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CREEP AT ELEVATED TEMPERATURES AND HIGH VACUUM

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ABSTRACT

A series of high purity copper wires were creep tested in a high vacuum at temperatures between 785 and 855° C. They were tested in both the argon bombarded, clean state, and after oxidizing in air at these elevated temperatures.

It was found that the creep rate of the sample decreased noticeably after oxidation for about one hour between 400 and 500° C. The sample fractures in the oxidized state in tests above 835° C. The largest minimum creep rate of the argon bombarded, clean sample was about 0.1% per minute, whereas the largest minimum creep rate for the oxidized sample under the same stress of about 850 gr/cm^2 was less than 0.02% per minute.

It is proposed that the creep phenomena can be explained with a model, in which oxide films act as dislocation barriers for dislocation. Further experiments are required to elucidate this mechanism and the fracture process.

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I. INTRODUCTION

The previously described Test System II [1] has been used for a series of creep tests on polycrystalline wire in the temperature range from 785° C to 855° C. The wire was either a commercial grade copper wire of 0.008" diameter or a 0.01" diameter high purity (99.999%) copper wire. Experimental results are given in Section IV.

Further improvements have been incorporated into the two test systems available for high temperature, high vacuum creep testing. Test System I has been completely reconstructed. It is now, except for the glass envelope of the test section, a metal system. Preliminary tests showed that the improved System I should be able to achieve the same level of performance as System II. Their high vacuum systems differ only in their electrical feed-through arrangement for the heater, the thermocouple and the connection to the high voltage supply for argon bombarding. A partial pressure gauge has recently been acquired. It should be most useful in future tests, in which the sample surface will react with a controlled atmosphere.

The creep experiments were continued to confirm previously reported [1] preliminary measurements made on commercial grade copper wire which had a lower creep rate if tested in the oxidized state than in the clean state. Similar results were found by other authors. Stress strain curves, fatigue and creep tests show [2] that a high vacuum or surface removal during testing can influence the strength of metals. The work hardening rate in stress-strain curves decreases and the fatigue life increases frequently with decreasing pressure. The experimental results of vacuum tests are, however, sometimes rather complicated. Shahanian and Achter [3,4,5] found for instance that the relative creep rates of high temperature alloys tested in air and in a vacuum were rate dependent. At low creep rates, the creep rate is lower in air than in a vacuum, at higher rates it is reversed. A lower work hardening rate in vacuum testing can be understood if one assumes that surface films obstruct the movement of dislocations and act as barriers [4]. It may be possible that dislocations may form stronger obstacles and entanglements near the surface under atmospheric conditions, because more impurities can diffuse into the metal if tested in air than if tested under vacuum conditions. The complicated load, temperature and pressure dependence of the creep rate indicates that both softening and hardening effects may be introduced by vacuum testing. Hardening may be due to changes in the impurity distribution near the surface; weakening due to a reduction of surface energies in air [4]. It was also proposed that nitrogen could reduce the creep strength because it could react with impurities in the grain boundaries. This could promote crack propagation [5].

This indicates that the effect of an atmosphere on deformation is rather complex. In section IV, results of creep tests are given in which the specimens are always tested in a high vacuum. This should make it easier to determine the effect of oxide films alone on creep rates, whereas in most previously published data tests in an atmosphere and in vacuum are compared with each other. It is expected that the interaction of dislocations with clean and oxidized surfaces can be more easily separated by this testing procedure.

II. EXPERIMENTAL PROCEDURE

The major features of the Test System II have been described in the previous report [1]. The experimental results described in Section IV of this report have been obtained in this Test System.

Figure 3 of the previous report [1] gives a schematic diagram of the experimental arrangement. The test section, given in Figure 4 of that report,[1] is practically unchanged. Only a new load support has been incorporated recently into System II. This support is now identical to the load support in the modified System I, as described below. The Redhead gauge has been recently replaced by a Varian Partial Pressure Gauge. This will be used extensively to study the effect of highly defined contaminating atmospheres, as soon as the present series of tests of the effect of oxygen by air is completed.

The schematic diagram of the modified Test System I is given in Figure I. The center of the high vacuum section consists of two cross-fittings. Attached to these are: an ultra high vacuum valve (A), which connects the high vacuum system to a diffusion pump, an electrical feed-through with eight leads (B), flexible bellows for the loading mechanism (C), a Vac-ion pump (D), and the test section (F). Figure 2 shows schematically the flexible bellows section, which allows it to release or support the weight attached to the specimen. Its operation is not affected by pressure changes between test section and atmosphere.

A diagram of the test section is given in Figure 3. It is very similar to the system used previously [1], except that it has 6 radiation shields instead of 3. They are supported by a stainless steel tube. Preliminary tests showed that the sample can be heated to 800°C, that it can be argon-bombarded without disturbing glow discharges in other sections of the test system, and that it can be loaded and unloaded easily. Only the thermocouple has to be changed before higher temperatures can be achieved.

The testing procedure has been described previously (Section II of ref. 1). Surface cleaning, oxidation procedure and sample loading was done usually in the following way: The system was evacuated with a diffusion pump. The unloaded wire was then annealed for about 2 hours at 800°C. The pressure in the system was about 10^{-5} mm Hg. Then the sample was argon bombarded for about 40 to 90 minutes at room temperature at a pressure of about 0.15 mm Hg. The current density was approximately 0.5 mA/cm^2. The purity level of argon is estimated to be of the order of 0.01%. The system was then evacuated to a pressure of a 10^{-8} to 10^{-9} mm Hg. The sample reached its testing temperature between 750 and 850°C usually within 20 to 30 minutes. This time interval and the temperature should be long enough for recovery from defects produced by argon bombarding. The temperature was measured with a thermocouple attached to the sample holder. The pressure during testing was about 10^{-7} mm Hg. Finally, the load was applied and the specimen length change was recorded photographically. Following this immediately, the sample was unloaded, air was admitted into the system for about one hour and the sample oxidized. The heater current was kept constant. The heat loss due to convection and diffusion in air reduced, however, the oxidation temperature to well below the testing temperature.

