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# Development, Characterization and Evaluation of Materials for Open Cycle MHD

Quarterly Report for the Period Ending June 1979

J. Lambert Bates D. D. Marchant

October 1979

Prepared for the U.S. Department of Energy under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory Operated for the U.S. Department of Energy by Battelle Memorial Institute



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DEVELOPMENT, CHARACTERIZATION AND EVALUATION OF MATERIALS FOR OPEN CYCLE MHD

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Pacific Northwest Laboratory Richland, Washington 99352

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#### 1.0 OBJECTIVES

The objectives of this program are to develop, test, characterize, and evaluate materials for open-cycle, coal-fired MHD power generators. The specific immediate goals emphasize electrode and insulator materials, including: 1) testing and evaluation of the enhanced effects of alkali seed on materials in a dc electric field; 2) development and testing of improved electrodes and insulators with controlled microstructures, compositions and properties; and 3) characterization and evaluation of materials relating to both the US MHD Program and the US-USSR Cooperative Program for MHD power generators.

#### 2.0 SCOPE

The scope of this program encompasses the following areas:

- Reproducible laboratory testing of both ceramic and metal electrode
  materials and insulator materials in alkali seed/slag under dc electric
  current and voltages, as functions of temperature, and seed/slag
  composition.
- Development and fabrication of electrodes, insulators, and other related materials with controlled electrical, chemical, and physical properties, including evaluation in laboratory tests in US MHD test facilities and as part of the US-USSR testing program.
- Characterization and evaluation of materials tested under the US MHD programs and the US-USSR including the measurement and analysis of structural, chemical, and thermophysical properties of electrodes, insulators, slag, and other related materials before and after testing.

#### 3.0 TECHNICAL PROGRESS

#### 3.1 ELECTROCHEMICAL TESTING OF ELECTRODE MATERIALS

Yttrium chromites have demonstrated in laboratory tests improved thermal and electrochemical properties when compared to analogous lanthanum chromite compositions.  $^{(1-3)}$  The present electrochemical tests were made to evaluate why the electrochemical corrosion of YMg $_{0.05}$ Cr $_{0.95}$ O $_3$  is less in an Eastern, high-iron coal slag, Illinois No. 6 (III #6-1) than in a Western low-iron coal slag, Montana "Rosebud" (MR-1). Two magnesium-doped yttrium chromites of slightly different compositions, but with similar structures and phases, were tested under similar conditions (Table 1). Both contained MgCrO $_3$  second phase uniformly distributed in a matrix of YCrO $_3$  equiaxed grains. The chromite contained some irregularly shaped pores ( $\sim$ 15  $\mu$ m) at the grain boundaries. No impurity concentrations were found at the grain boundaries.

<u>TABLE 1</u>. Properties and Corrosion of Yttrium Chromites

Test No.	Composition	Density, g/cm3	Slag	Temp.,	Density, Amp/cm <sup>2</sup>	Time	Corrosion Rate Cathode	μ/coul. Anode
158	YMg <sub>0.05</sub> Cr <sub>0.95</sub> 0 <sub>3</sub>	5.3	111-6-1	1723	0.8	47.5 hr	14	7
198	Y <sub>0.95</sub> Mg <sub>0.05</sub> Cr0 <sub>3</sub>	4.9	MR-1	1729	0.9	9 hr 35 m	75	42

The YMg $_{0.05}$ Cr $_{0.95}$ O $_3$  was tested in an electrochemical cell $^{(4)}$  with an anode, cathode, and Pt voltage probe immersed in the molten slag contained in a sintered  $\alpha$ -Al $_2$ O $_3$  crucible. Because the corrosion in the III. #6-1, Test 158, was significantly less than in the MR-1, Test 198, the time of the test was  $\sim$ 5 times longer. However, the remaining test parameters, i.e., voltage, current, temperature and atmosphere, were nearly identical (Tables 2 & 3). However, the time may have influenced the corrosion product morphology and corrosion rate of the sample; therefore, the analytical compositions of the reaction products and the evaluations may differ. However, these effects are considered small compared to the large differences in the electrochemical corrosion rates. It should be noted that electrical conductivity of the two slags are significantly different, both in magnitude and in conduction transport (transference number).  $^{(5-6)}$ 

TABLE 2. Comparison of Anodes/Cathodes From Test 158 and Test 198 (Reference Area)

