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RELATION BETWEEN OXYGEN DISTRIBUTION AND CORROSION IN SOME REFRACTORY METAL-LITHIUM SYSTEMS

J. R. DiStefano
E. E. Hoffman

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ABSTRACT

The attractive physical and nuclear properties of lithium-7 make it highly desirable as a coolant or heat-transfer media in nuclear power reactors. Investigations to determine the compatibility of various container materials with lithium at elevated temperatures have indicated that, in general, the refractory metals – tungsten, rhenium, molybdenum, tantalum, niobium, vanadium, and zirconium – have good resistance to dissolution type of attack. This investigation studied four of these materials – tantalum, niobium, vanadium, and zirconium – to determine the influence of small amounts of oxygen in these materials on their corrosion resistance.

The Nb-O-Li system was studied in some detail and leads to the following conclusions. As the oxygen concentration in niobium increases, its corrosion resistance to lithium decreases. This type of corrosion action by lithium is not a dissolutioning process, but rather involves penetration of the material by lithium and the precipitation of a "corrosion phase." Under the conditions of these tests the thermodynamic distribution coefficient for the partitioning of oxygen in the system is such that oxygen is also gettered from the niobium by lithium and therefore weight-change data are not indicative of the extent of corrosion. Corrosion is a strong function of both time and temperature and has a deleterious effect on the mechanical properties of the niobium.

Studies of polycrystalline niobium specimens indicate that corrosion occurs both intergranularly and transgranularly. The transgranular mode of penetration was found to have a crystallographic pattern. Orientation determinations in single crystal specimens revealed the habit plane of the corrosion product.

The other systems which were investigated were Zr-O-Li, Ta-O-Li, and V-O-Li. Additions of oxygen to zirconium and vanadium did not affect their corrosion resistance to lithium; although, when the contamination reached a high level, the oxygen was gettered by the lithium just as in the case of niobium. Tantalum was shown to be even more sensitive to oxygen contamination than niobium.

RELATION BETWEEN OXYGEN DISTRIBUTION AND CORROSION IN SOME REFRACTORY METAL-LITHIUM SYSTEMS

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INTRODUCTION

Lithium has long been under consideration for application as a heat-transfer fluid in nuclear reactors because of its attractive physical properties. However, its potential as a reactor coolant was not seriously considered until preliminary studies in the early 1950's indicated that niobium and other refractory metals were more resistant to dissolution and other types of liquid-metal corrosion than were conventional materials of reactor construction. In more recent tests to determine the compatibility of a niobium-lithium system under varying conditions of time, temperature, and lithium flow rate, random corrosion effects were observed. When it became apparent that weldments and similar areas subject to possible contamination with oxygen, nitrogen, or carbon were preferentially attacked, an investigation was undertaken to determine whether there was a relation between these impurities and corrosion.

SUMMARY OF PRELIMINARY WORK

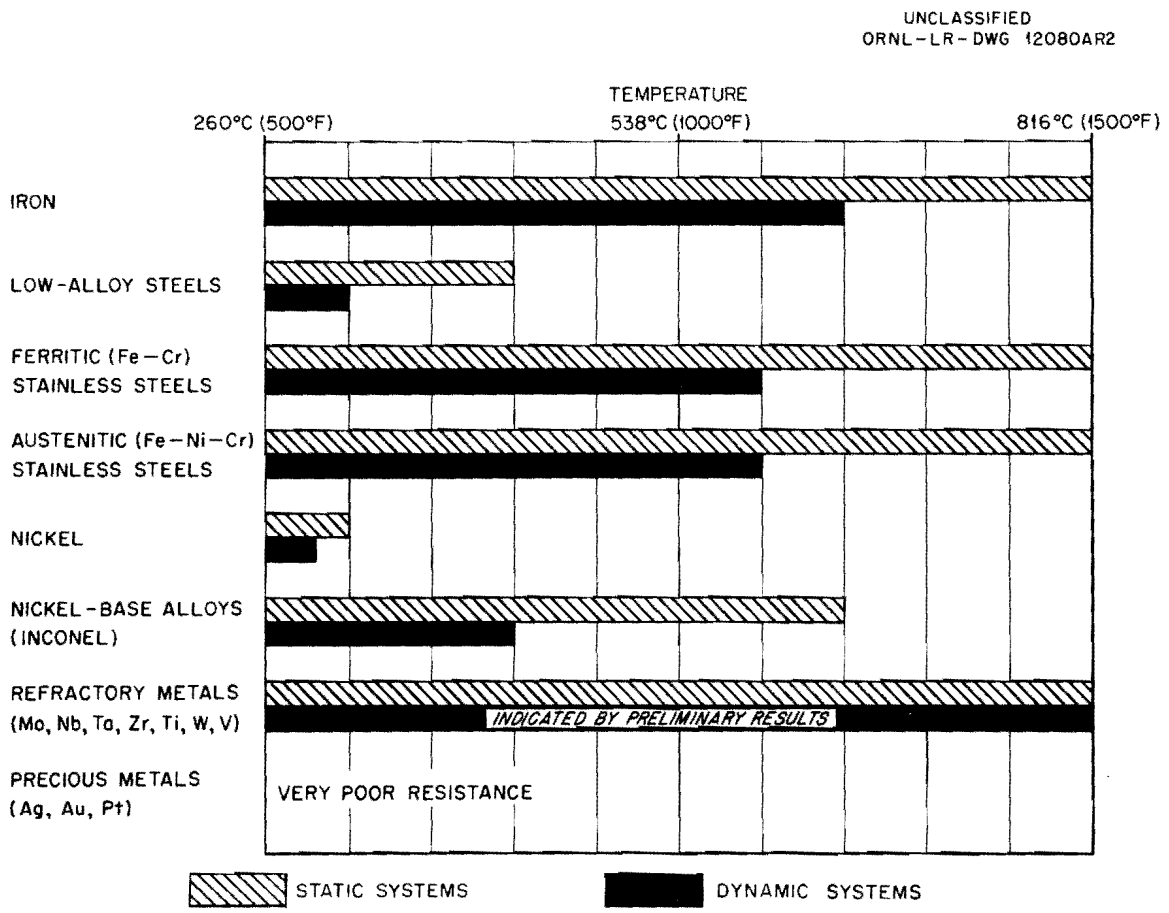
Static corrosion test data [1] and solubility studies [2] indicated the refractory metals to be more resistant to dissolution and other types of liquid-metal corrosion than were the more common metals and alloys. In addition, extensive lithium corrosion studies [3] were conducted in both static and dynamic test systems on a variety of materials, and only

the refractory metals seem appropriate for application in dynamic, nonisothermal systems [4] operating at temperatures in excess of 650°C (1200°F). Results of these exploratory studies are summarized in bar-graph form in Fig. 1.

Niobium was the refractory metal selected for further study because of its seemingly good resistance to attack by lithium at elevated temperature, plus the numerous properties which make it a potentially useful structural material for use in high-temperature reactor systems. Results of initial tests performed inside a vacuum chamber at 871°C (1600°F), however, indicated penetration of the niobium tube walls by the lithium in time periods of less than 23 hr. Metallographic examination of the test components revealed lithium penetration in weld regions and random attack in the base material. A typical example of this random type of attack is given in Fig. 2, which shows a translucent, gray, nonmetallic corrosion product.

The full extent of lithium penetration, especially in the nonwelded regions, was not discovered until cross sections from the loop were polished and allowed to stand in air for several days. Lithium in the penetrated regions reacted with moisture in the air to form the stained regions shown in Fig. 3.

The corrosion observed metallographically was verified by chemical analysis to be associated with lithium penetration. Analysis of turnings machined from a section of a thermal-convection loop revealed a lithium gradient (Table I) in the tube wall that was in agreement with the metallographic observations.



BARS INDICATE APPROXIMATE TEMPERATURES BELOW WHICH A SYSTEM MIGHT BE OPERATED FOR 1000 HOURS WITH LESS THAN 0.005 in. OF ATTACK OR CONTAINER SURFACE REMOVAL.

Fig. 1. Corrosion Resistance of Various Metals and Alloys to Lithium.

