

STUDY OF THE LONG-TERM STABILITY OF Y_2O_3 MHD COATINGS FOR FUSION REACTOR APPLICATIONS -- B. A. Pint and L. D. Chitwood (Oak Ridge National Laboratory)

OBJECTIVE

The objective of this task is to assess the long-term, high-temperature compatibility of high electrical resistance coatings with lithium at high temperatures. Electrically insulating coatings on the first wall of magnetic confinement reactors are essential to reduce the magnetohydrodynamic (MHD) force that would otherwise inhibit the flow of the lithium coolant. Initial experimental work was conducted on bulk ceramics to determine basic lithium compatibility and maximum-use temperatures of candidate ceramics such as AlN and Y_2O_3 . As the next step, coatings of Y_2O_3 are now being evaluated.

SUMMARY

Two batches of Y_2O_3 coatings (12.5 μ m thick) were formed on V-4Cr-4Ti substrates using electron-beam assisted, physical vapor deposition (EB-PVD). The performance of the first batch of coatings was previously reported¹ and was promising. However, additional results on the second batch showed spallation after extended capsule exposures in Li at 700° and 800°C. These observations may be the result of an incompatibility between Y_2O_3 and Li, problems with the processing technique, or batch to batch variation in coating performance. A vacuum rig has been built to measure coating electrical resistance up to 800°C.

PROGRESS AND STATUS

Introduction

Previous work^{2,3} using capsules tests of bulk ceramics in Li showed that Y_2O_3 was an attractive MHD coating candidate. Bulk, polycrystalline Y_2O_3 specimens showed little mass change after 1000h exposures in Li at 700° and 800°C. Also, its resistivity at 700°C was sufficient for the MHD coating application.⁴ Therefore, Y_2O_3 coatings were fabricated to examine their performance before and after exposure to Li.

Experimental Procedure

The Y_2O_3 coatings were made by EB-PVD at Lawrence Livermore National Laboratory by A. Jankowski and J. Hayes. Ten 12.5mm diameter x 1mm thick V-4Cr-4Ti substrates polished to a 1 μ m finish were coated with Y_2O_3 in two batches (designated A and B) by rastering an electron beam over a pressed powder Y_2O_3 target. Using laser profilometry, the thickness of the as-received coatings was determined to be 12.5 μ m. The experimental procedure for lithium exposures has been outlined elsewhere.^{5,6} Coated specimens from both batches were exposed for various times at 700° and 800°C, Table I. Two specimens were exposed to 3, 100h cycles to check the effect of thermal cycling on coating adhesion. One specimen was tested at 700°C and the other at 800°C, Table I. Specimen dimensions and mass were measured before and after exposure (0.02mg accuracy). After exposure, the first specimen (100h at 700°C) was distilled in vacuum at 500-550°C to remove residual Li from the specimen. (The same procedure was used

Table I. Summary of results from lithium exposures.

<u>Conditions</u>		<u>Batch</u>	<u>Mass Change</u>	<u>Comments</u>
700°C	100h	A	-0.77 mg	vacuum distilled
	1000h	A	1.72	no visible change, spalled after handling
	2000h	B	-2.28	remnants only
800°C	3 x 100h	B	0.66	flaky spall
	100h	A	1.03	no visible change
	1000h	A	1.48	no visible change
	2000h	B	-4.19	no coating remained
	3 x 100h	B	1.34	no visible change

to clean the bulk specimens.) However, the V-4Cr-4Ti substrate oxidized due to the relatively low vacuum possible in the distillation system. To avoid this problem, subsequent specimens were cleaned by submerging them in methanol for 24h at room temperature. Previously, coatings were characterized using field emission gun, scanning electron microscopy (SEM), auger electron spectroscopy (AES) and x-ray diffraction (XRD) with 300kV Cu K radiation. Coating resistance was measured up to 500°C with a vacuum pressure of 10^{-6} Torr to limit oxidation of the V-4Cr-4Ti substrate.¹

Results and Discussion

Results for the eight specimens exposed to Li are shown in Table I. The results for batch A specimens and the 800°C cycled specimen were reported previously.¹ The three specimens from batch B that were exposed subsequently all showed near total loss of the coating (after 2000h at 700°C and 2000h at 800°C) or partial spallation of the coating (3 x 100h at 700°C). Thus no resistance measurements could be made after these exposures.