The pressure was again reduced to about the same level of the first part of the creep test, the load applied, and the length change again recorded in 1/4, 1/2 and 1 minute intervals. The sample was usually replaced at the end of this sequence. In one case, the same sample was again argon bombarded, tested, oxidized, and retested at higher temperatures. It was found, however, that argon bombarding did not remove the oxide film completely. The elongation was too small to be measured.

III. EXPERIMENTAL RESULTS AND DISCUSSION

Experimental results are given in Figs. 4 to 10 which contain also data on test temperatures, pressures, and specimen preparation. The test temperature in each test sequence, consisting of a test on one sample after argon bombarding and after oxidation, is estimated to be constant within $\pm 5°C$. The testing temperature is accurate to about $\pm$ 10°C, except for the tests 6 and 7, for which the specimen heater had probably a shorted section. The actual temperature in these two tests is probably about 20°C higher than the temperature indicated by the thermocouple.

These measurements on high purity copper confirm preliminary results obtained on commercial grade copper [1]. They show that the creep rate of samples tested in a high vacuum decreases markedly after oxidation. The sample fractures in the oxidized state at sufficiently high temperatures after an elongation of only a fraction of a percent.

Our results agree qualitatively with creep-rupture tests by Shahanian and Achter [4,5], who found for low loads that nickel-chromium and nickel specimens have a lower creep rate in air than in a vacuum. Under higher loads and lower temperatures, the behavior changed and samples tested in air had a higher creep rate. These authors suggest that one needs at least two competing processes to explain the results; a strengthening due to oxidation, and a weakening due to a reduction in surface energy by gas absorption.

Our present investigation confirms the suggestion that oxide films strengthen the material. This is not due to actual load carrying of the oxide film, but should be attributed to dislocation interaction with the oxide film. We would assume that dislocations pile up at the surface, as Head has suggested[6], and that the back stress of the pile up dislocations on the dislocation sources increases the strength of the metal.

It seems difficult to explain the low fracture strength of the oxidized sample with the idea that the surface energy is reduced by gas-adsorption. It may be possible that the surface diffusion of oxygen from the oxide film to slipsteps and cracks would lower the surface energy of cracks. It seems more likely, however, that impurities react during the oxidation process with grain boundaries or other defects of the crystal, which could make the sample more brittle.

Further studies in single crystals, variations of oxidation or nitriding, etc. time and temperature, and detailed metallographic studies on film thickness, grain boundary structure are required before it will be possible to construct a reliable model of the effect of surface films on the deformation processes in high vacuum.

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5. P. Shahanian and M. R. Achter Proc. Joint. Int. Conf. on Creep, 7-47 (1963).

