

STUDY OF THE LONG-TERM STABILITY OF MHD COATINGS FOR FUSION REACTOR APPLICATIONS -- B. A. Pint and J. R. DiStefano (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, magnetohydrodynamic (MHD) coatings on the first wall of magnetic confinement reactors are essential to reduce the MHD force that would otherwise inhibit the flow of the lithium coolant. Initial experimental work is being conducted on bulk ceramics to determine basic lithium compatibility and maximum-use temperatures of candidate ceramics such as AlN and CaO.

SUMMARY

Bulk specimens of two candidate compositions, CaO and AlN+0.04wt.%Y, for insulating coatings in a lithium-cooled fusion reactor have been exposed to lithium in 1000 h isothermal tests at 500°-800°C to determine the maximum temperature at which acceptable compatibility is likely. Because high mass losses for AlN were observed at 700°C, additional testing has focused on this temperature in order to examine what changes may slow the rate of dissolution. Changing from a vanadium alloy test capsule to a Mo capsule appears to have the largest effect at 700°C. For single crystal specimens of CaO, high mass losses have been observed after exposure at 600°C. In this case, changing to a Mo capsule or adding Ca or O to the lithium have not consistently showed a beneficial effect. For testing at 700°C, neither doping the Li with Ca or O altered the high mass losses. These results suggest that CaO may be limited to exposure temperatures of less than 600°C but that AlN may be able to operate above 600°C.

PROGRESS AND STATUS

Experimental Procedure

Current work focused on two compositions of bulk specimens. High purity AlN (0.9wt%O, 0.04wt% Y) from Tokuyama Corp (Shapal SH-50) was made by nitriding Al₂O₃. The AlN specimens measured 2x8x12mm, had an average grain size of 7-8µm and a density of 3.25g/cm³. Single crystals of CaO were obtained from Commercial Crystal Laboratories (Naples, FL) and measured 2x6x12mm. Previous work on these materials had used AlN with 5wt%Y₂O₃ and polycrystalline CaO specimens.¹ Specimen dimensions and mass were measured before and after exposure. The mass change accuracy was 0.01mg/cm².

The experimental procedure for lithium exposures has been outlined elsewhere^{2,3}. Bulk ceramic specimens were exposed for 1000h at 500°-800°C. In some tests, additions of Li₃N, Li₂O or Ca (99.5% purity) granules were made to increase the impurity levels in the lithium to nominally 1000ppmw of nitrogen, 1000-2000ppmw oxygen or 1-10wt%Ca, respectively. In order to explore the effect of the vanadium inner capsule on the experiment, a molybdenum capsule that was expected to be more inert to reactions of interstitial impurities in the lithium was used for some experiments. After exposure to Li, specimens were distilled in vacuum at 500-550°C to remove any residual Li from the specimen. Additional characterization was performed on selected specimens.

Results and Discussion

Previous work on AlN showed relatively low mass gains at 600°C but much higher mass losses (indicating dissolution) at 700°C. Thus the focus in these experiments was on altering the dissolution rate at the higher temperature. Figure 1 summarizes the mass change results for AlN. With a V alloy capsule, the addition of 1000ppm nitrogen did not significantly change the mass loss at 700°C. However, switching to a Mo capsule did reduce the mass loss after 1000h to 1.7mg/cm². The reason for this difference is believed to be the gettering of N by the V alloy capsule. Previous work¹ showed the Al content of the Li increased with exposure temperature but not the N content. If nitrogen from dissolved AlN is continually getterred from the lithium into the V capsule, then the rate of dissolution may be higher than might be expected in a reactor environment where the AlN coating would separate the lithium from the vanadium.

Table I shows the composition of the lithium after several recent exposures. Unfortunately, two of the samples were contaminated prior to spectrographic analysis and contained very high N levels. However, the available data appears to support a hypothesis that N is getterred by a V alloy capsule but not by a Mo capsule. In particular, the nitrogen level was only 120ppm when 1000ppm N was added prior to the test with a V capsule at 700°C, Table I. In contrast, with a Mo capsule at 700°C, 330ppm N was measured in the lithium after exposure. The Al levels likewise reflected the reduced attack at 700°C when a Mo capsule was used.