The mass change data for these experiments are not easily explained. Depending on the density assumed for EB-PVD Y_2O_3 , the coating should have had a starting mass of no more than 3.1 mg. Thus the mass gains observed when the coating was adherent of 1.0-1.7mg suggests a substantial reaction with Li or the V-4Cr-4Ti substrate. However, the V-4Cr-4Ti substrate has a mass of 700mg so the total mass change may be dominated by any change in the substrate. There are several possible explanations for the observed mass changes:

- 1) The mass loss of 4.2mg after 2000h at 800°C is too large to be explained by the loss of just the coating. At this time, no Li exposures of bare substrates have been performed for comparison. Some dissolution of the V alloy substrate or selective loss of certain elements (e.g. O or Ti) may have occurred during the extended exposure to Li (2000h at 800°C) resulting in an additional mass loss. Recent analysis of V-4Cr-4Ti after exposure to Li-2%Ca showed that selective attack could occur.⁷ While this exposure was the longest time at the highest temperature, none of the other exposures resulted in a mass loss that could not be accounted for by loss of the coating.
- 2) The large mass gains for the intact coatings may reflect a reaction with the V-4Cr-4Ti substrate, possibly the uptake of N from the Li. The starting N content of the Li was measured³ as 140ppmw giving 0.7mg N in the 5g of Li in the capsule. However, the V alloy capsule walls should have absorbed some of this total. Therefore, it is difficult to account for this mass gain by the substrate. (Future capsule experiments will use Mo capsules to reduce the possibility of the capsule affecting

the results.)

3) Previously, AES analysis had observed the formation of Y-Ti oxides on the surface of the Y_2O_3 coating after exposure to Li for 1000h at 800°C.¹ This suggests that Ti may be selectively being removed from the substrate or V alloy capsule walls. Complete conversion of Y_2O_3 to $YTiO_3$ (or another Y-Ti oxide) could increase the mass by up to 2mg but XRD results indicated complete conversion did not occur.

4) The examination by AES also revealed that there was some residual Li in the coatings after cleaning¹ and XRD analysis detected the formation of $YLiO_2$ but the mass increase due to formation of this compound would only be as much as 0.4mg for complete conversion.

A new resistance measurement rig was built using the existing vacuum system to enable higher temperature measurements. The previous system had a maximum temperature capability of 550°C due to its large thermal mass and small resistance heating elements. The current specimen holder (Figure 1) places the specimen on the Mo-enclosed heating element resulting in better temperature control to >800°C. In the previous report, the possible degradation of the Au electrode by residual Li in the coating during the resistance measurement at 550°C in vacuum was discussed. However, further examination by AES of that specimen after heating revealed that the electrode was largely intact but covered with carbon. Several of the surviving coated specimens were recoated with an Au electrode for examination with the new equipment but all were shorted. Further work will be done to characterize the remaining coatings by cross-sectioning them.

More testing is needed on Y_2O_3 coatings. The current results do not provide sufficient information to draw a strong conclusion about the compatibility of Y_2O_3 with Li. The formation of the $LiYO_2$ phase, which was observed in a previous study,⁸ suggests that Y_2O_3 may not be sufficiently compatible. The spallation observed in these tests may be due to the thermal expansion mismatch between V-4Cr-4Ti and Y_2O_3 . However, it also could be due to the coating process or batch-to-batch variation in the EB-PVD process. Additional Y_2O_3 coatings made by other processes need to be evaluated before a stronger conclusion can be made.

REFERENCES

1. B. A. Pint and L.D. Chitwood, DOE/ER-0313/32 (2002) 101.
2. B. A. Pint, J. H. DeVan and J. R. DiStefano, DOE/ER-0313/31 (2001) 132.
3. B. A. Pint, J. H. DeVan and J. R. DiStefano, J. Nucl. Mater. in press.

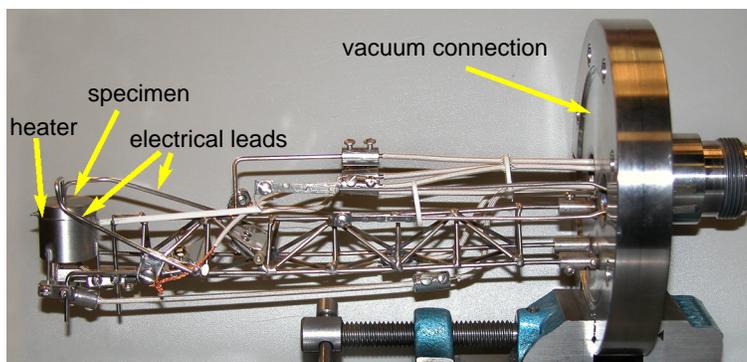


Figure 1. Specimen heater/holder that is inserted into the resistance measurement rig vacuum chamber.

4. K. Natesan, C. B. Reed, M. Uz, J. H. Park and D. L. Smith, ANL/TD/TM00-10 (2000).
5. B. A. Pint, L.D. Chitwood, J. H. DeVan and J. R. DiStefano, DOE/ER-0313/27 (2000) 49.
6. B. A. Pint, L. D. Chitwood and J. R. DiStefano, J. Nucl. Mater. 289 (2001) 52.
7. O. I. Eliseeva, Mater. Sci. 37 (2001) 910.
8. T. Mitsuyama, T. Terai, T. Yoneoka and S. Tanaka, Fusion Eng. Design 39-4 (1998) 811.