6. A. K. Head, Austr. J. Physics 13, 778 (1962).

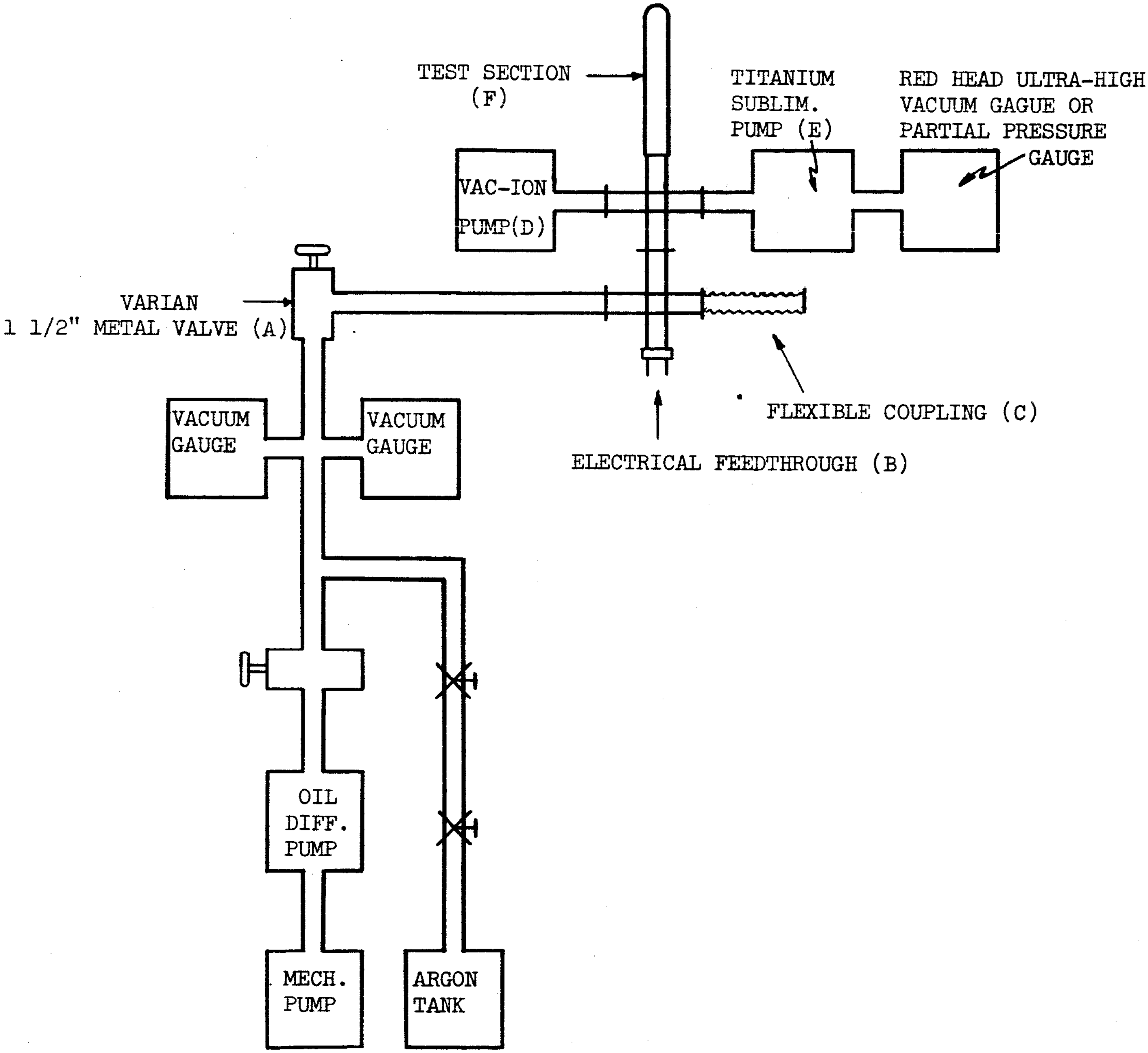

FIG. 1 SCHEMATIC DIAGRAM OF VACUUM SYSTEM

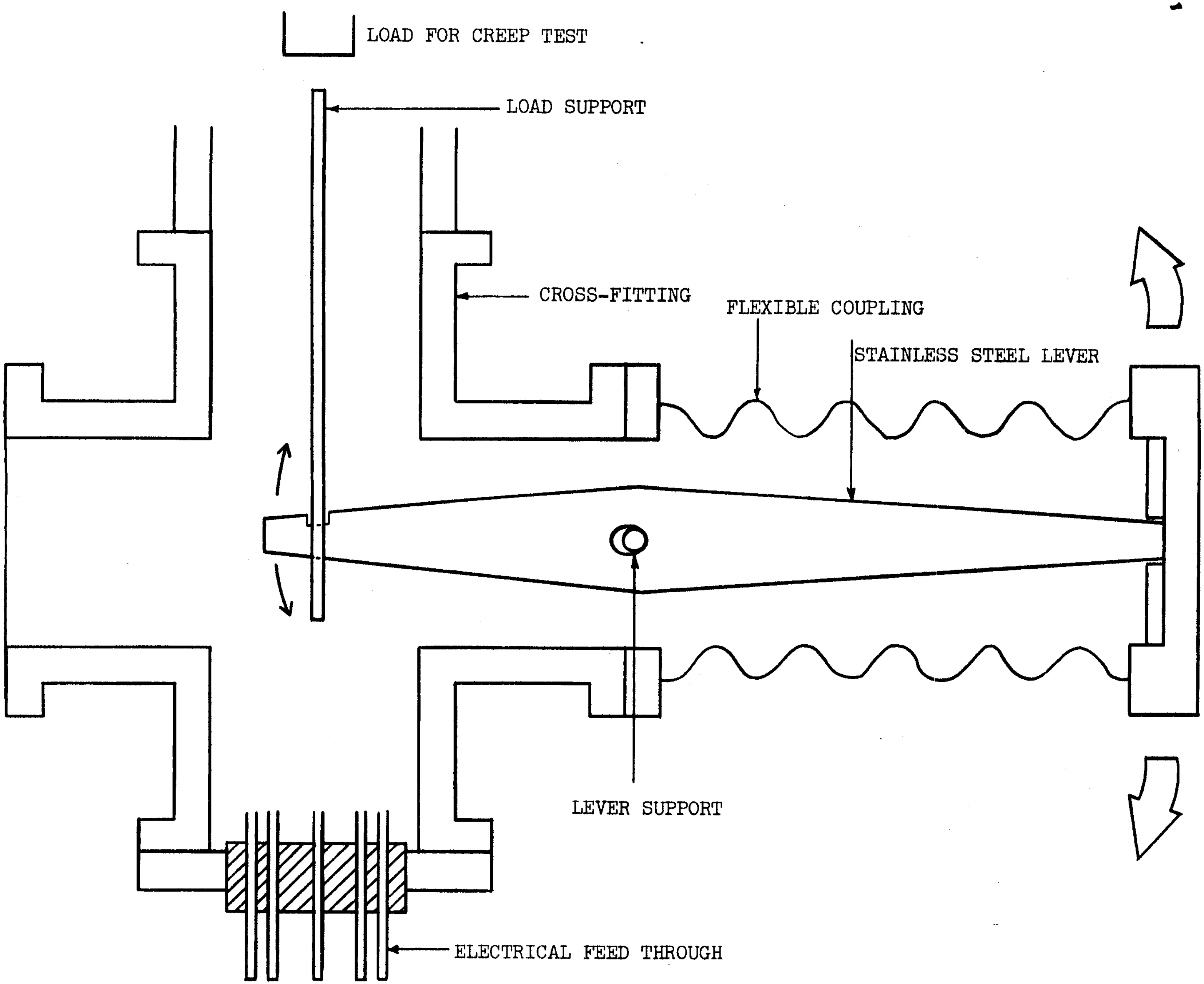

