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The Reduction of Al_2O_3 in Niobium-Lithium Systems at 1000°C

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THE REDUCTION OF ALUMINUM OXIDE IN NIOBIUM-LITHIUM SYSTEMS AT 1000°C

J. E. Selle and J. H. DeVan

ABSTRACT

Various grades of aluminum oxide (Al_2O_3) were sealed inside capsules of niobium and niobium-1% zirconium alloy, which were then exposed to liquid lithium for 3000 hr at 1000°C. Similar unsealed capsules were exposed to a high vacuum. Reduction of the Al_2O_3 occurred in the lithium-treated capsules, but no reaction occurred in the vacuum-treated capsules. Metallography and electron-microprobe analysis showed that reaction products in the form of compounds of niobium, aluminum, and zirconium were formed. Lithium acted as a sink for oxygen.

INTRODUCTION

In a system as complex as a Controlled Thermonuclear Reactor (CTR) very subtle materials problems can arise. One such problem can arise from the use of certain ceramic insulators in contact with a refractory metal whose opposite side is in contact with liquid lithium metal. This condition can occur in the θ -pinch reactor where the first wall must be electrically insulated against the plasma-backed electromotive force (EMF) which develops during implosion heating. If this first wall is cooled with liquid lithium, the condition described above exists. This condition is also present in any system in which lithium is circulated by means of an electromagnetic pump system that will require a coating of an electric insulator on the outside of the lithium conduit. A niobium blanket structure is a good possibility for use in CTR construction.¹

One would not expect thermodynamic incompatibility between an insulator such as aluminum oxide (Al_2O_3) and niobium, or niobium-1% zirconium alloy (Nb-1% Zr alloy). Table 1 summarizes thermochemical calculations for the following general reaction:

Other than lithium, only zirconium shows potential for reducing alumina, but even this potential is marginal because zirconium would only be used as an alloying element as in Nb-1% Zr alloy.

However, it has been discovered that a potential exists for incompatibility between Nb-1% Zr alloy and Al_2O_3 under certain conditions.² In this experiment an Nb-1% Zr thermal convection loop containing lithium was operated in a vacuum for 3000 hr with a hot-leg temperature of 1200°C and a cold-leg temperature of 1070°C. Thermocouples with Al_2O_3 insulators were placed in thermocouple wells at strategic locations around the loop. A schematic diagram of the prevailing condition is shown in Fig. 1. Metallographic analysis of the thermocouple well at the colder section of the loop (about 1070°C) showed no evidence of reaction. However, analysis of the wells in the higher temperature regions showed a reaction layer between the Al_2O_3 insulator and the alloy (Fig. 2). Spectrographic and x-ray diffraction analyses

1. A. P. Fraas, *Comparative Study of the More Promising Combinations of Blanket Materials, Power Conversion Systems, and Tritium Recovery and Containment Systems for Fusion Reactors*, ERDA Report, ORNL-TM-4999, Oak Ridge National Laboratory, November 1975.

2. J. H. DeVan, unpublished data.

identified the layers as the intermetallic compounds NbAl_3 and Nb_2Al . A third layer was also present, but its composition could not be determined. About 1% silicon of unknown origin was also present, as indicated by spectrographic analysis.

These results have strong implications for the usefulness of certain material combinations in lithium-containing CTRs. Therefore, it was decided to investigate this problem under more controlled conditions.

Table 1. Thermodynamic Calculations for the reaction

Metal	1073K	1273K	1473K
	(K cal/g-atom oxygen)		
Niobium	+28,959	+28,115	+27,299
Zirconium	-43	-600	-1,308
Vanadium	+30,078	+34,410	+38,770
Titanium	+6,880	+6,012	+5,172
Lithium	-2,831	-1,399	+61

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Fig. 1. Schematic diagram of conditions prevailing in niobium-1% zirconium-lithium loop.

Fig. 2. Reaction layer between Al_2O_3 insulator and Nb-1% Zr alloy (2)250X.

TEST PROCEDURES

Sandwich-type specimens, shown in Fig. 3, were fabricated using 0.027-in.-thick niobium or 0.020-in.-thick Nb-1% Zr alloy sheets. Three different types of Al_2O_3 were used: sapphire, Lucalox, and 99.9% dense sintered Al_2O_3 . Both niobium and Nb-1% Zr alloy were used to fabricate the specimens. Oxygen and nitrogen analyses of the starting material are summarized in Table 2. After fabrication by electron-beam welding completely around the periphery, the samples to be run in lithium were annealed for 1 hr at 1200°C under a vacuum of 1×10^{-6} to 1×10^{-8} torr, checked for leaks, and weighed. The samples were then loaded into a Nb-1% Zr alloy container which was seal-welded closed and was loaded with 290 g of lithium. An overpressure of 10 psig of argon was introduced into the container to promote intimate contact between the ceramic wafer and the metal. This contact occurs as a result of both the overpressure and the vacuum produced inside the sample assembly by the electron-beam welding.

