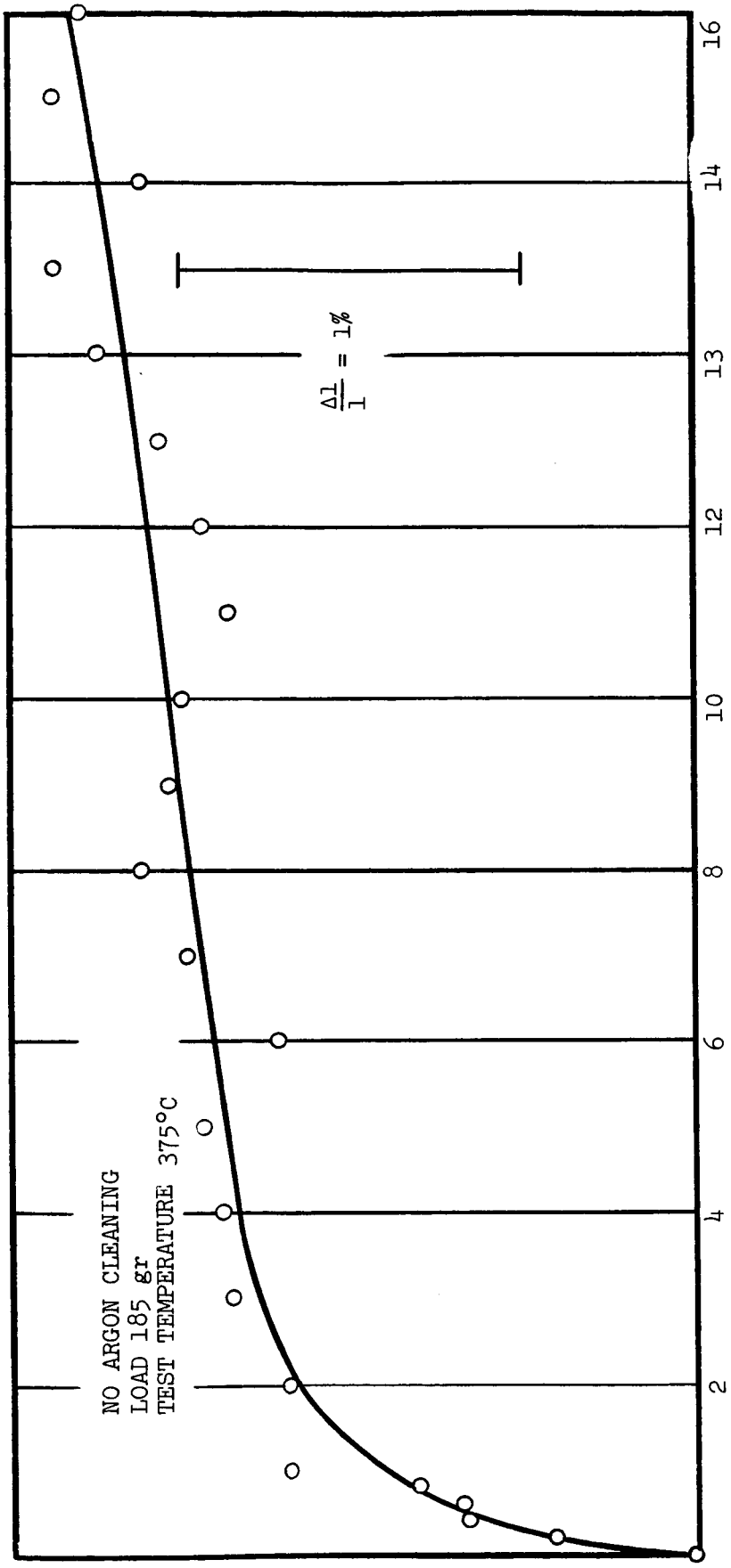


FIG. 5 CREEP OF 0.008" COPPER WIRE.