FIG. 2 SCHEMATIC DIAGRAM OF LOADING SYSTEM

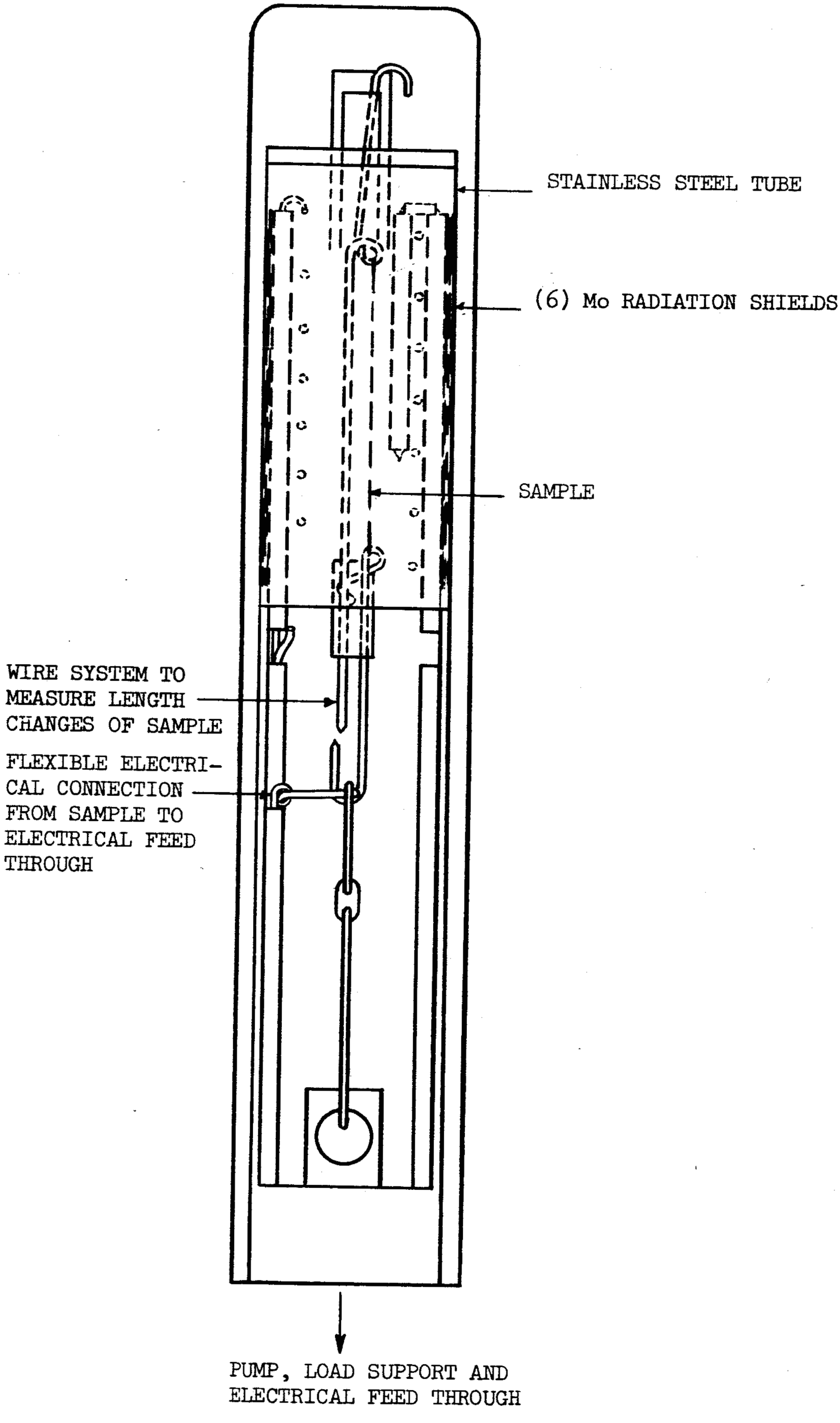

FIG. 3 SCHEMATIC DIAGRAM OF TEST SECTION