	ILL No. 6 (Test 158)	Similarities	MR-1 (Test 198)
ANODE:	Granular (Mg <sub>X</sub> Cry)O <sub>z</sub>	No Slag	Granular (Mg <sub>X</sub> Cr <sub>y</sub> )0 <sub>z</sub>
	Small quantities ~	Overall composition same	Y2Si <sub>2</sub> 07 phase at grain boundaries
CATHODE	Phase contains Fe and less Al	Mg-Cr rich phase	Higher Si and Ca content in slag near electrode
	Grain boundaries contain less Y <sub>2</sub> Si <sub>2</sub> O <sub>7</sub>	Unknown phase at grain boundaries	Electrode matrix higher in Si and lower in Y
	More K near Pt leadout		More 2nd phase along grain boundary
			Grain boundaries contain more Y <sub>2</sub> Si <sub>2</sub> O <sub>7</sub>

TABLE 3. Comparison of Anodes/Cathodes from Tests 158 and Test 198 (Reaction Zone)

	ILL No. 6 (Test 158)	Similarities	MR-1 (Test 198)
ANODE:	Metallic iron	Matrix relatively unchanged except near slag interface	Major product (CR <sub>.8</sub> A1 <sub>.2</sub> ) <sub>2</sub> 0 <sub>3</sub>
	(Fe, Al, Cr) <sub>2</sub> 0 <sub>3</sub> with Fe/Al/Cr = 2:2:3	Little grain boundary penetration of slag	Dense $\text{Cr}_2\text{O}_3$ found in reaction zone
	Slag single phase	Slight increase Y/Cr ratio	Slag multiphase with Y2Si2O7 and depleted in K
		Reaction zone is discrete granular particles surround by slag	led
CATHODE	Matrix relatively unchanged except at reaction zone	Slag surrounds reaction products	Slag matrix consists of isolated grains of Y and YCrO <sub>3</sub>
	Massive K penetration and interaction near Pt	Slag content 1/2 of starting value	Y <sub>2</sub> O <sub>3</sub> and Y <sub>2</sub> Si <sub>2</sub> O <sub>7</sub> at grain boundaries
	Reaction product (Al,Fe,Cr) <sub>2</sub> O <sub>3</sub> with Al/Fe/Cr = 2:3:4		$^{\text{Mg}(\text{Al}_{0.4}\text{Cr}_{0.6})_2^0}$ 3
	Slag depleted in Fe		

The voltage characteristics and variations at both anode and cathode were monitored with a Pt voltage probe positioned in the slag equidistant from each electrode. Fluctuations in voltage, up to  $\pm 20\%$  of the anode-cathode voltage, occurred in the MR-1 low iron slag. The fluctuations are observed most frequently and intense on the anode with a frequency of 0.1 to 1 hertz. As discussed later, these fluctuations may result from the formation of either a cation depleted boundary and/or the formation of oxygen gas bubbles in the slag near the electrode interface.

The electrical potential through the electrodes and across the slag in the III #6-1 high-iron slag ( $\sim 4$  volts) during the entire test was lower than in the MR-1 slag (7 volts initially rising to 14 volts at the termination of the test). The difference between the two initial electrical potentials, 4 to 7 volts in the III #6-1 and MR-1 slags, is undoubtedly due to higher resistivity of the MR-1 slag. The increase in voltage with time in the MR-1 slag can be attributed to electrochemical decomposition of the slag and/or ionic buildup in the slag near the slag/electrode interface, in particular at the cathode. Thermodynamic data suggest that at these higher voltages, direct decomposition of these silicates does occur with subsequent increased reaction rates, specifically at the cathode.

The chromites were frozen in the slag and examined by optical and scanning electron microscopy with EDX. The corrosion rates were determined from the microstructural cross sections. These results are summarized in Tables 2 and 3. A reference area was selected for each chromite near the Pt lead-out and attachment where corrosion did not occur. Some reaction with potassium seed was observed above the surface of the slag near the platinum for Test 158.

The anodes and cathodes differed significantly. The anodes exhibited a smooth reaction surface compared to the rough corroded surface of the cathode (Figure 1). The reaction of the cathodes was significantly more severe, although some similar reaction products were observed. These differences and similarities are summarized in Table 2 and are illustrated in the detailed microstructures (Figures 2-4.)