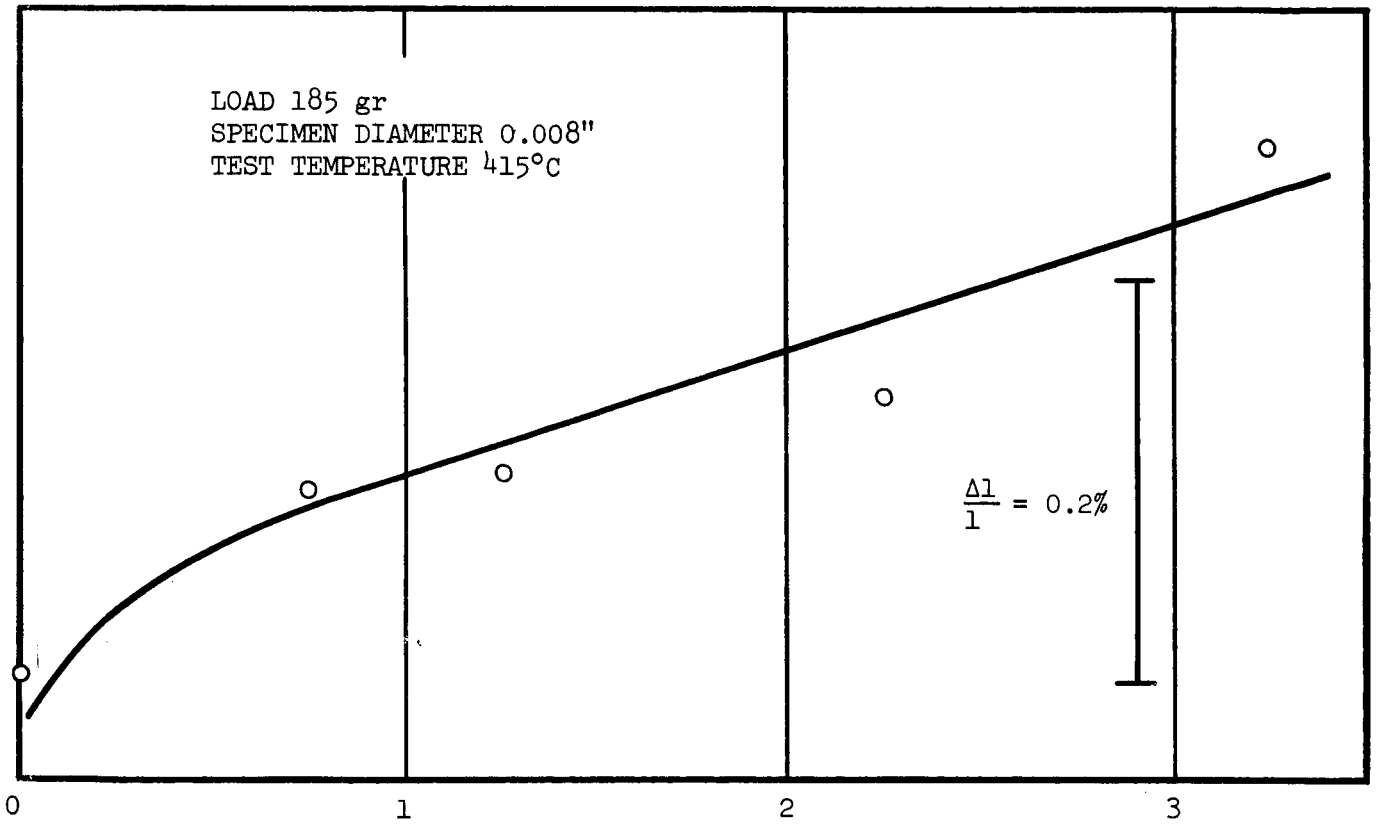


FIG. 6 CREEP OF 0.008 INCH COPPER WIRE SPECIMEN ARGON BOMBARDED PRIOR TO TESTING

FIG. 7B TEST ON OXYDIZED SAMPLE

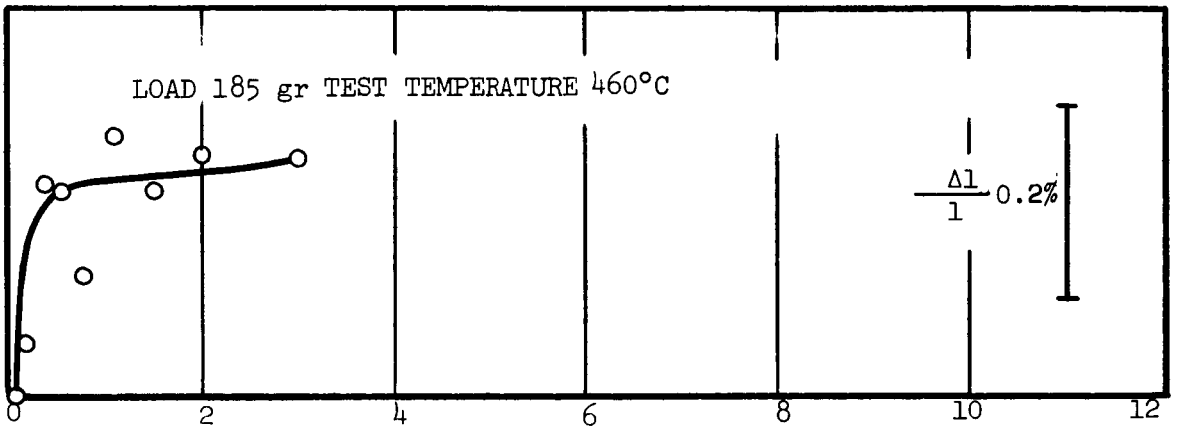