For single crystal CaO, high mass losses were observed at 600°C. Therefore, additions were made to the lithium in an effort to improve performance at this temperature. Figure 2 summarizes the results for various test conditions. In this case, neither V or Mo would be expected to getter O from Li; therefore, no effect of capsule type was expected and virtually none was observed. Based on earlier work⁴, it was hoped that O or Ca doping of the lithium would reduce the rate of

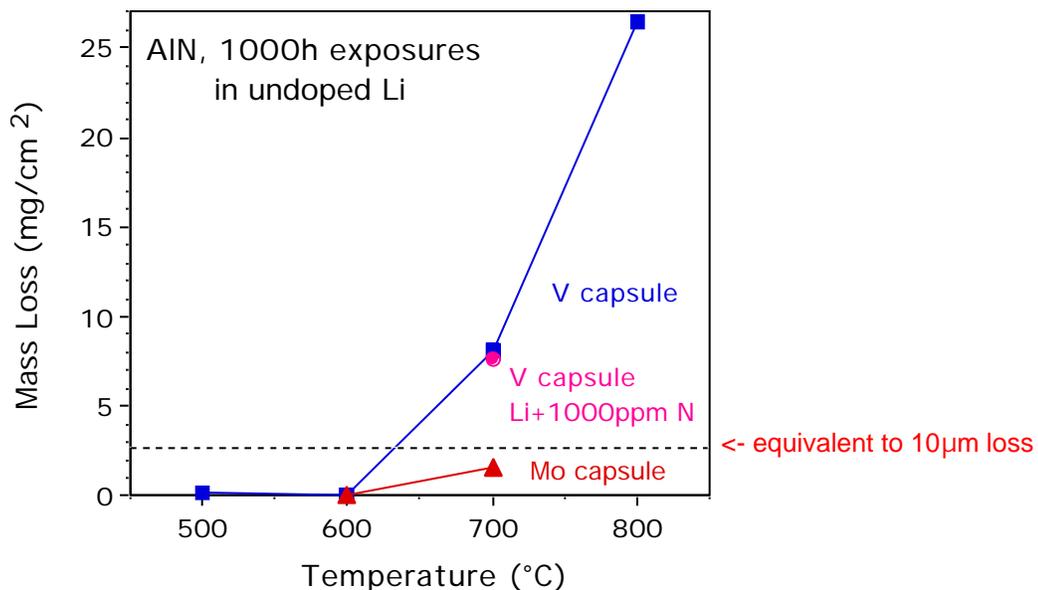


Figure 1. Mass losses for AlN+0.04Y after 1000h at various temperatures. At 700°C, switching from a V alloy capsule to a Mo capsule had a more significant effect than the addition of 1000ppm N to the lithium. A mass loss of 3.2 mg/cm² corresponds to a 10µm loss in material thickness.

Table I. Lithium composition determined by spectrographic analysis.

<u>Conditions</u>	<u>Nitrogen</u> (wppm)	<u>Aluminum</u> (wppm)	<u>Yttrium</u> (wppm)
Starting Lithium*	140	<0.3	<0.3
500°C V can	150	1	<0.3
600°C Mo can	contaminated	1	<0.4
700°C V can	contaminated	110	<0.4
700°C V can + 1000ppm N	120	160	<0.4
700°C Mo can	330	3	<0.4

* 40 metallic elements examined, only Cu (15wppm) was above detectability limit

dissolution at 600°C. The addition of 1000 or 2000ppm O resulted in a modest reduction in the amount of dissolution. However, the mass losses were still equivalent to more than a 10 μ m loss in material after 1000h. An addition of 1wt%Ca in two separate tests resulted in a mass gain rather than a mass loss without Ca. The reason for the mass gain is not known but may be the result of Ca remaining on the specimen after distillation of the lithium after the test. Unexpectedly, the addition of more Ca (3 and 5wt%) resulted in specimen mass losses, Figure 2. The specimen broke after exposure to Li + 10wt.%Ca preventing an accurate mass change measurement. The results with higher Ca levels suggests that Ca doping may not be an effective strategy.