The Mg-Cr rich oxide second phase present in the original YCrO  $_{\!3}$  cathode reacted with the Al and Fe of the slag, and in the MR-1 slag the yttrium

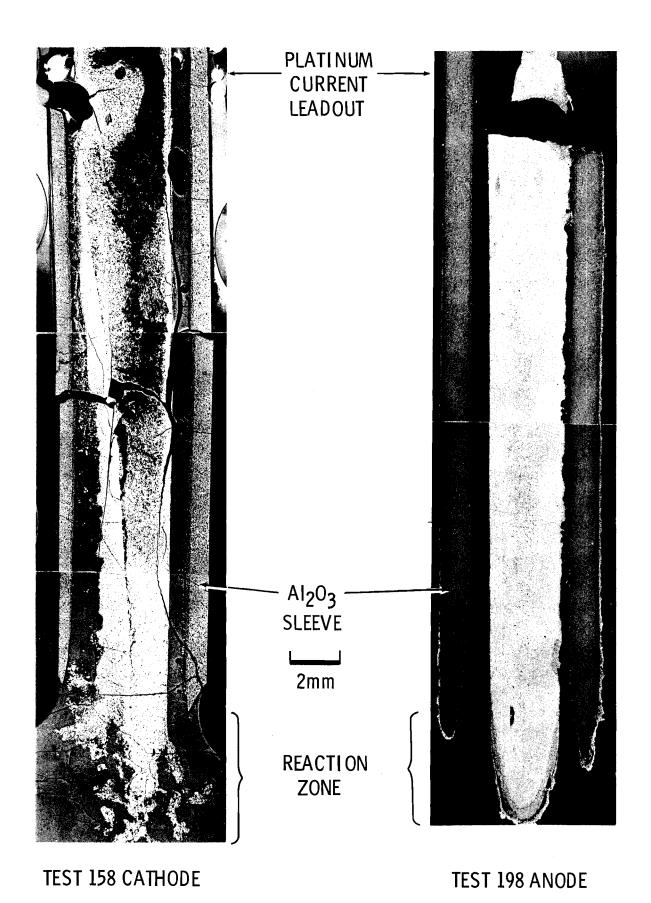


FIGURE 1. Microstructure of YMg0.05Cr0.9503, Test 158 (Cathode in MR-1 slag) and Test (Anode in III. #6-1 slag).

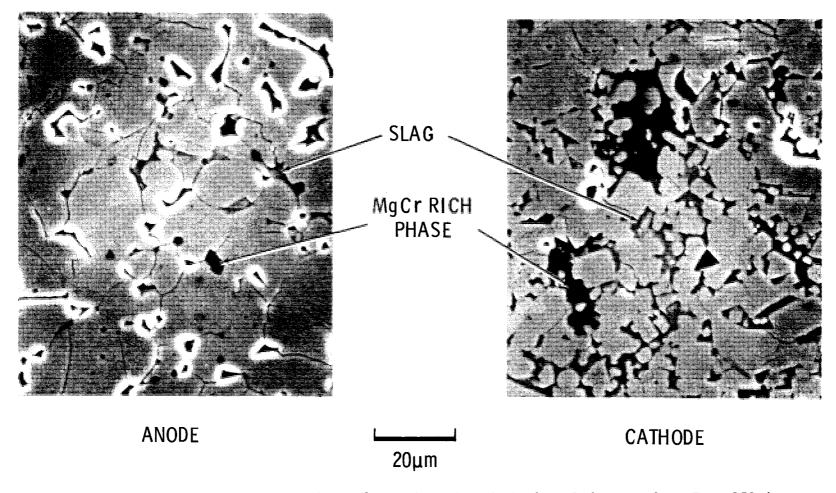


FIGURE 2. Microstructure of  $\rm YMg_{0.05}Cr_{0.95}^{0}_{3}$  anode and cathode from Reference Area Test 158 in MR-1 slag.