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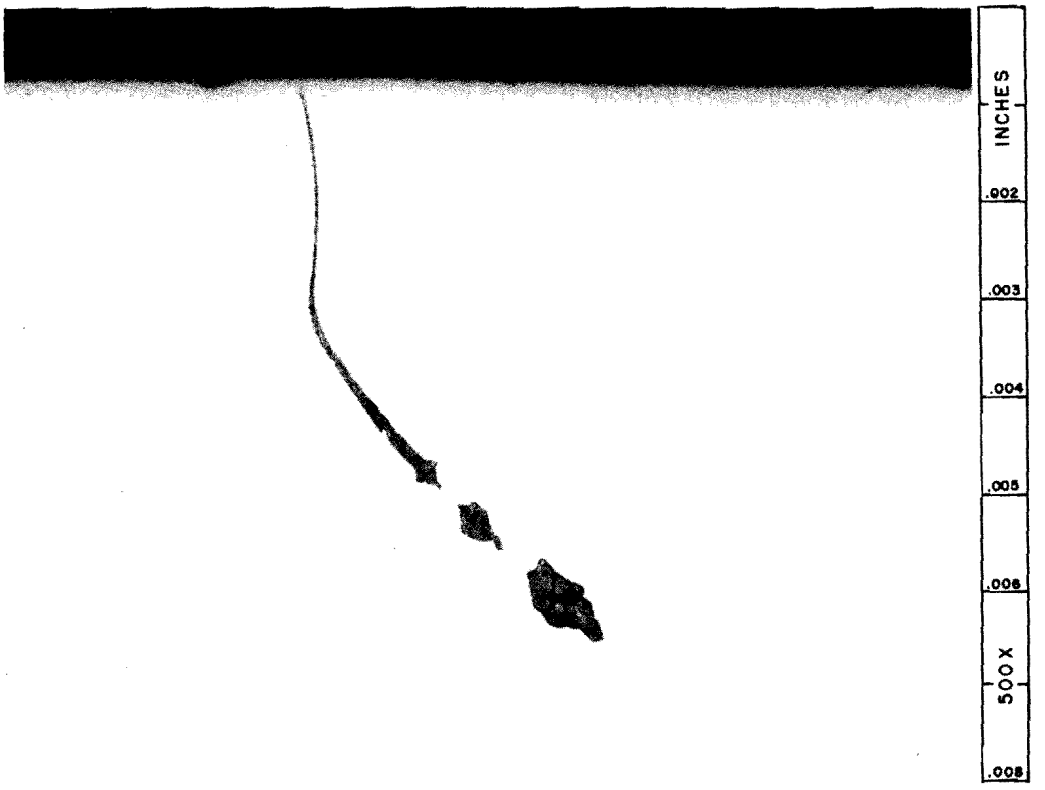

Fig. 2. Niobium Tube Wall Following Exposure to Lithium at 871°C (1600°F) for 36 hr. As-polished.

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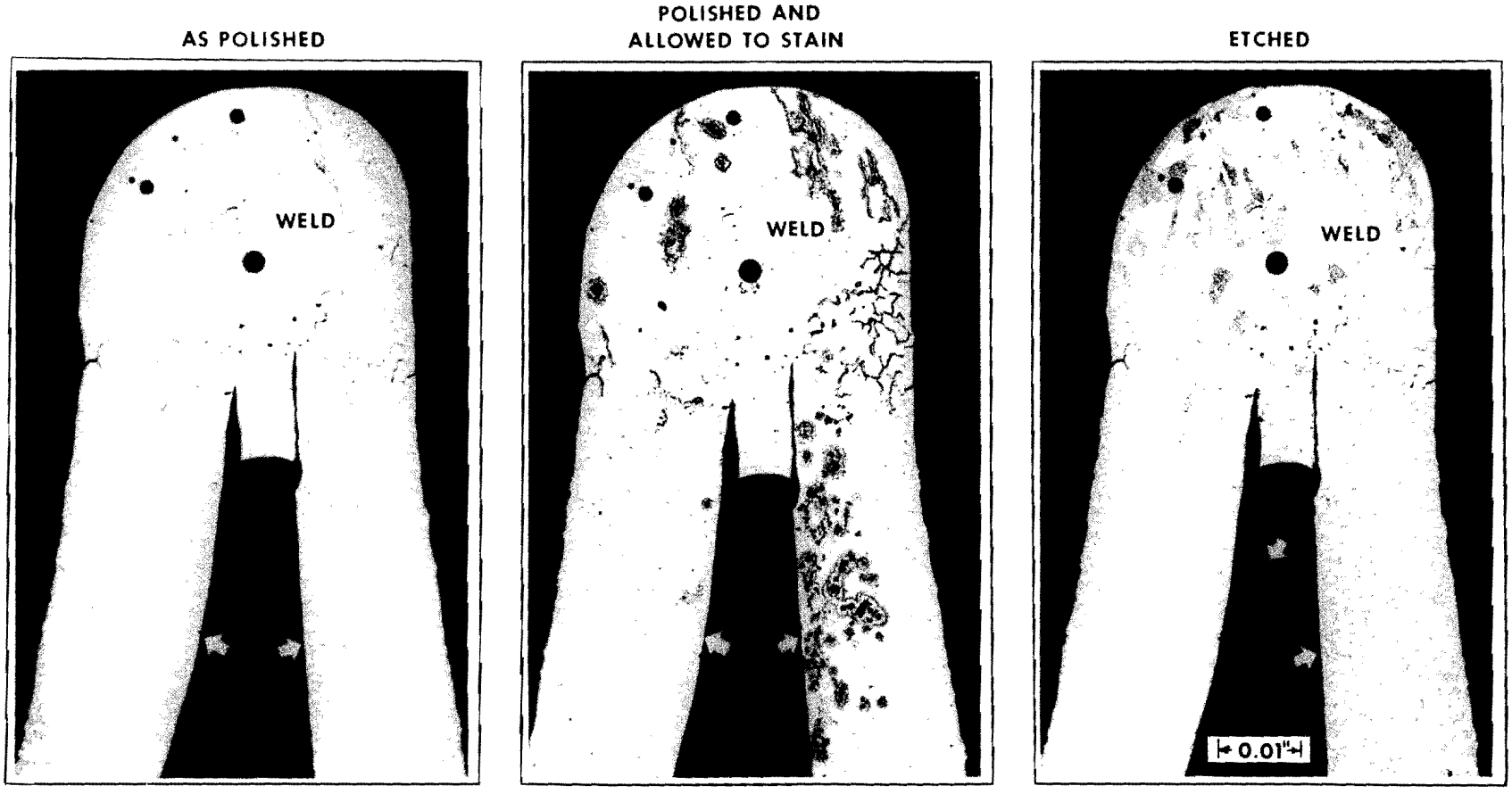

Fig. 3. Tube Wall and Weld Region of Niobium Loop in Which Lithium Was Circulated at 871°C (1600°F) for 23 hr. Arrow indicates the surfaces exposed to lithium.

Table I. Lithium Concentration as a Function of Distance from Outside of Niobium Tube Wall

Time: 23 hr
Hot Leg: 1600°F (871°C)
Cold Leg: 1300°F (704°C)

Distance (mils) from Outside Diameter of Tube Wall	Lithium Concentration (ppm)	
	Hot Leg[a]	Cold Leg[b]
0[c]–4	135	< 1
4–8	230	< 1
8–12	450	< 1
12–16	450	< 1
16–20[d]	470	10

[a]Depth of corrosion was 20 mils in weld and 10 mils in base material.

[b]No corrosion observed.

[c]Outside surface.

[d]Inside surface.

The results from this preliminary work led to initiation of a detailed investigation of the cause of the observed corrosion phenomenon. The major portion of the current investigation deals with niobium and some cursory work reported on tantalum, vanadium, and zirconium.

CURRENT RESEARCH

Experimental Procedure

The refractory metals used in these studies were generally obtained from high-purity electron-beam-melted stock. A typical chemical analysis of a heat of niobium is given below:

Element	Concentration (ppm)
Fe	< 100
Si	< 100
Ti	< 150
Ta	760
Zr	< 1000
B	< 1
H	< 1
C	50
N	60
O	160

Standard subsize sheet-type tensile specimens with a 6:1 ratio of gage width to thickness were used to evaluate the effect of corrosion on mechanical properties.