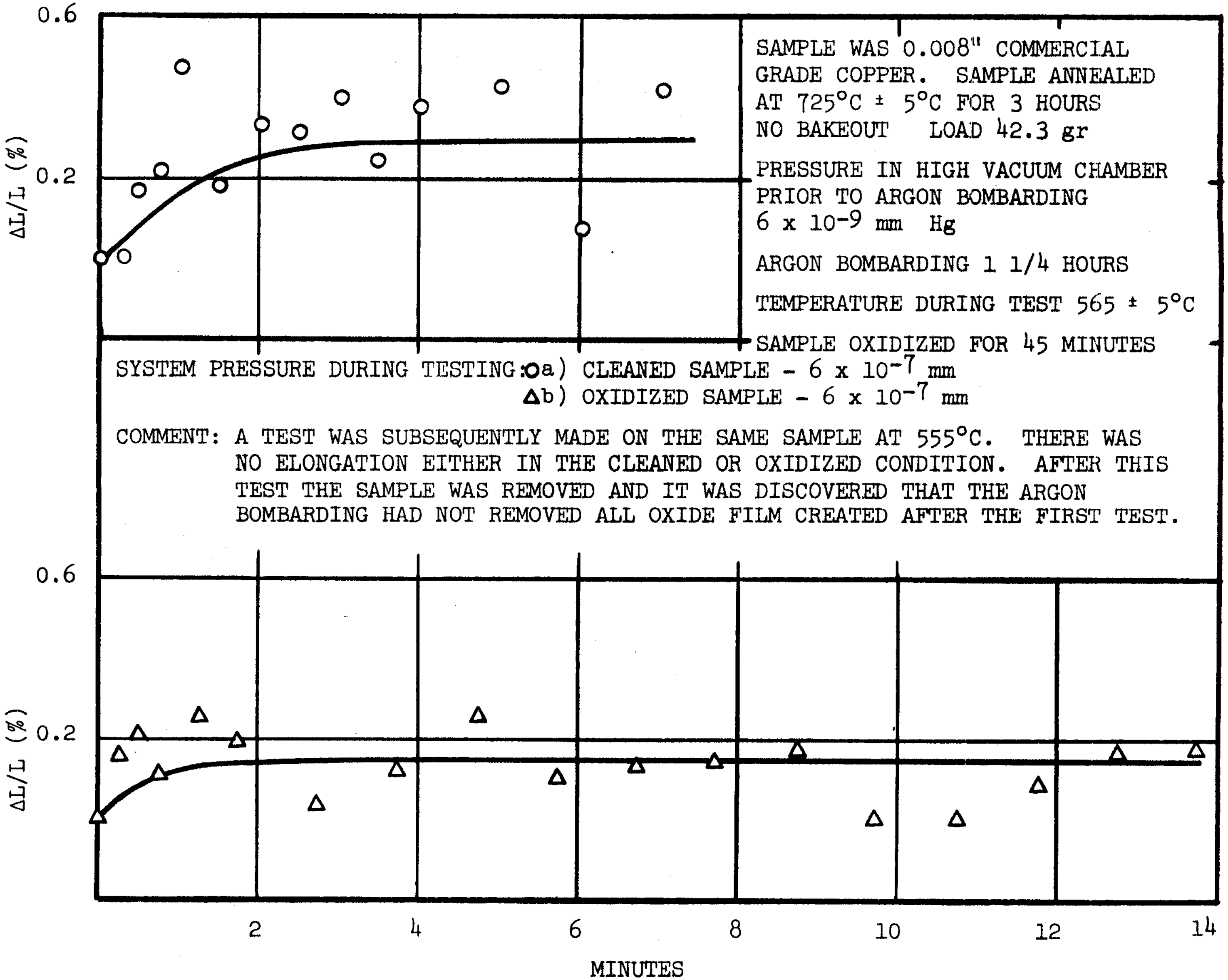

FIG. 4 CREEP TEST NUMBER 1 ON COMMERCIAL GRADE COPPER

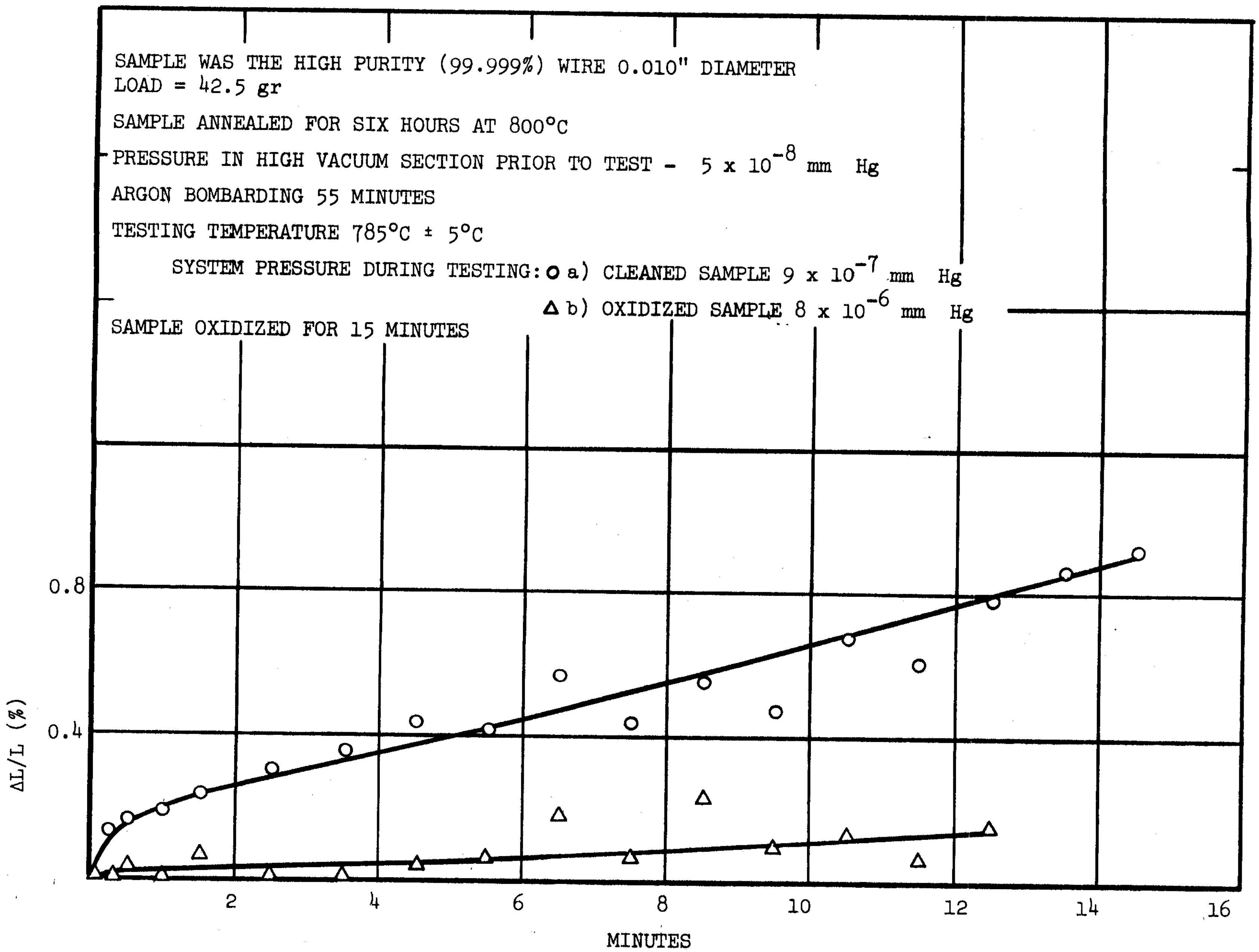

FIG. 5 CREEP TEST NUMBER 2 ON HIGH PURITY COPPER