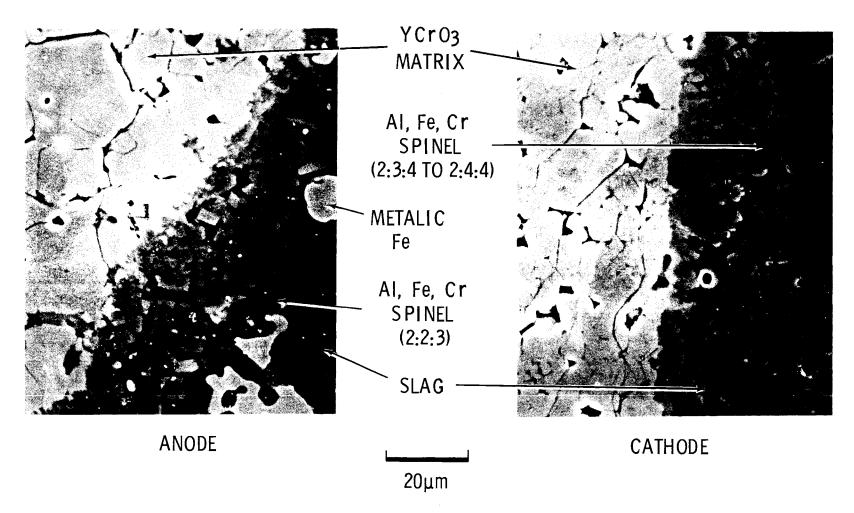


FIGURE 3. Microstructure of reaction zone for  ${\rm YMg}_{0.05}{\rm Cr}_{0.98}{\rm O}_3$  anode and cathode from Test 158 in MR-1 slag.

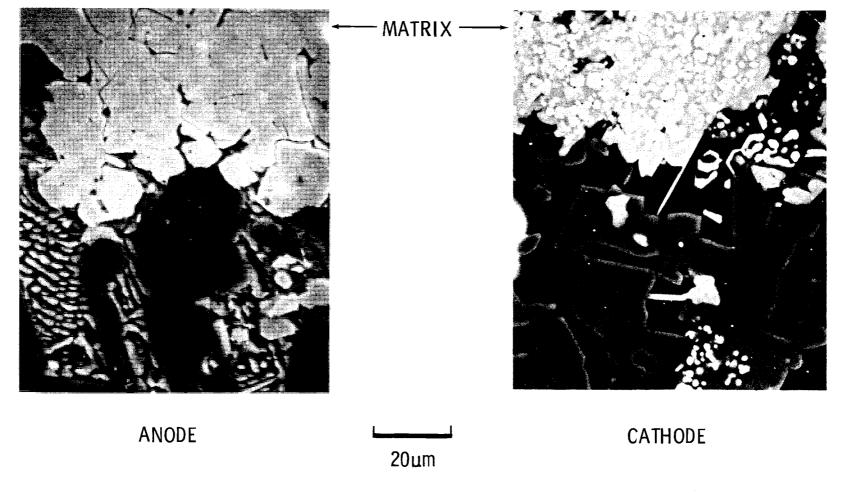


FIGURE 4. Microstructure at reaction zone of  $YMg_{0.05}Cr_{0.95}O_3$  anode and cathode from Test 198 in Ill #6-1 slag.

reacted with the silicate of the slag forming  $Y_2Si_2O_7$  near the slag/electrode surface and also in the cathode grain boundaries where slag penetrated. In the anode tested in MR-1 the leaching of yttrium from the electrode near the reaction interface is almost complete, leaving an  $(Al,Cr)_2O_3$  or Mg(Al,Cr) $_2O_4$  spinel. In the high-iron slag (Ill #6-1), the reaction resulting in the formation of  $Y_2Si_2O_7$  is less. The resultant spinel also contains iron  $[(Al,Cr,Fe)_2O_3]$ . In addition, free metallic iron particles are found in the cathode-slag interface resulting from the decomposition (reduction) of the high-iron slag. The source of alumina for the spinel appears to be the slag. Some Al may have come from the dissolution of the  $Al_2O_3$  sleeve or crucible.

The potassium does not appear to be directly involved in the reactions. Potassium did, however, migrate through the cathode of Test 158 (high-iron slag) to interact near the Pt current attachment. However, no potassium was found in the grain boundaries of the chromite matrix or in the reaction products. Potassium is depleted from the slags in the reaction zones at the anode with very slight depletion at the cathode, suggesting some potassium migration.

The corrosion of the YCrO $_3$  in the slag appears to result from the reaction of silicon in the slag with the YCrO $_3$  forming Y $_2$ Si $_2$ O $_7$ . The Cr and Mg combine with the Al and Fe to form stable spinels. The enhanced corrosion at the cathode is attributed to the concentration of cations in the slag adjacent to the cathode, either from migration or decomposition of the slag. The slag penetrates the grain boundaries. This would suggest that the K or Ca which can migrate most easily may be involved in the corrosion, but does not form reaction products.

