6.1 ADDITIONAL CHARACTERIZATION OF V-4Cr-4Ti AND MHD COATINGS EXPOSED TO FLOWING Li – B. A. Pint and K. A. Unocic (Oak Ridge National Laboratory)

OBJECTIVE

A flowing Li thermal convection loop was operated with V-4Cr-4Ti specimens and multi-layer electrically-insulating coatings needed to reduce the magneto hydrodynamic (MHD) force in the first wall of a lithium cooled blanket. Both types of specimens are now being characterized to examine the effect of exposure in flowing Li.

SUMMARY

Additional results are presented on the characterization of V-4Cr-4Ti tensile specimens and MHD coatings exposed to flowing Li. For the alloy specimens, anneals were performed at 400° and 550°C on tensile specimens to determine the effect of the thermal exposure without Li. The 550°C anneal resulted in a higher yield stress and lower serration amplitude for the dynamic strain aging at 500°C. For the MHD specimens, the higher temperature exposures showed a degradation in the high temperature resistivity after exposure. Metallographic cross-sections indicated the formation of a second phase at the alloy-coating interface. While $YLiO_2$ is suspected, this phase has not been identified by EELS (electron energy loss spectroscopy) or EPMA (electron microprobe analysis).

PROGRESS AND STATUS

Introduction

Previous reports[1-4] presented 500°C tensile behavior of V-4Cr-4Ti after various exposures including flowing Li in a thermal convection loop with a peak temperature of 700°C for 2,355h.[5] The results were unusual and more characterization was warranted along with additional thermal anneals to separate the effects of time at temperature from the flowing Li. Also, the dual layer (Y_2O_3/V) MHD coatings deposited by physical vapor deposition that were exposed in the same loop [5] were not fully characterized because a method was not apparent to evaluate the electrical resistivity without damaging the coatings. A micro-milling technique has been used to cut specimens suitable for resistivity measurements out of the original coated coupons. A vacuum rig for measuring resistance at high temperature has been reassembled and each of the coatings measured and then metallographic cross-sections made for characterization. Focused ion beam (FIB) milling is being used to prepare specimens for transmission electron microscopy (TEM).

Experimental Procedure

Details of the thermal convection loop exposure have been presented previously.[5] The specimens consisted of miniature tensile specimens (type SS-3: 25 x 4 x 0.9mm), tab specimens and specimens with a dual layer MHD coating linked in a chain held together with V-4Cr-4Ti wire. The tensile specimens were annealed for 1h at 1050°C prior to exposure in Li. The exposure temperature for each specimen is estimated by using a linear extrapolation of the temperatures measured at the top and bottom of the each leg.[5] Tensile specimens from the same batch of specimens were annealed in Ar-filled quartz ampoules at 400° and 550°C for 2,355h to simulate the loop temperature exposure. Previously, a 700°C anneal was conducted [5]. Tensile testing was conducted at 500°C in a vacuum with a base pressure of 10^{-6}Pa (10^{-8}Torr) and a strain rate of $10^{-3}s^{-1}$. After tensile testing, the specimens were sectioned and metallographically polished to measure the grain size and hardness. TEM specimens were prepared to further examine the microstructure. For the MHD coated specimens, electrical resistance across the coating was measured in vacuum (~10^{-5}-10^{-6} Pa) at 100° increments from 25°-700°C on sub-specimens

(4mm x 4mm) cut from the original coated specimens in order to electrically isolate the outer vanadium layer from the V-4Cr-4Ti substrate. The remaining pieces were characterized by scanning electron microscopy (SEM), EPMA, x-ray photoelectron spectroscopy (XPS), and scanning transmission microscopy (STEM), including energy x-ray dispersive spectroscopy (EDS) and EELS with specimens prepared by focused ion beam milling.

Results and Discussion

Figure 1 shows the stress-strain behavior of several specimens during tensile testing at 500°C. The recently completed anneal at 550°C exhibited similar DSA behavior as the 459°C specimen from the bottom of the hot leg in the loop. At higher temperatures, there was very little effect on the DSA behavior. Figure 2 shows the amplitude of the DSA serrations measured during tensile testing at 500°C as a function of the measured yield stress at 500°C. The new 550°C data point fits along the same curve including an 800°C isothermal Li exposure for 1000h and two previous studies [6,7]. A similar relationship was observed with the ultimate tensile strength. A previous explanation was that the lower temperature exposures showed a lower amplitude because O was depleted in the presence of Li but little C and N update occurred. However, with a similar amplitude observed in the 550°C anneal, this explanation does not seem to be applicable. It appears more likely that the Li exposure had little effect on the properties and instead the thermal aging dominated the observed changes.