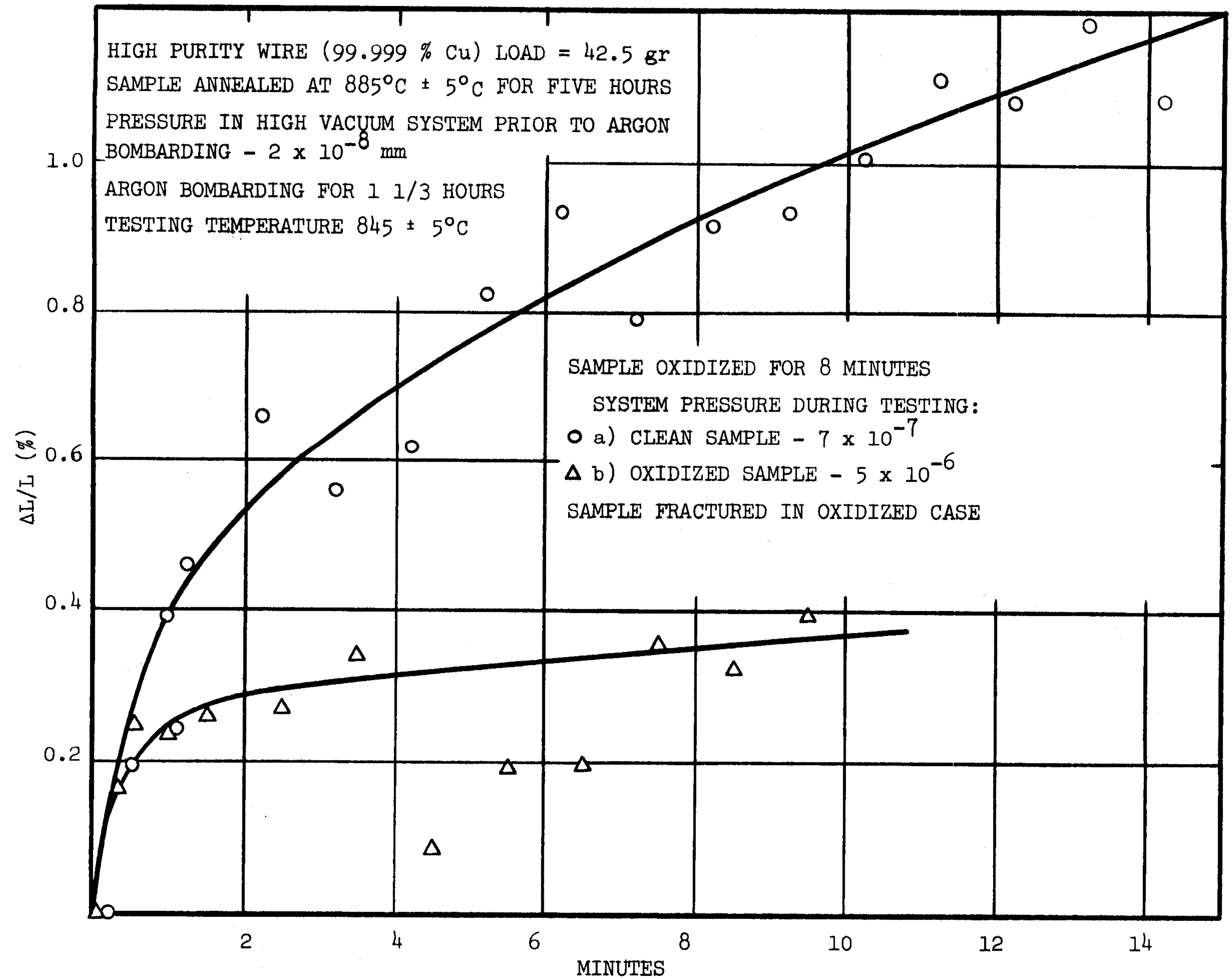

FIG. 6 CREEP TEST NUMBER 3 ON HIGH PURITY COPPER

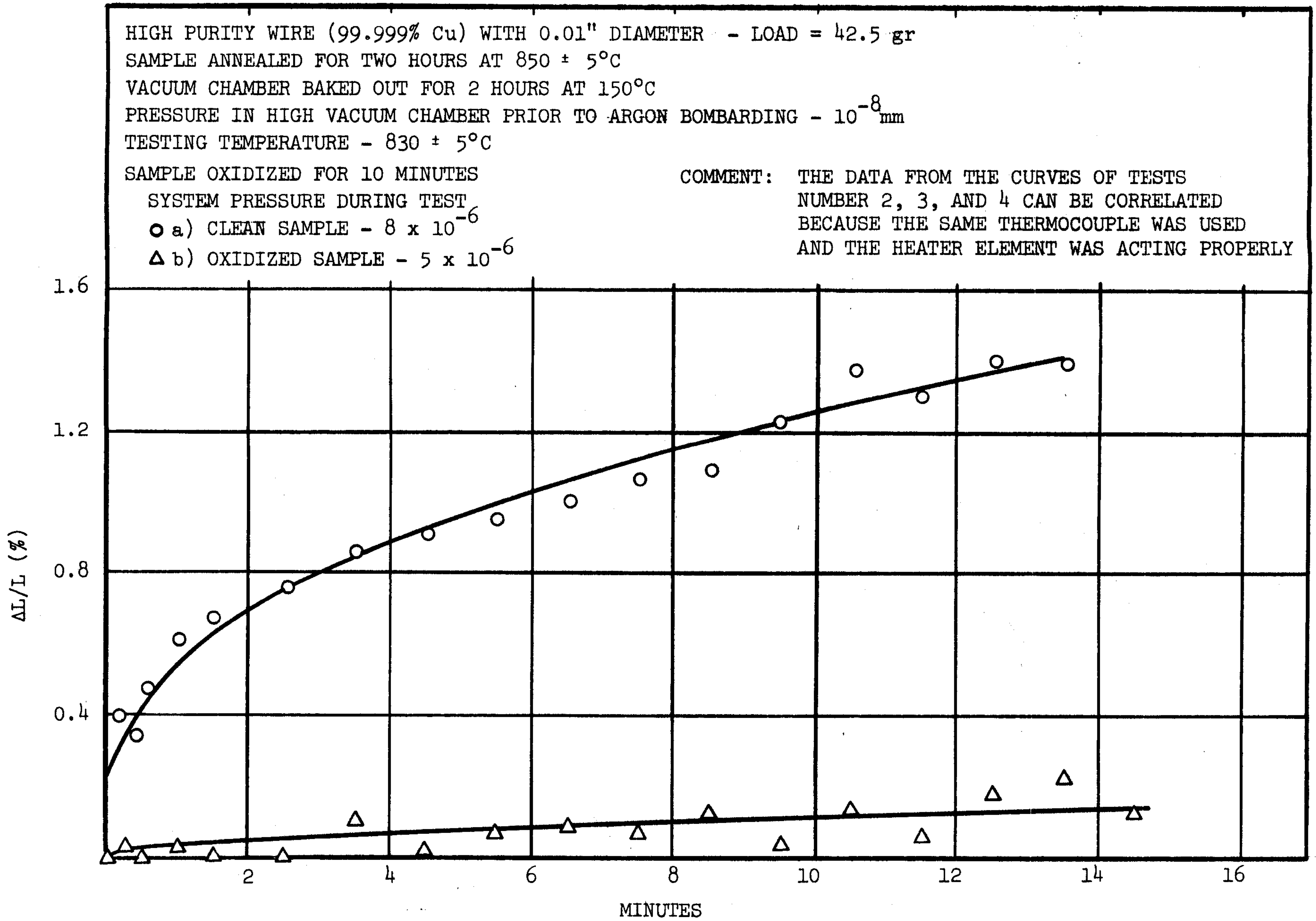