Specimens run in vacuum were not seal welded at the edge so that the ceramic-metal interface was also exposed to the vacuum environment. The vacuum was maintained at $1-5 \times 10^{-8}$ torr. These specimens were stacked with tungsten foil between them, and the stack was weighted to assure contact between the ceramic wafer and the metal. The tests were run for 3000 hr at 1000°C . This temperature was chosen as a reasonable duplication of probable CTR operating conditions.

In addition, metallography, microhardness measurements, electron-microprobe analyses, and chemical analyses were performed on each test specimen at locations at the ceramic-metal interface and away from the ceramic-metal interface.

METAL	INSULATOR	ENVIRONMENT	TEST CONDITIONS
Nb AND Nb-1% Zr	SAPPHIRE LUCALOX 99.9% SINTER- ED Al_2O_3	VACUUM AND LITHIUM	3000 hr AT 1000°C

Fig. 3. Compatibility tests of Al_2O_3 -Nb alloy compacts.

Table 2. Analysis of starting material

	Oxygen	Nitrogen
	(ppm)	
Niobium	108	54
Niobium 1% Zirconium	282	79

TEST RESULTS

Upon removal from the lithium pot, the lithium-treated specimens exhibited a surface discoloration not found on the vacuum-treated specimens. This discoloration was yellowish brown and was somewhat akin to the discoloration found on nitrated zirconium.

Chemical analyses of lithium samples taken before and after the test are summarized in Table 3. These data indicate a gain in oxygen and a loss of nitrogen by the lithium.

It was not possible to obtain electrical resistivity measurements of the ceramic wafers because of the problem of obtaining satisfactory electrical contact with the ceramic wafer. During cooling, the ceramic wafer separated from the metal sheet, thereby precluding an electrical contact utilizing the metal sheet.

Typical microhardness traverses for niobium and niobium-1% Zr alloy are shown in Figs. 4 and 5, respectively. There was no appreciable difference in the appearance of the microhardness traverses between the various grades of alumina.

Table 3. Lithium analyses before and after test

	Oxygen	Nitrogen
	(ppm)	
Before test	132	56,65
After test	249	23,14

Fig. 4. Typical microhardness traverses from niobium.

A summary of oxygen and nitrogen analyses and average microhardness values for each of the tests is given in Table 4. Each microhardness value given in the table is the average of 20–22 values obtained from the traverses. The data in Table 4 yields a number of observations concerning microhardness values and oxygen and nitrogen values.

Microhardness Values

In the niobium-lithium tests, the hardness values at the ceramic wafer are about the same as the values away from the ceramic wafer. However, in the vacuum tests the values were greater away from the ceramic wafer. For the Nb-1% Zr alloy tests, the values away from the ceramic wafer are considerably higher than those at the ceramic wafer.

Fig. 5. Typical microhardness traverses from niobium-1% zirconium alloy.

Table 4. Summary of oxygen and nitrogen analyses and microhardness values (DPH)

	Lithium tests	Vacuum tests
Niobium specimens		
At ceramic wafer	N 19 ppm O 47 ppm Hardness 61.6 DPH	N 100 ppm O 427 ppm Hardness 125.9 DPH
Away from ceramic wafer	N 14 ppm O 167 ppm Hardness 63.8 DPH	N 70 ppm O 457 ppm Hardness 134.5 DPH
Niobium-1% Zirconium alloy specimens		
At ceramic wafer	N 82 ppm O 1126 ppm Hardness 91.2 DPH	N 100 ppm O 267 ppm Hardness 91.4 DPH
Away from ceramic wafer	N 89 ppm O 51 ppm Hardness 103.1 DPH	N 96 ppm O 803 ppm Hardness 103.8 DPH

The results for the niobium tests show that the hardness values are much lower in lithium tests than in vacuum tests, while the results for the Nb-1% Zr alloy show that the values are the same in both tests.

Oxygen Analyses

In the niobium-lithium tests, the oxygen values away from the ceramic wafer are higher than values at the ceramic wafer. This is also true of the niobium-vacuum tests, although this difference is small.

For the Nb-1% Zr alloy, the oxygen values at the ceramic wafer are considerably higher in the lithium test; the reverse is true in the vacuum test.

Nitrogen Analyses

Lithium removes nitrogen from solution in the niobium, whereas the vacuum tests showed a slight increase in nitrogen.