Lithium specified by the manufacturer (Maywood Chemical Works) to contain 0.2 wt % maximum metallic impurities was used in the study. Purification procedures as described in the literature [5] were used to reduce the oxygen concentration from approximately 2000 ppm to 100 ppm

and the nitrogen content from approximately 3000 ppm to less than 20 ppm.

Individual additions of oxygen, nitrogen, and carbon were made to the solid metal in order to evaluate the effect of these interstitial impurities on the corrosion process. Oxygen and nitrogen, in gaseous form, were added at 1000°C and at a pressure of 1×10^{-4} torr. Each specimen was homogenized for 2 hr at 1300°C and at 5×10^{-6} torr air pressure to ensure random distribution of the element. The amount of each addition was measured by changes in weight as well as chemical analysis.

The method for adding carbon to niobium involved electron-beam melting niobium and various amounts of niobium carbide. The alloys were subsequently cold-rolled to sheet and annealed at 1400°C for 2 hr.

Corrosion testing was performed in an assembly maintained in a stationary position at a controlled constant temperature. The test system, shown schematically in Fig. 4, consisted of refractory metal specimens in a capsule of the same refractory metal. The outer capsule was used to protect the refractory metal from oxidation during the test.

The methods used to determine the extent and nature of corrosion included most of the metallurgical and chemical analytical techniques currently available, such as weight change, chemical analysis, x-ray and electron transmission analysis, mechanical-property change, hardness measurement, and metallography. Weight change and chemical analysis data were not useful in determining the amount of attack by lithium but did give information on the redistribution of interstitial elements in the system. The hardness readings reported are the average of three or

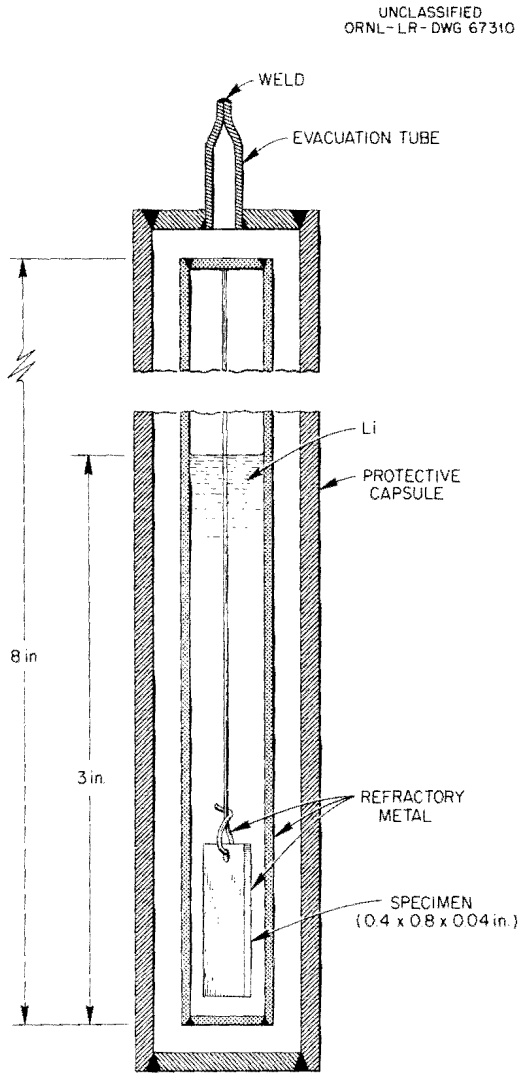

Fig. 4. Typical Refractory Metal–Lithium Corrosion Test System.

more determinations for each point plotted. Careful metallographic techniques were required because of the sensitivity of the corrosion product to polishing and etching. The method for polishing the refractory metal specimens in this investigation was that reported by Gray and Long [6].

Results and Discussion

General Impurity Effects on Corrosion of Niobium

It was previously pointed out that although the corrosion of niobium by lithium appeared to be a random occurrence it did occur more often in welds and in other areas subject to possible contamination by elements such as oxygen, nitrogen, and carbon. Therefore, to determine whether or not any relation existed between the presence of these interstitial elements and the corrosion phenomenon observed, single additions of oxygen, nitrogen, or carbon were made to relatively pure starting material. These specimens were then exposed to lithium for 100 hr at 816°C (1500°F).

The presence of nitrogen or carbon up to levels of approximately 0.1 wt % had no effect on the corrosion resistance of niobium to lithium. Therefore it was concluded that although both elements, and especially nitrogen, have significant effects on the mechanical properties of niobium they do not influence its corrosion resistance to lithium.

The exposure to lithium of a niobium weld specimen containing approximately 0.5 wt % O resulted in complete intercrystalline penetration (Fig. 5). More quantitative data on the effect of oxygen are given in Fig. 6. The results clearly show that the degree of attack of niobium by lithium increased as a function of the oxygen present in

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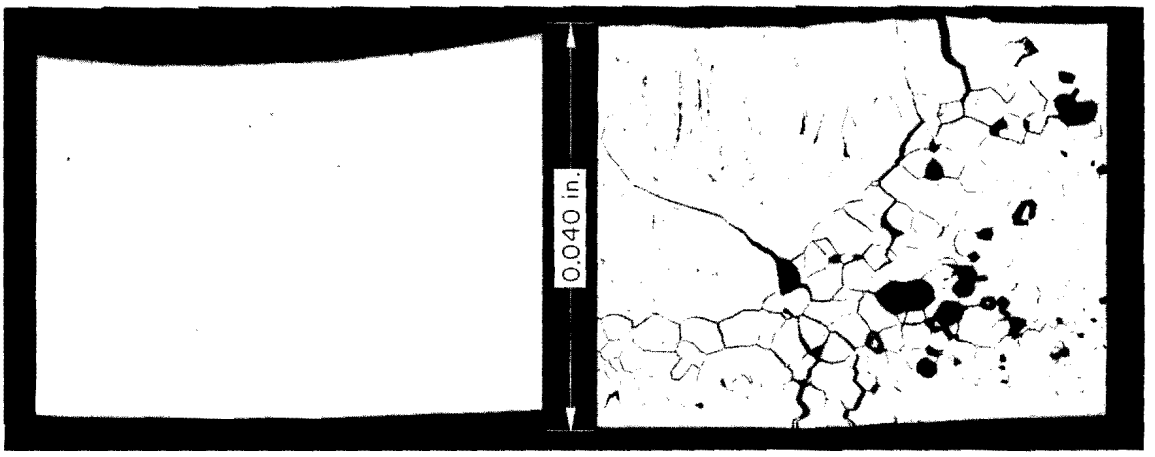

(AS POLISHED-75X) (AS POLISHED-75X)

(*a*) BEFORE EXPOSURE TO LITHIUM (*b*) AFTER EXPOSURE TO LITHIUM

Fig. 5. Weld Zones of Niobium Specimens To Which 0.5 wt % O Was Added Prior to Inert-Gas Tungsten-Arc Welding. Corrosion test conditions: 816°C (1500°F), 100 hr.