Figure 3 shows additional nanohardness measurements made on these specimens after sectioning. The specimens exposed in the loop show increased surface hardness that may be attributed to C and N uptake near the surface. The higher temperature loop and 800°C Li exposures show reduced hardness away from the surface, perhaps due to O depletion. These measurements are generally consistent with the bulk hardness measurements that were lowest after the highest temperature exposures [4]. TEM characterization is continuing for these specimens but does not appear to be resolving the effect of interstitials.

For the V/Y_2O_3 MHD coatings, Figure 4 shows the resistivity as a function of temperature for the coatings exposed at various temperatures in the loop and one annealed at 700°C for the same exposure time [8].

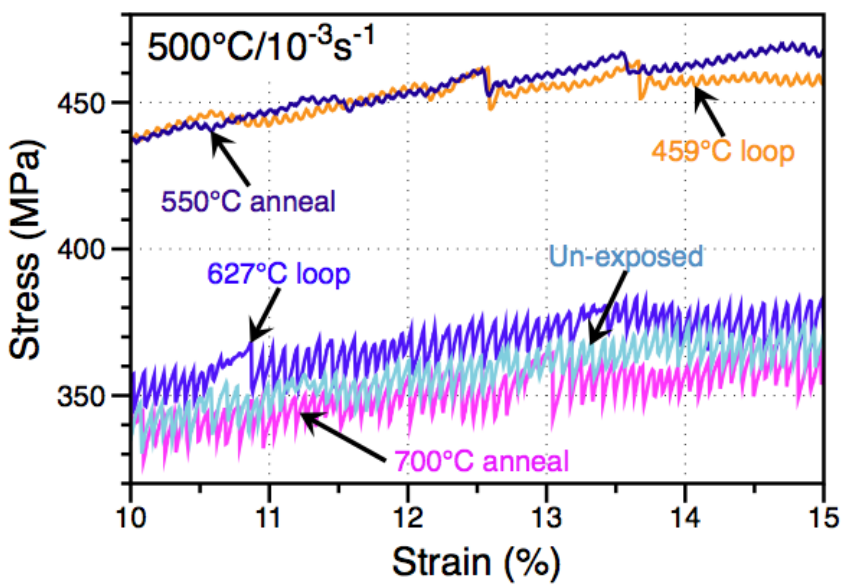

Figure 1. Stress-strain behavior of V-4Cr-4Ti specimens exposed for 2,355h either in a Li loop or in an Ar-filled ampoule. The values were narrowed to illustrate the DSA serrations.

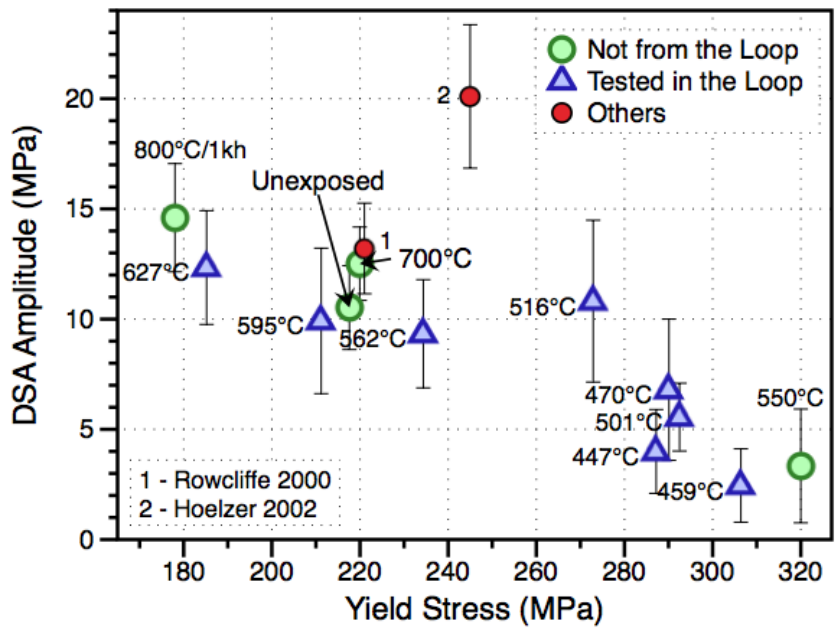

Figure 2. Amplitude of the dynamic strain aging observed at 500°C as a function of 500°C yield stress for V-4Cr-4Ti specimens exposed in the Li loop, other exposures and other reports [6,7] from the literature. The bars note the standard deviation of the measurements.