No significant changes were found in the Nb-1% Zr alloy lithium tests. A slight increase is seen in the vacuum tests.

Photomicrographs of the inside edge of the niobium and the Nb-1% Zr alloy are given in Figs. 6–9. A reaction layer is seen on the inside edge of the niobium at the ceramic wafer and away from the ceramic wafer, although the reaction layer away from the ceramic wafer is much thinner. Two layers are seen on the inside edge of the Nb-1% Zr alloy, and one layer is seen away from the ceramic wafer.

Electron-microprobe data for Nb-1% Zr alloy are shown in Figs. 10 and 11 which show the inside edge at the ceramic wafer and away from the ceramic wafer, respectively. At the ceramic wafer, the outer layer contains zirconium and aluminum, while the inner layer contains niobium and aluminum. Away from the ceramic wafer only niobium and aluminum were found. The aluminum presumably was transported in the vapor phase.

No reaction layers were observed on any of the vacuum-treated niobium samples although a very thin reaction layer was found at the ceramic wafer on the Nb-1% Zr alloy samples. No reaction layers were found on the outside edges of the specimens.

Fig. 6. Photomicrograph of inside edge of niobium sample at the ceramic wafer (etched)500X.

Fig. 7. Photomicrograph of inside edge of niobium sample away from the ceramic wafer (unetched)500X.

Fig. 8. Photomicrograph of inside edge of niobium-1% zirconium sample at the ceramic wafer (etched)500X.

Fig. 9. Photomicrograph of inside edge of niobium-1% zirconium sample away from the ceramic wafer (etched)500X.

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Fig. 10. Electron-microprobe traces on Nb-1% Zr alloy.

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Fig. 11. Electron-microprobe traces on niobium sample.

DISCUSSION

This work shows, as did the earlier work, that under certain conditions niobium-aluminum intermetallic compounds can be formed from niobium and Al_2O_3 . This suggests that the following reactions are possible.

and

Thermodynamic data for niobium-aluminum compounds are not available; therefore, the validity of reaction (2) is unknown. However, data relating to reaction (3) in Figs. 12 and 13 included in these figures are ΔF° , the free energy of formation in the standard state of Li_2O , NbO , Al_2O_3 , and ZrO_2 ; and $\Delta \bar{F}^\circ$, the estimated partial molar free energy of oxygen at various oxygen concentrations in lithium, niobium, and zirconium.³ These latter values are calculated from the relationship:

$$\Delta \bar{F}^\circ = \Delta F^\circ + RT \ln a_m \quad (4)$$

3. D. L. Smith and K. Natesan, "Influence of Nonmetallic Impurity Elements on the Compatibility of Liquid Lithium with Potential CTR Containment Materials," *Nucl. Techn.*, **22** 392-404 (1974).

Fig. 12. Thermodynamic data for niobium-oxygen system.

Fig. 13. Thermodynamic data for zirconium-oxygen system.

where R is the gas constant, T is the absolute temperature, and a_m is the activity of oxygen in the metal, when

$$a_m = \frac{\text{concentration of oxygen in metal}}{\text{solubility of oxygen in metal}} \quad (5)$$

From these figures it is apparent that at 1273K (1000°C), Li_2O is more stable than solutions of oxygen in niobium for all concentrations greater than 0.056 ppm oxygen. As the oxygen content of the lithium decreases, $\Delta\bar{F}_{\text{Li}}^\circ$ becomes even more negative, as seen in Fig. 12, showing that reaction (3) can occur. Values for oxygen in zirconium in Fig. 13 show that a reaction analogous to reaction (2) can occur between oxygen in zirconium and lithium. Figure 14 shows values for the various nitrides and shows increasing stability in the order: Li_3N , Nb_2N , and ZrN .

Fig. 14. Thermodynamic data for nitrides.

The fact that no reaction layer is present on the niobium specimens tested in vacuum is significant. Fromm and Jehn⁴ have pointed out that in the oxygen-niobium system thermodynamic equilibrium is precluded in practical vacuum systems due to the extremely low oxygen equilibrium pressure and the relatively high partial pressure of oxygen in the vacuum. Thus, under vacuum conditions oxygen is not removed from niobium, but will be absorbed so that the oxygen content of the niobium will be high and reaction (2) will be shifted to the left. This is verified in Table 4 by the increase over the initial oxygen content in the niobium for the vacuum tests.

4. E. Fromm and H. Jehn, "Reactions of Niobium and Tantalum with Gases at High Temperatures and Low Pressures," *Vacuum*, 19:4, 191-197 (1969).