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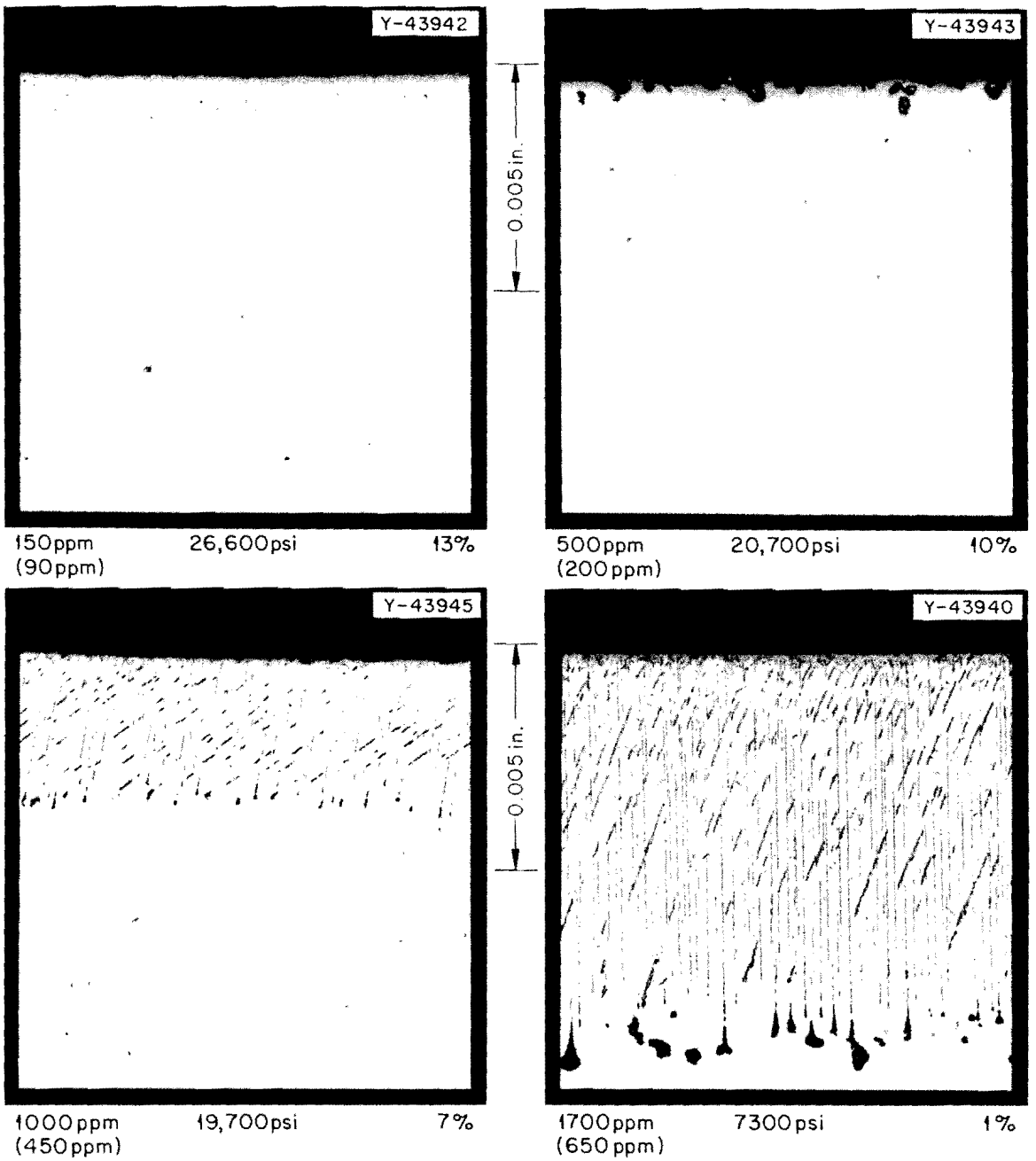

Fig. 6. Effect of Initial Oxygen Concentration, 150–1700 ppm, in Niobium on the Depth of Attack by Lithium. Test conditions: 816°C (1500°F) for 100 hr. Following test, the room-temperature tensile strength varied from 26,600 to 7300 psi, the elongation in 2-in. gage length varied from 13 to 1%, and the oxygen concentration varied from 90 to 650 ppm. Etchant: $HF-HNO_3-H_2SO_4-H_2O$.

niobium prior to test and occurred at oxygen concentrations less than the reported solubility limit of oxygen in niobium at the test temperature [7].

A second observation was that in addition to the intercrystalline type of attack cited previously an oriented transcrystalline mode of attack occurred. The data in Fig. 6, which indicate a general decline of both tensile strength and ductility with increased depth of corrosion, are presented in more detail in Fig. 7, where these properties are plotted as a function of initial oxygen concentration. The loss of strength and ductility in the corrosion test specimens as compared with the control specimens (heated in argon) is attributed to the brittle nature of the corrosion product. The hardness data shown in Fig. 8 as well as the oxygen analyses given in Fig. 6 indicate that oxygen was gettered from the niobium by the lithium. However, the area in the vicinity of the corrosion product "front" shows a peak in the hardness. This effect is magnified by the microhardness data shown in Fig. 9, in which the hardness data are plotted on a photomicrograph of a cross section of the niobium specimen referred to in Fig. 8. The presence of such a brittle phase within grains or along grain boundaries could account for the observed deterioration in mechanical properties.

The small size of the corrosion-product particles and the inability to recover sufficient amounts for quantitative analysis prevented positive identification. When the particles were large, they could not be retained during metallographic preparation. An acid mixture (HF-HNO_3-H_2SO_4-H_2O) was used to etch a corroded niobium specimen which had been distilled after test to remove any free lithium. The corrosion product was soluble

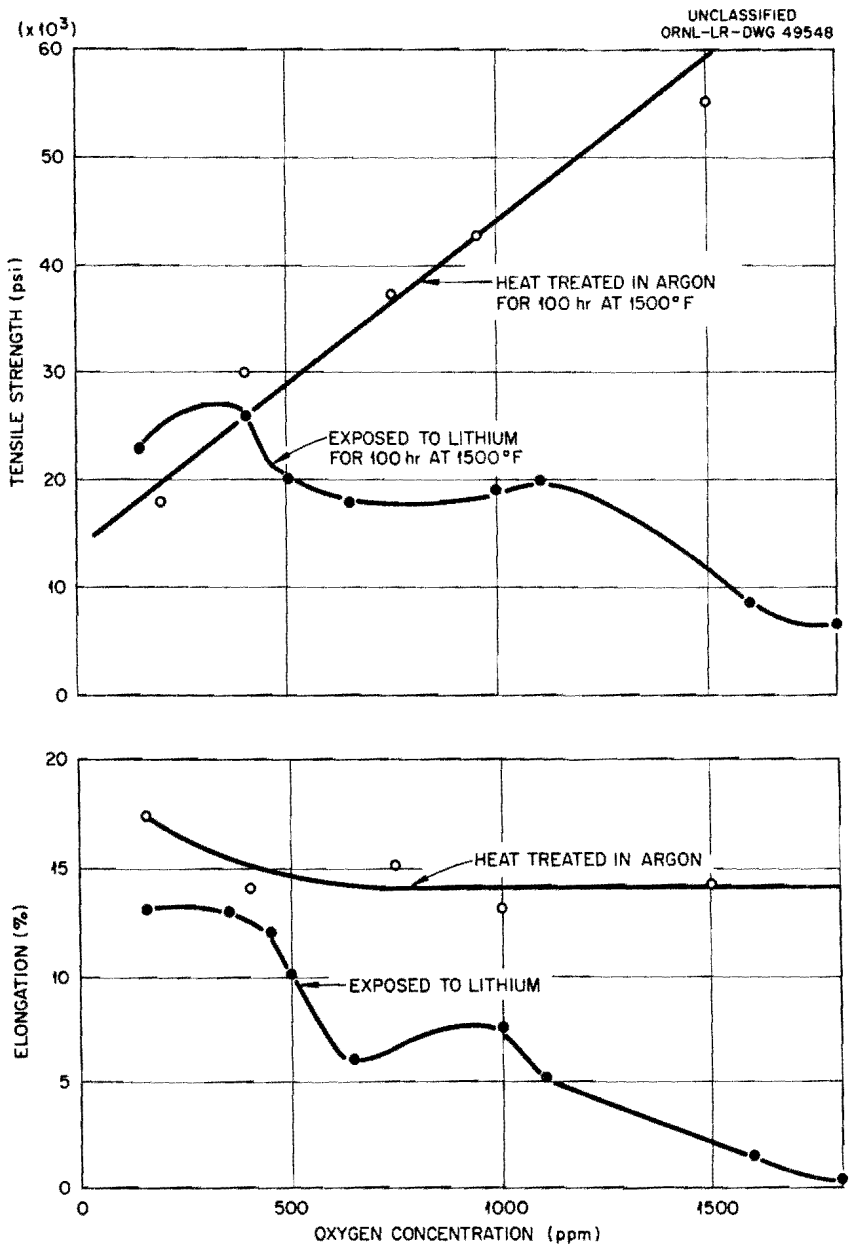

Fig. 7. Effect of Initial Oxygen Concentration on the Mechanical Properties of Niobium Following Heat Treatment in Argon and Exposure to Lithium at 816°C (1500°F). Specimen thickness, 0.040 in.