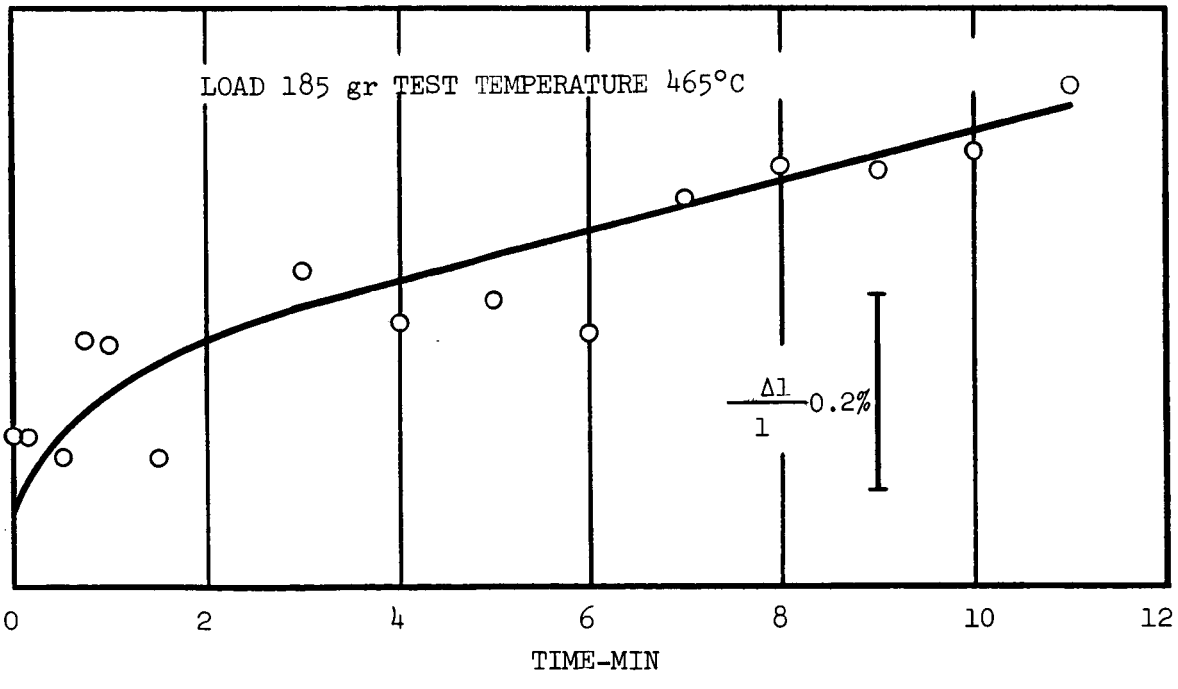


FIG. 7A TEST ON "CLEAN" SAMPLE



7. CREEP TEST OF 0.008" Cu WIRE SPECIMEN CLEANED AND TESTED, THEN OXIDIZED AND TESTED.