Above ~504°C in the loop, the coatings were strongly degraded and the resistivity was below the established metric for effectiveness [9]. Based on the cross-sections, this decrease is not surprising because the coatings exposed at higher temperature appeared to form a second phase at the Y_2O_3-V-4Cr-4Ti substrate, Figure 5. The layer did not form during the 700°C thermal anneal, Figure 5a, and got

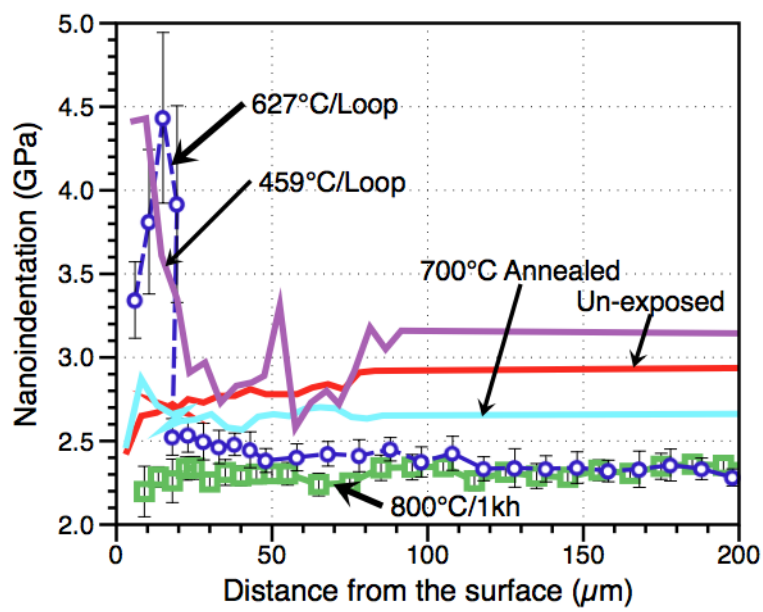

Figure 3. Nanoindentation hardness as a function of depth for various V-4Cr-4Ti specimens. The bars mark standard deviations for the measurements.

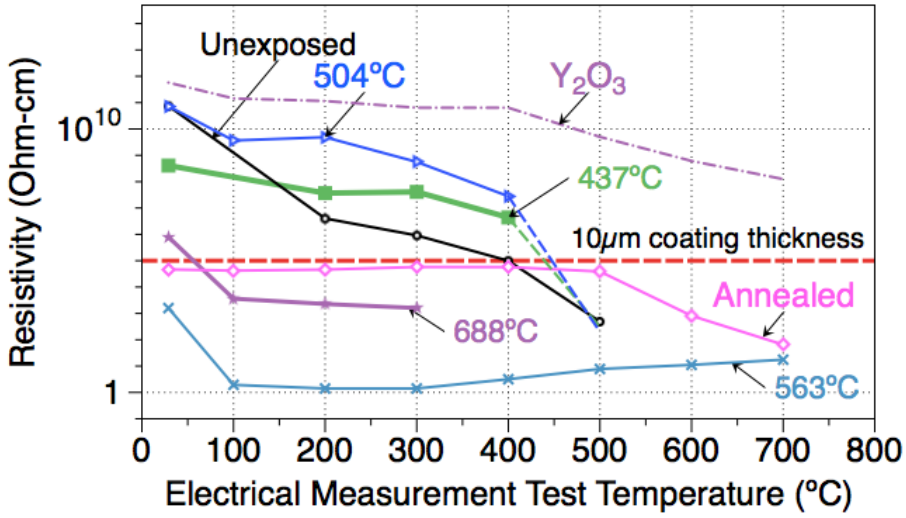

Figure 4. Resistivity of the MHD coatings exposed at different temperature in the loop as a function of measurement temperature. For reference, values for bulk Y_2O_3 and the MHD coating metric are shown.

progressive larger at higher loop exposure temperatures. Furthermore, no other elements could be detected in the layer besides Y and O using EPMA, Figure 6. Therefore, it does not involve a reaction between the coating and substrate. The most likely conclusion is that the V over coating was not sound and Li leaked along the V-alloy interface resulting in $LiYO_2$ formation [10,11]. However, XPS and EELS