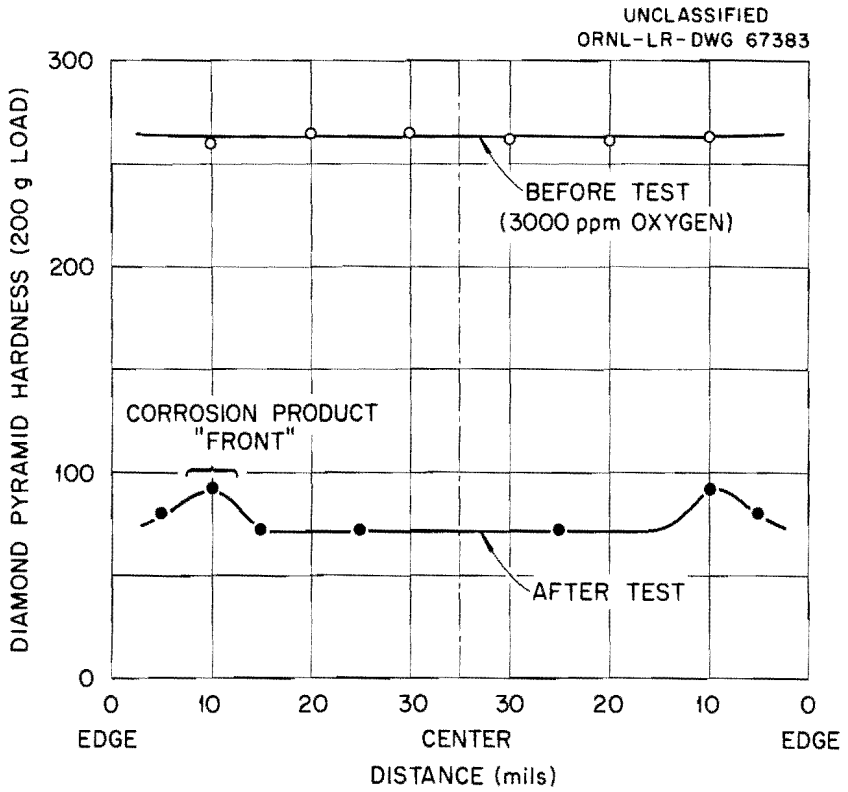

Fig. 8. Hardness Profile Across Niobium Specimen Before and After Exposure to Lithium for 100 hr at 1500°F (816°C).

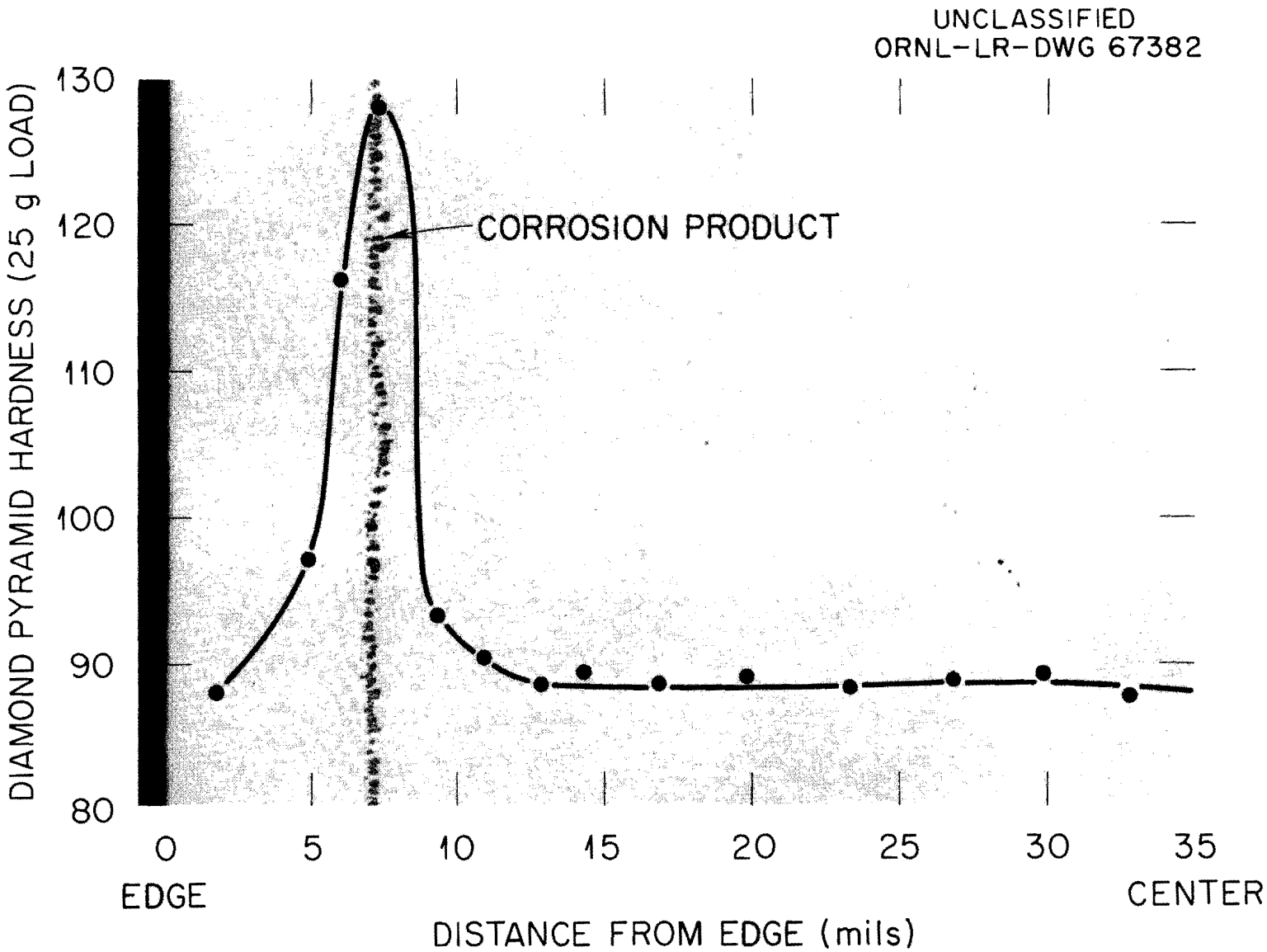

Fig. 9. Microhardness Profile Across Niobium Specimen After Exposure to Lithium for 100 hr at 1500°F (816°C). As-polished.

in this etchant, and a chemical analysis of the solution indicated that lithium was a component of the corrosion phase. Chemical analyses of niobium specimens after test always showed that the oxygen concentration had decreased. However, when the area containing the corrosion product was removed from these specimens by machining, electropolishing, or other suitable techniques, a further decrease in oxygen concentration was noted. This indicated that oxygen was also a component of the corrosion product. Although sufficient amount of this phase for quantitative chemical analysis could not be recovered, an x-ray pattern was obtained. The data from this pattern were indexed as a rhombohedral unit cell with hexagonal parameters a $\cong$ 7.49 A and c $\cong$ 7.97 A. Compounds of various niobates of lithium, such as $LiNbO_3$, Li_3NbO_4, and Li_2NbO_3, were synthesized according to directions outlined in the literature [8-10], and x-ray patterns obtained. Comparison of these patterns and those previously recorded for Li_2O and Li_2O_2 with the unknown pattern did not show any close similarity.

From examination of polished surfaces as well as polished cross sections the geometry of the transcrystalline type of corrosion product was established as that of a thin platelet. The depth of transcrystalline penetration in polycrystalline specimens was a function of grain orientation, as shown in Fig. 10, and orientation studies with niobium single crystals have shown that the habit plane of these platelets is {110}.