Partitioning of oxygen between the various constituents has been expressed by several investigators^{3,5,6} through the equilibrium distribution coefficient which is the ratio of the impurity element concentration in the metal to that in lithium; or

$$K_w = \frac{C_{iM}}{C_{iLi}} \quad (6)$$

where C_{iM} and C_{iLi} are the concentrations (in wt %) of the nonmetallic elements in the metal and in the lithium under consideration. By means of this value it is possible to determine the tendency of the nonmetallic element to transfer from the metal to the lithium or vice versa. When the equilibrium distribution coefficient is less than unity, reduced strength may result because of loss of interstitial elements to the lithium. Embrittlement may occur because of transfer of the nonmetallic element from the lithium to the metal when the distribution coefficient is greater than one. Determination of the equilibrium distribution coefficient is made by means of the expression:

$$K_w = \frac{C_{iM}}{C_{iLi}} = \frac{C_{iM}^{\circ}}{C_{iLi}^{\circ}} \exp \left(\frac{\Delta F_{Li}^{\circ} - \Delta F_M^{\circ}}{RT} \right) \quad (7)$$

where C_{iM}° and C_{iLi}° are the saturation concentrations of component, i , in the metal and lithium, respectively, and ΔF_{Li}° and ΔF_M° are the free energies of formation of the stable compounds of the nonmetallic elements with lithium and the metal, respectively.

Distribution coefficients calculated from the data given by Smith and Natesan³ are summarized in Table 5. The values of K_w given in the last column for the Nb-1% Zr alloy were determined by assuming C_{iM}° to be equal to the amount of nonmetallic element required to react completely with the zirconium in the alloy in order to form the stable compound.

Table 5. Equilibrium distribution coefficients for oxygen and nitrogen in lithium-metal couples at 1273K

	Niobium	Zirconium	Zirconium in Nb-1% Zr alloy
Oxygen	2.13×10^{-6}	2.92	0.153
Nitrogen	2460.7	1.75×10^9	6.26×10^7

Niobium would be expected to be deoxidized by lithium since the distribution coefficient is considerably less than 1 (2.13×10^{-6}). However, the data of Table 4 for lithium tests show an increase in oxygen even for areas away from the ceramic wafer. No explanation can be given for this phenomenon except that the system may not be in equilibrium.

For pure zirconium, some tendency for oxygen pickup by the zirconium might be expected because the distribution coefficient has a value of greater than 1 (2.92). However, the value for the alloy is less than 1

5. J. R. DiStefano and E. E. Hoffman, *Corrosion Mechanisms in Refractory Metal-Alkali Metal Systems*, USAEC Report, ORNL-3424, Oak Ridge National Laboratory, September 16, 1963.

6. J. R. DiStefano and A. P. Litman, "Effects of Impurities in Some Refractory Metal-Alkali Metal Systems," *Corrosion* 20:12 392-99 (1964)

(0.153); therefore, the alloy would be expected to lose oxygen. This is verified in Table 4 by the data for the alloy away from the ceramic wafer. The high oxygen value at the ceramic wafer can be rationalized by the reaction (2) and the fact that at the ceramic wafer, equilibrium conditions do not prevail in only 3000 hr.

Distribution coefficients for nitrogen do not explain the results in Table 4 which shows a loss of nitrogen from niobium even though K_w is greater than 1. Two explanations for this are possible. The first explanation can be found in the stability of Li_3N . This compound has a melting point of 815°C so that values for C_{Li} and $\Delta F_{\text{Li}}^\circ$ used in equation (7) are invalid.⁷ Thus, the use of distribution coefficients as calculated for nitrogen may not be justified in the present case. The second explanation could be that the distribution coefficient for nitrogen and zirconium in the alloy is so high (6.26×10^7) that the alloy container removes nitrogen first from the lithium and subsequently from the niobium. The nitrogen content of the lithium decreased from 60 to 19 ppm during the test, so that the latter explanation seems plausible. Since the container for the test was made of Nb-1% Zr alloy, there is a very large mass of alloy for nitrogen removal.

CONCLUSIONS

From this work it is apparent that considerable care must be exercised in the selection of ceramic insulators for CTR application. The presence of a strong reducing agent, such as lithium, can promote reactions not normally expected in a vacuum environment.

Oxygen partitioning between the niobium and Nb-1% Zr alloy and lithium can be explained in terms of the relative partial molar free energies of oxygen in the lithium as compared to those in Al_2O_3 . As the oxygen content of niobium is reduced by lithium, the partial molar free energy of oxygen in niobium is reduced enough so that niobium can reduce Al_2O_3 . Equilibrium distribution coefficients can also be used to achieve the same end.

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7. R. P. Elliott, *Constitution of Binary Alloys: First Supplement*, p. 582, McGraw-Hill Book Company, Inc., New York, 1965.

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