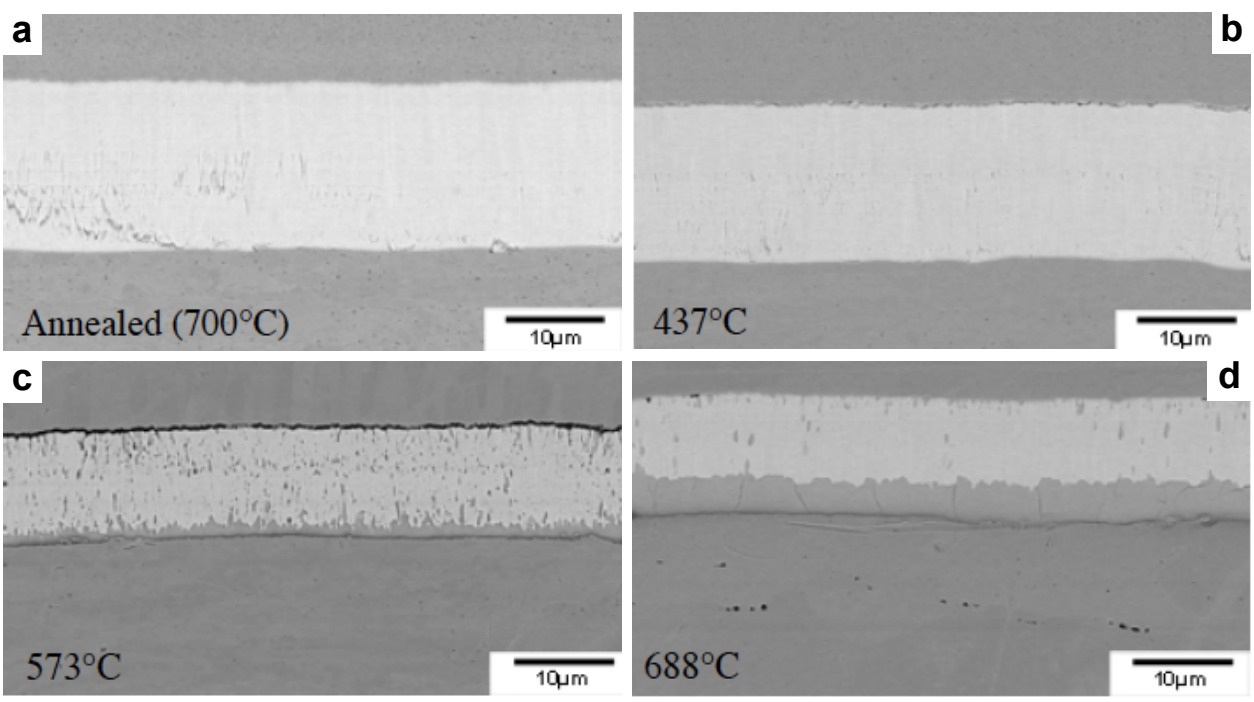

Figure 5. SEM backscattered electron images of polished sections of MHD coatings (a) after 700°C anneal and after loop exposures at (b) 437°C, (c) 573°C and (d) 688°C.

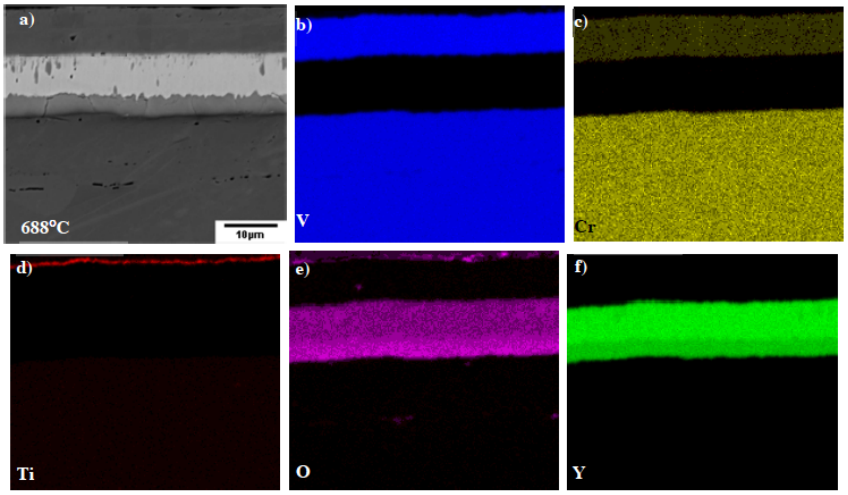

Figure 6. a) BSE image of the area used for EPMA analysis from the coating exposed to Li at 688°C and EPMA maps (b) V, (c) Cr, (d) Ti, (e) O and (f) Y.

have not been able to detect Li in this new phase. Additional TEM work is in progress to attempt to use selected area diffraction to identify the phase.

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