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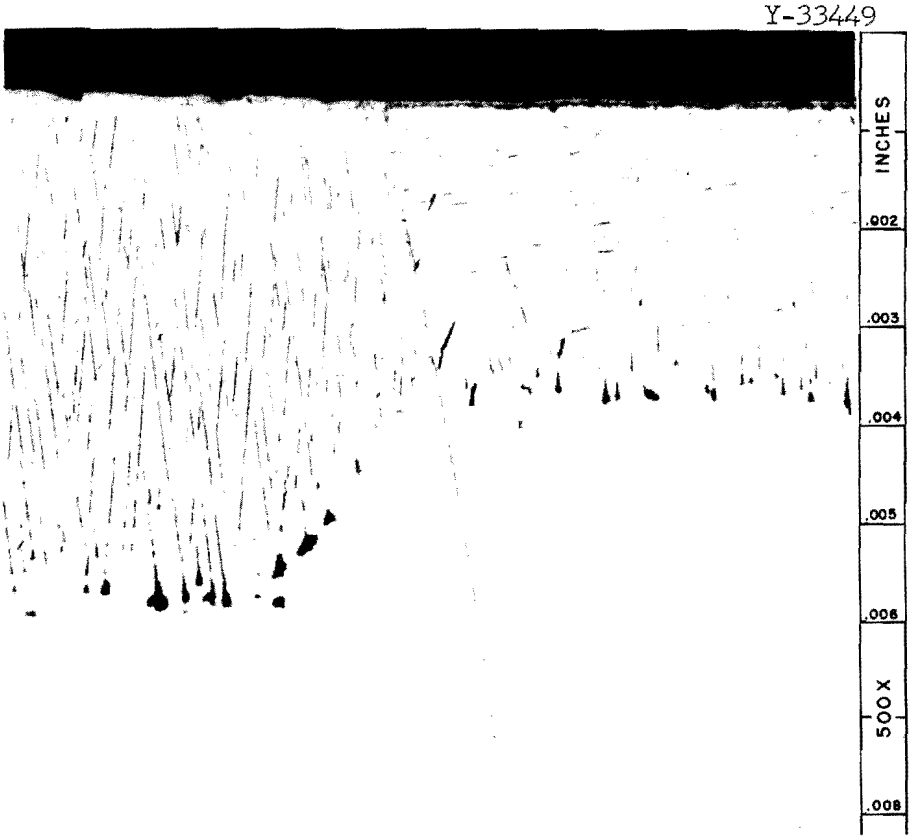

Fig. 10. Effect of Niobium Grain Orientation on the Depth of Lithium Penetration. Test conditions: 816°C (1500°F), 100 hr. Etchant: $HF-HNO_3-H_2SO_4-H_2O$.

Effect of Time on Oxygen-Contaminated Niobium

Since oxygen was identified as the impurity having a significant effect on corrosion, further studies were conducted in order to understand the mechanism. In a discussion of the effect of time, the distinction between lithium attack and oxygen redistribution must be emphasized.

The rate of attack by lithium was found to be rapid in both single-crystal and polycrystalline specimens of niobium. Although the rate may be influenced by temperature and oxygen concentration in single crystals, it appears to be relatively insensitive to such variables in polycrystals. Maximum penetration of polycrystalline niobium was observed in times as short as 1 hr and at temperatures as low as 260°C (500°F). Only single crystals were exposed for times shorter than 1 hr, and some time effect was noted, especially for relatively low initial oxygen concentrations (< 0.15 wt %) in the niobium.

Specimens of niobium were exposed for periods varying from 1 to 500 hr. Figure 11 compares the results of specimens exposed for periods of 1 and 250 hr. Although attack was completed in 1 hr, chemical analyses indicated that the process of oxygen transfer from the niobium to the lithium had continued up to approximately 100 hr. No further changes in oxygen concentration were noted after 250 and 500 hr. These data were confirmed by the hardness plots shown in Fig. 12. Comparison of the hardness profiles across niobium specimens as a function of exposure time shows that there is a sharp gradient in specimens exposed for less than 100 hr and that after 100 hr the gradient is almost completely eliminated

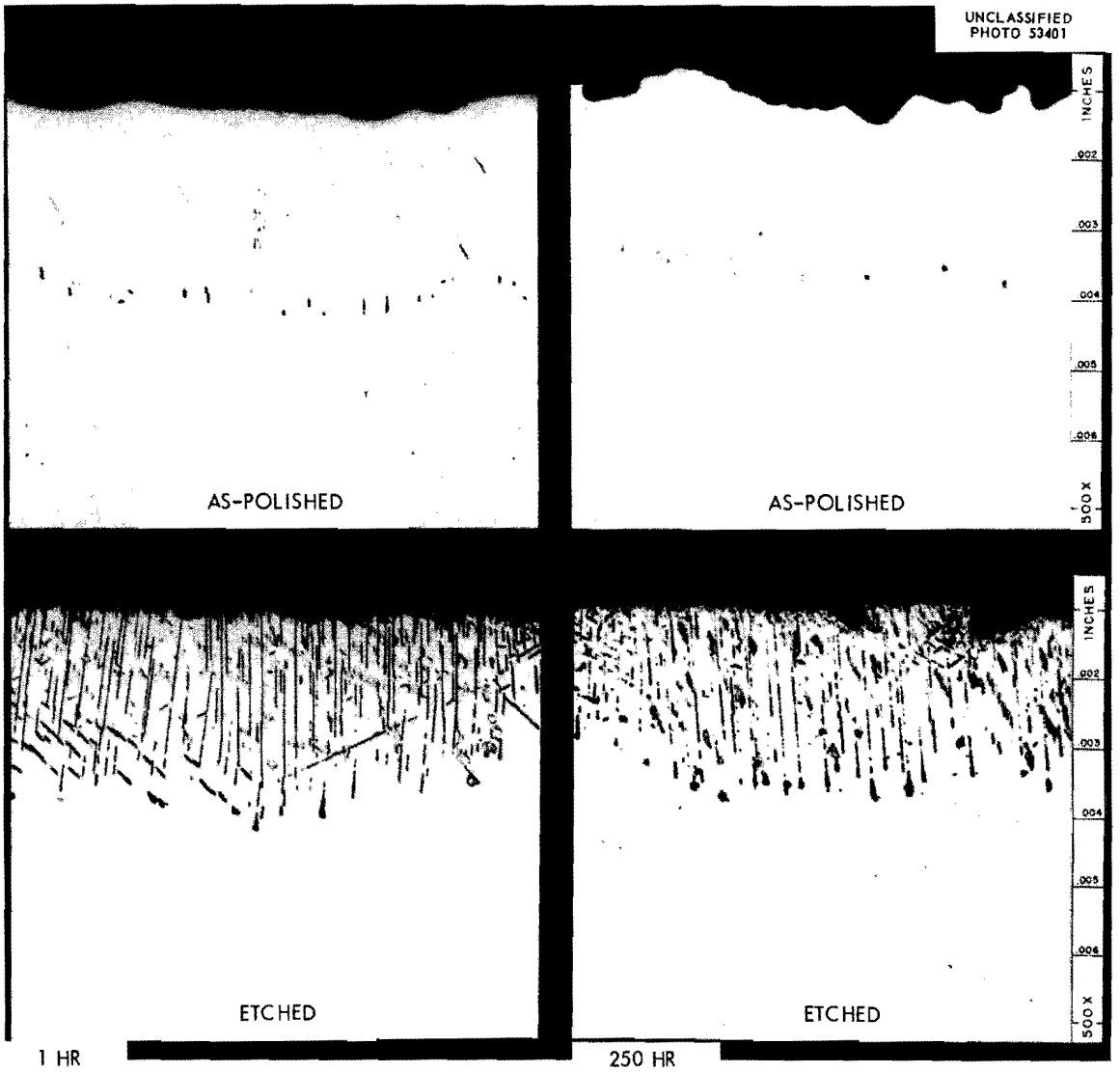

Fig. 11. Effect of Time on the Depth of Attack of Niobium by Lithium at 816°C (1500°F). Initial oxygen concentration of niobium, 0.1 wt %. Etchant: $HF-HNO_3-H_2SO_4-H_2O$.