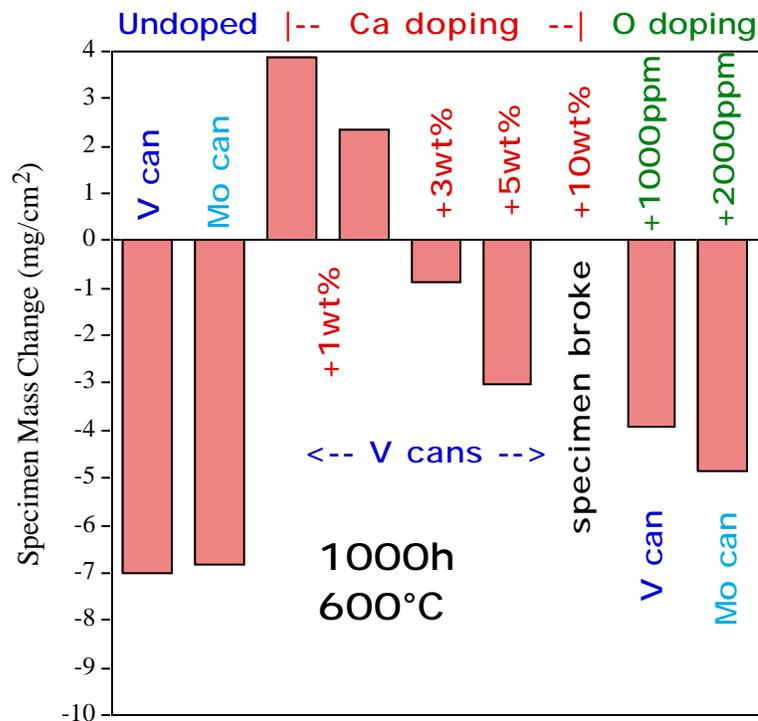


Figure 2. Mass changes for single crystal CaO after 1000h at 600°C with various additives to the lithium and both V and Mo capsules. A mass loss of 3.2 mg/cm² corresponds to a 10 μ m loss in material thickness.

Similar doping tests also were performed at 700°C. In this case, the addition of 1wt.%Ca had very little effect on the mass loss. An addition of 1000ppm O reduced the mass loss compared to undoped Li but a significant amount of material was still lost during the 1000h exposure. As noted previously, these rates of dissolution are consistent with the high equilibrium levels of Ca and O in solution in Li at these temperature.⁴

Because of the desire for operating temperatures as high as 750°-800°C,^{5,6} new ceramic candidates are being tested in bulk form. Single crystals of Sc_2O_3 and Y_2O_3 are being exposed at 600°-800°C as are polycrystalline samples of Er_2O_3 and YScO_3 . The latter two compositions were not available in single crystal form so they were produced by conventional ceramic processing from powders (cold compacted and sintered). These tests should indicate if they would be possible candidates for higher temperature operation.

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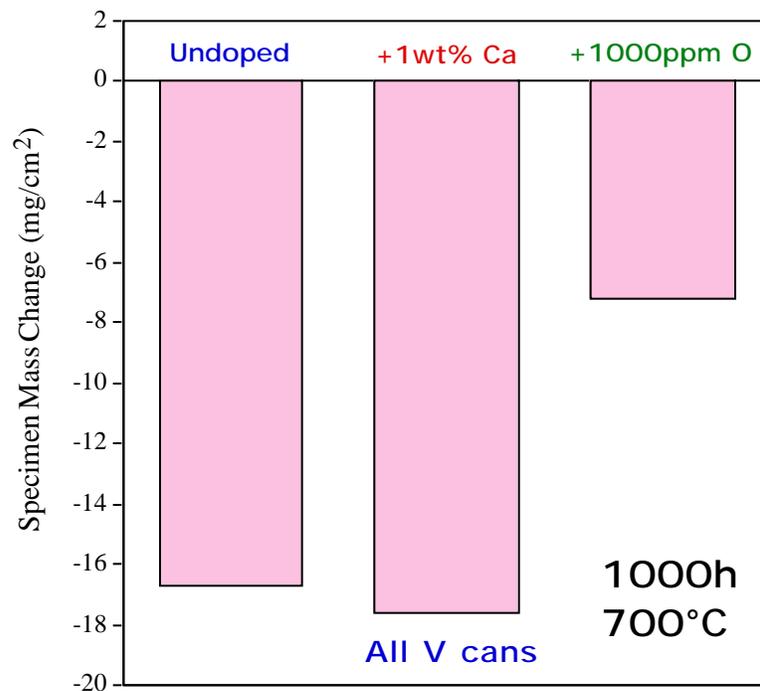


Figure 3. Mass changes for single crystal CaO after 1000h at 700°C with lithium and lithium doped with Ca or O. Neither addition prevented very high levels of dissolution at this temperature.