FIG. 7 CREEP TEST NUMBER 4 ON HIGH PURITY COPPER

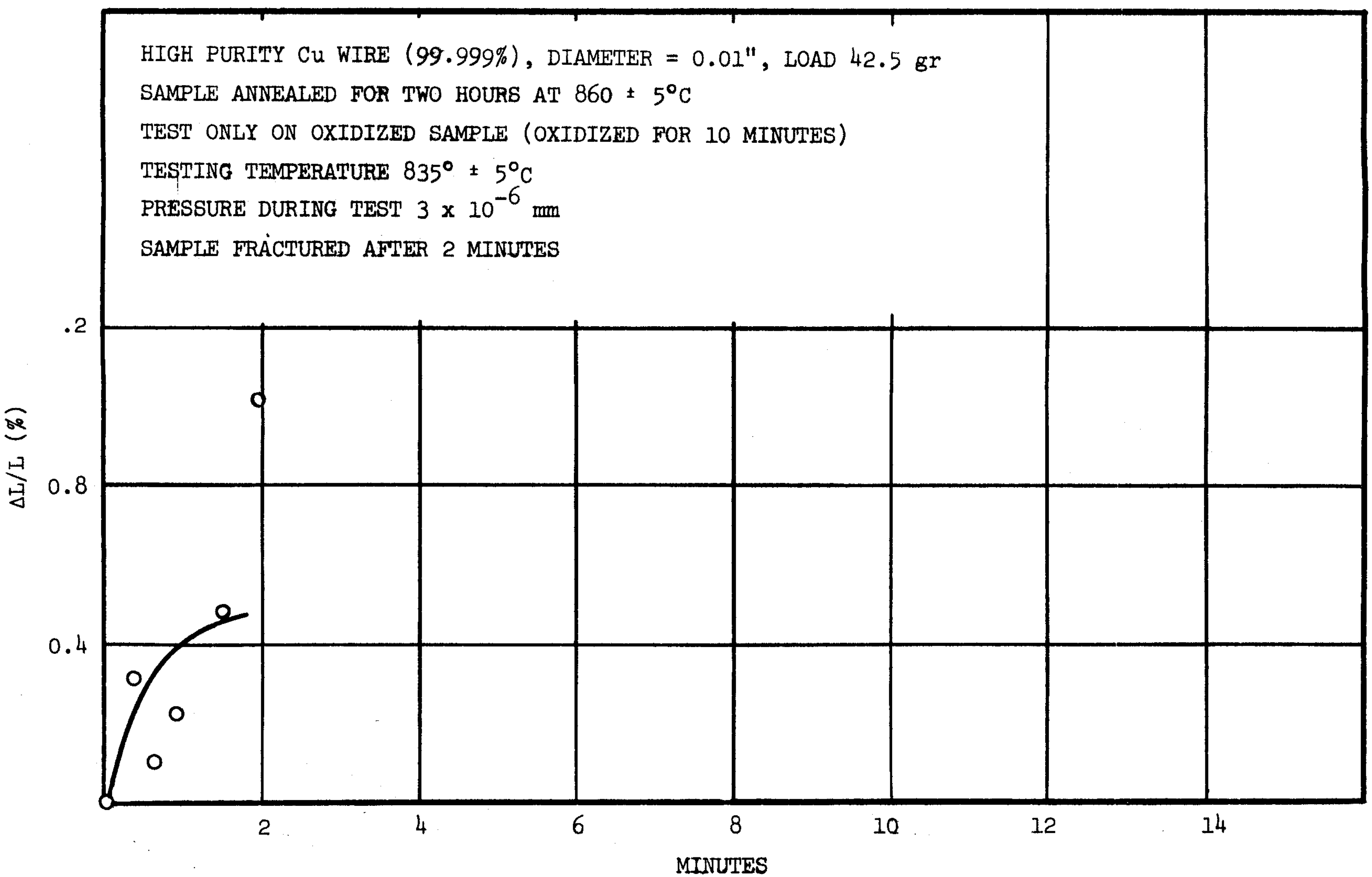

FIG. 8 CREEP TEST NUMBER 5 ON HIGH PURITY COPPER

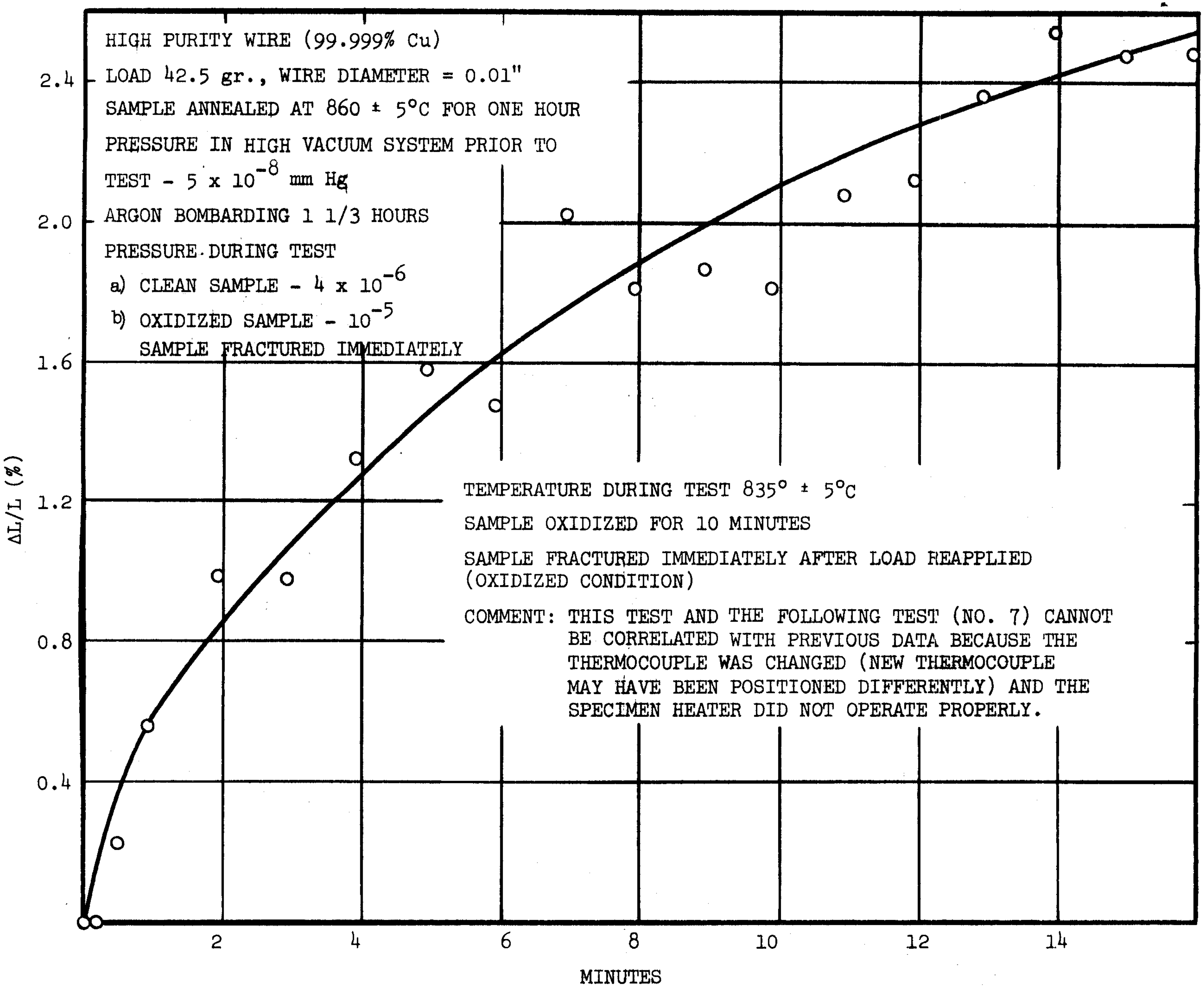