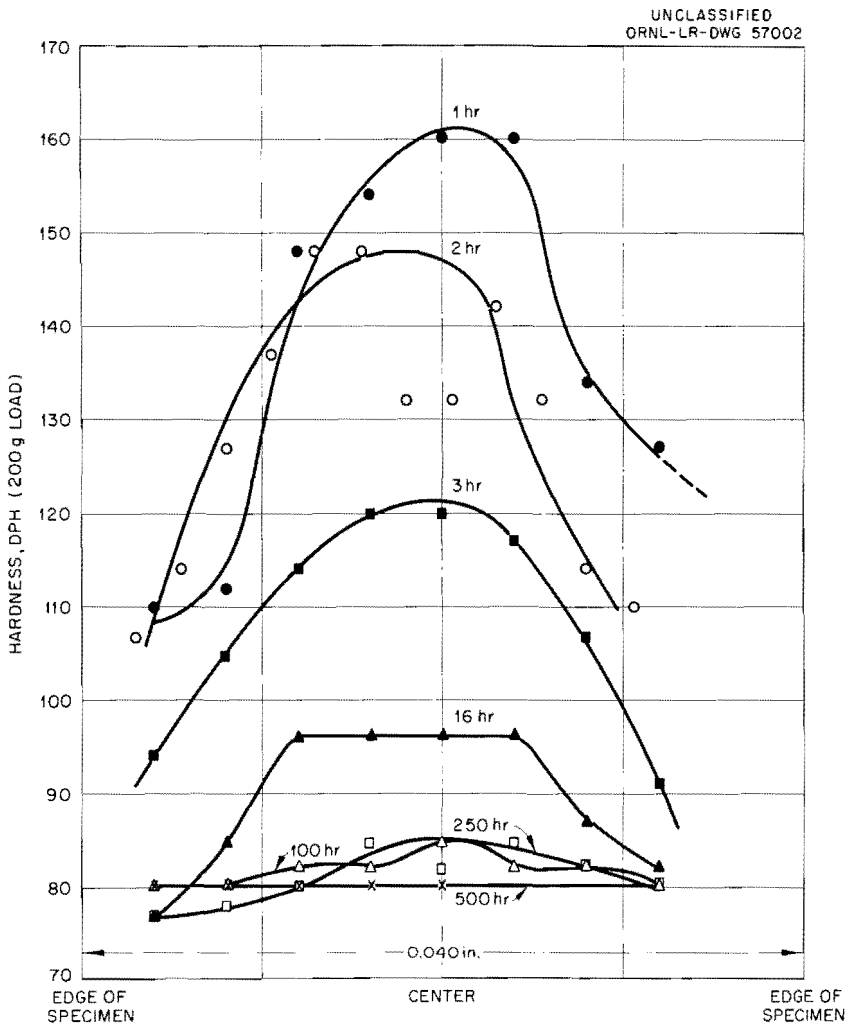

Fig. 12. Hardness Profile Across Niobium Specimens Following Exposure to Lithium for Various Times at 816°C (1500°F).

and the center hardness is constant within a diamond pyramid hardness range of 80 to 85.

Effect of Temperature on Oxygen-Contaminated Niobium

The effect of temperature on the depth of corrosion was found to be a complicated function of oxygen concentration and test procedure. Also, because in single crystals the mode of penetration is limited to the transcrystalline process, a distinction must be made between effects of temperature on single crystals and on polycrystalline niobium.

In the early corrosion tests the container holding the niobium test sample was packed with solid lithium and then put into a furnace which was preheated to the test temperature. Since the niobium was actually in contact with liquid lithium from its melting point on up to the test temperature, some corrosion occurred prior to the test temperature being reached. In later tests the niobium and lithium were separated until the test temperature was reached, and it was found that the rate of attack was relatively insensitive to temperature but that the maximum depth of penetration was a function of the temperature as well as the initial oxygen concentration.

The mode of attack of polycrystalline niobium changed from primarily transcrystalline to intercrystalline in the range 600 to 800°C (1112–1472°F). Because of this shift in mode of attack oxygen-contaminated polycrystalline niobium became increasingly more sensitive to lithium attack as a function of increasing temperature. Above 1000°C (1832°F) there was very rapid penetration of specimens even when they were exposed only in the vapor region above the liquid lithium. This temperature effect, however, was not true of single crystals. As

illustrated by the photomicrographs in Fig. 13, greater penetration occurred at 371 and 593°C (700 and 1100°F) than at 816 and 1038°C (1500 and 1900°F). A similar effect was also observed in single crystals containing an initial oxygen concentration of 2600 ppm and tested under otherwise similar conditions. This apparent anomaly may have been caused by the increased diffusion rate of oxygen in niobium at the higher temperatures. If the rate of transfer of oxygen from niobium to lithium was faster than the rate of transcrystalline lithium penetration at the elevated temperatures, the attack at high temperature would have been reduced due to the very rapid lowering of the oxygen concentration of the niobium.

A second possible explanation for this anomaly is that the critical oxygen level required for lithium penetration may have increased with increasing test temperature. This increase in critical level could have resulted from a more homogeneous distribution of oxygen. If it is assumed that some submicroscopic inhomogeneities in oxygen distribution must exist prior to lithium attack, then a possible mechanism which could lead to such inhomogeneities is the concentration of oxygen atoms at dislocations. Such concentrations have been associated with etch pit formation [11-13] in niobium and in tantalum, and the number of oxygen-dislocation interactions has been shown to decrease with increasing temperature [14].

Effects of Oxygen on Corrosion of Tantalum, Vanadium, and Zirconium

Results of tests with tantalum at 816°C (1500°F) indicated it to be considerably more sensitive than niobium to lithium penetration as a

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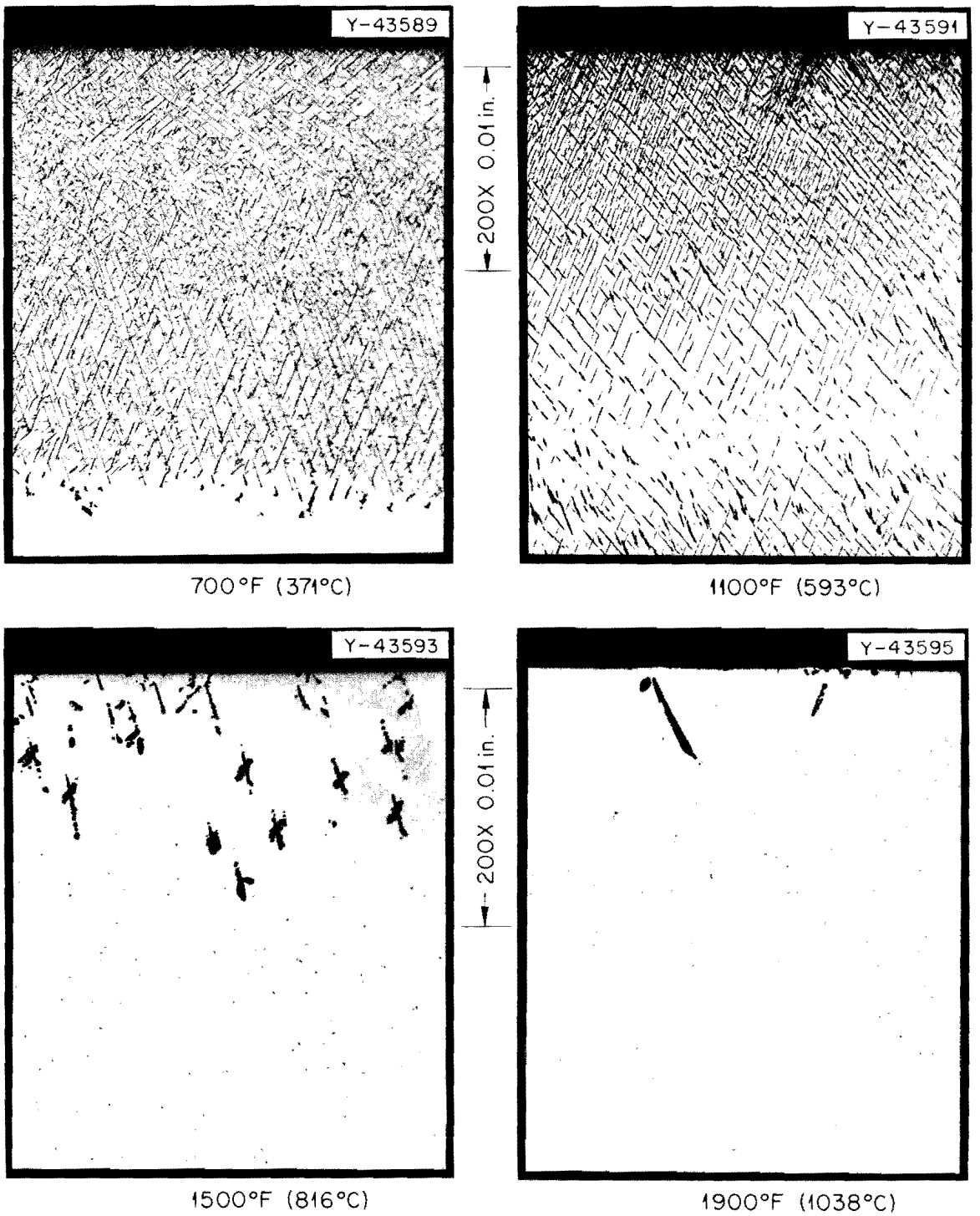