FIG. 9 CREEP TEST NUMBER 6 ON HIGH PURITY COPPER

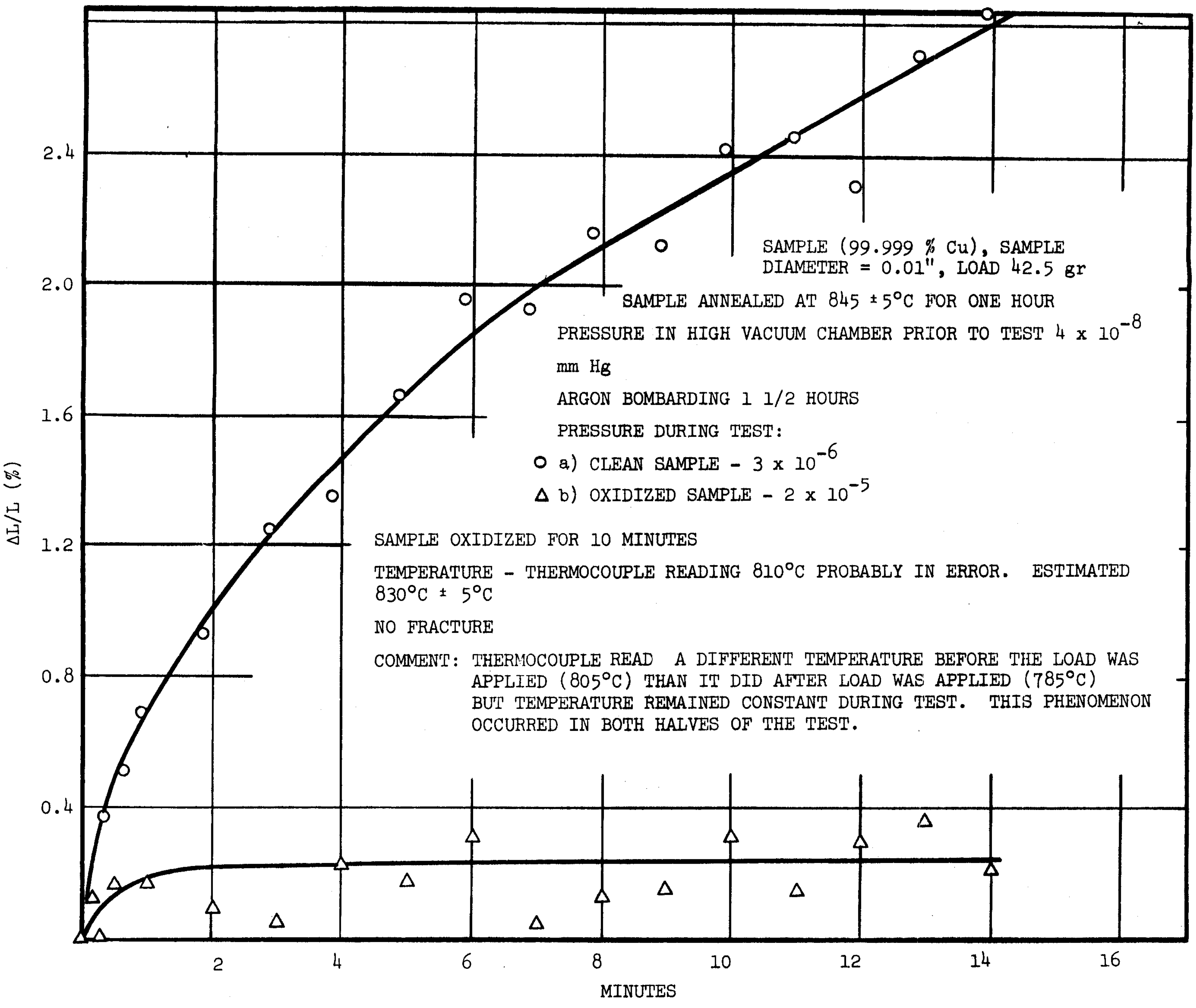

FIG. 10 CREEP TEST NUMBER 7 ON HIGH PURITY COPPER