Fig. 13. Effect of Temperature on the Depth of Attack by Lithium of an Oxygen-Contaminated (1500 ppm) Niobium Single Crystal. Test duration, 100 hr. Etchant: $HF-HNO_3-H_2SO_4-H_2O$.

function of oxygen concentration. Figure 14 shows that complete penetration of 0.040-in. specimens occurred at 816°C (1500°F) with an initial oxygen concentration of 0.03 wt %. A transfer of oxygen from the tantalum to the lithium was indicated from chemical analyses.

The addition of 0.2 wt % O to vanadium and of 0.4 wt % O to zirconium did not result in any penetration by lithium when exposed for 100 hr at 816°C (1500°F), but a reduction in the oxygen concentration of these metals was noted.

Distribution of Oxygen in Refractory Metal–Lithium Systems

The presence of small quantities of oxygen in niobium and tantalum was shown to decrease the resistance of these metals to lithium penetration. In addition a transfer of oxygen between the solid and liquid metal was observed to have taken place in all the systems whether lithium penetration occurred or not. It was found that lithium gettered oxygen more thoroughly from vanadium and to a lesser extent from tantalum, niobium, and zirconium. This indicated that there was at least a qualitative relation between the differences in the standard free energies of formation of the solid-metal oxides and lithium oxide and the amount of oxygen transferred. However, in order to calculate the quantitative amount of oxygen interchange, further information is required. Since all the systems investigated represent two relatively immiscible solvents (refractory metal and lithium) in contact, both containing the same substance in solution (oxygen), the distribution of oxygen between the refractory metal and lithium should follow Nernst's law: $a_1/a_2 = K$, where a_1 is the activity of oxygen in the lithium phase, a_2 is the activity of oxygen in the solid-metal phase, and K is a distribution coefficient

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a) 0.008% O_2 b) 0.03% O_2 c) 0.07% O_2 d) 0.09% O_2

Fig. 14. Effect of Initial Oxygen Concentration of Tantalum on the Depth of Attack by Lithium After 100 hr at 816°C (1500°F). Etchant: $HF-HNO_3-H_2SO_4-H_2O$.

and constant at a given temperature. If Henry's law is applicable, and this would appear to be reasonable since the solutions are dilute, then the activity is essentially equal to the concentration and the above equation reduces to the more common form $c_1/c_2 = K'$, where c_1 and c_2 are the oxygen concentrations in the liquid and solid metal, respectively. The test data showed that at 816°C (1500°F) lithium containing approximately 0.01 wt % O gettered oxygen from vanadium, tantalum, and niobium even when their oxygen concentrations were as low as 0.01 wt %. However, lithium gettered oxygen from zirconium only when it contained greater than 0.1 wt % O. Although chemical analyses for oxygen in the various refractory metals were easily obtained, the procedures for accurately determining oxygen in lithium are difficult and the results often unreliable. Therefore distribution coefficient values are not reported.

SUMMARY

The pure metals niobium, tantalum, vanadium, and zirconium exhibit excellent resistance to attack by lithium at temperatures even in excess of 800°C (1472°F). However, the presence of small quantities of oxygen in either niobium or tantalum can lead to a very rapid penetration of these metals by lithium over a wide range of temperatures. Vanadium and zirconium, on the other hand, show no attack when contaminated with oxygen to levels of 2000 and 4000 ppm, respectively.

Chemical interaction between molten lithium and oxygen-contaminated niobium results in the formation of a corrosion product most often found in either the grain boundaries or along {110} planes. The product was

found to contain lithium and oxygen, but no further identification was made. The major variables affecting the extent of lithium penetration in niobium are oxygen concentration and temperature. Time was found to be an insensitive variable in the sense that the maximum depth of corrosion generally occurred in approximately 1 hr.

The depth and mode of attack in single-crystal and polycrystalline specimens of niobium differed as a function of temperature. It is possible that these results are related to differences in the diffusion rate of oxygen in these specimens or to possible interactions of oxygen with dislocations as a function of temperature.

In addition to lithium penetration, a transfer of oxygen was observed in all the refractory metal—lithium systems investigated. Oxygen transfer appears to occur according to Nernst's law of distribution for a solute between two immiscible solvents. Although no distribution coefficient was calculated, the qualitative extent of gettering observed was in agreement with that predicted by the standard free energies of formation for the various oxides of the solid metals and lithium oxide.

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BIBLIOGRAPHY

1. A. de S. Brasunas, Interim Report on Static Liquid-Metal Corrosion, ORNL-1647, pp 32–44 (May 11, 1954).

2. U. F. Bychkov, A. N. Rozanov, and V. B. Yakobleva, "The Solubility of Metals in Lithium," Atomnaya Energiya 7(6), 531–536 (1959).

3. E. E. Hoffman, Corrosion of Materials by Lithium at Elevated Temperatures, ORNL-2924 (Oct. 3, 1960).

4. E. E. Hoffman, op. cit., p 124.

5. E. E. Hoffman, "Solubility of Nitrogen and Oxygen in Lithium and Methods of Lithium Purification," pp 195–206 in Symposium on Newer Metals, Special Technical Publication No. 272, American Society for Testing Materials, Philadelphia, Pa., 1959.

6. R. J. Gray and E. L. Long, Jr., "Polishing by Vibration," Metal Progress 74(4), 145–48 (1958).

7. A. U. Seybolt, "Solid Solubility of Oxygen in Niobium," Journal of Metals 6, 774–76 (1954).

8. A. V. Lapitiskii, "Anhydrous Lithium Metaniobate," Journal of General Chemistry of the U.S.S.R. (English Translation) 22, 41–43 (1952).

9. A. V. Lapitiskii and Yu. P. Simanov, "Lithium Salts of Orthoniobic and Orthotantali Acid," Vestnik Moskovskogo Universiteta Seriya Fiziko-Matematecheskikh i Estestvennykh Nauk No. 1 9, 69–72 (1954).

10. E. I. Krylov and A. A. Sharnin, "Synthesis and Properties of Niobium Bronzes," Journal of General Chemistry of the U.S.S.R. (English Translation) 25, 1637–40 (1955).

11. L. C. Lovell, F. L. Vogel, and J. H. Wernick, "Etching Reagents for Dislocations in Metal Crystals," Metal Progress 75, 96 (1959).

12. A. B. Nickael and F. J. Huegel, "On Dislocation Configurations in Rolled Columbium," Transactions of the Metallurgical Society of AIME 212, 818-20 (1958).

13. R. Bakish, "Dislocation Configurations and Densities in Tantalum Crystals," Acta Metallurgica 6, 120-122 (1958).

14. D. Gupta and S. Weinig, "The Dislocation Oxygen Interaction in Alpha Titanium and Its Effect on the Ductile-to-Brittle Transition," Transactions of the Metallurgical Society of AIME 215, 216 (1959).

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