The enhanced corrosion in the Western low-iron slag (MR-1) compared to the low-iron III #6 slag is attributed to 1) the higher electrical conductivity of the slag, and 2) the lower ionic and higher electronic conduction in the iron containing slags. This is equivalent to a decrease in the ionic transport and results in lower voltages and less potential decomposition of the slag, and lower effective number of coulombs experienced by the YCrO $_3$  and slag.

Further evaluation of  $Y(Mg_{0.05}Cr_{0.95}O_3)$  will be made in more realistic MHD conditions in the Westinghouse MHD Test facility (WESTF). Materials for this test are now being fabricated.

#### 3.2 FABRICATION OF HAFNIUM-RARE EARTH OXIDE ELECTRODES

Hafnium-rare earth oxides demonstrated during laboratory tests excellent thermal stability, adequate electrical conductivity, and high electrochemical resistance in molten potassium seed and coal slags (1,7,8). Further testing is planned under simulated MHD conditions in WESTF MHD Test Facility (Westinghouse Research Center). However, use of reproducible and high-density bars is required. Because these materials are not commercially available and techniques for fabricating the compositions have not been developed, it has been necessary to develop methods for fabricating powders from the raw materials and for sintering dense compacts.

Simple techniques of mixing the individual oxides, pressing, and sintering were not successful, resulting in low densities and multiphase compacts. Some high-density single-phase compacts could be prepared by sintering above 2150K, but the results could not be reproduced. In addition, it was desirable to lower the sintering temperature to <1875K so that lower temperature hafnium oxide based, current leadouts could be prepared as an integral part of the highly refractory oxide.

A coprecipitation fabrication process is being developed which will yield reproducible, high-density sintered bodies with uniform microstructure and properties. The technique being developed involves the dissolution of rare earth oxide powders in nitric acid with hafnium oxychloride in water, mixing the solutions and coprecipitating using dilute NH40H, filtering, washing, and drying. The salt is calcined to form the oxide. These powders are ball milled, cold pressed, and sintered. The exact conditions for fabricating a wide variety of hafnium-rare earth oxide compositions are being developed. Emphasis is being concentrated on the following compositions which will be tested in the WESTF facility. These include:

- 0.29Pr0<sub>2</sub>·0.05Yb<sub>2</sub>0<sub>3</sub>·0.66Hf0<sub>2</sub>
- 0.06Tb<sub>4</sub>0<sub>7</sub>•0.06Y<sub>2</sub>0<sub>3</sub>•0.88Hf0<sub>2</sub>
- 0.10Er<sub>2</sub>0<sub>3</sub>•0.10Tb<sub>4</sub>0<sub>7</sub>•0.80Hf0<sub>2</sub>

Final details of these methods for each of the above oxides will be reported next quarter.

# 3.3 POST-TEST CHARACTERIZATION OF MgO INSULATING WALL FROM USSR U-02 PHASE III MHD TEST

The MgO insulating wall from the USSR's U- $^{0}$ 2 MHD generator used in the test with US electrode/channel walls $^{(9)}$ 1 was evaluated. The hexagonally shaped blocks removed from the U- $^{0}$ 2 insulating wall were examined by metallographic, SEM, and EDX methods, and the results compared with similar evaluations of a USSR reference untested MgO insulating wall material. $^{(8)}$ 3 The MgO blocks were taken immediately adjacent to cathode 1209 (UC-1209) and 1527 (UC-1527) and anodes 2209 (UA-2209) and 2577 (UA-2527). A small portion of each insulator block used for the examination was covered with the electrode/insulator walls. This created a variation in the surface temperatures and profiles during the test. The cross sections for examination were perpendicular to the plasma flow and to the channel walls.

The top portion of the insulators which was exposed to the plasma, one-third of the area, was coated with a white-pinkish oxide carried downstream by the plasma from upstream components and deposited on the MgO wall (Figure 5.) This deposit was also found on the electrode-insulator surfaces. This once molten coating has been identified as  $\alpha-\text{Al}_2\text{O}_3$  with smaller amounts of Ca stabilized ZrO<sub>2</sub>.

The initially white MgO insulator was colored by the combustion products and  $K_2CO_3$  seed. These colorations formed layers of white, yellow, and brown, generally in that order from the top plasma surface.

The composite, polished cross sections of the MgO insulator pegs exhibited very similar results with a few characteristic features common to the four examined (Figures 6 and 7).

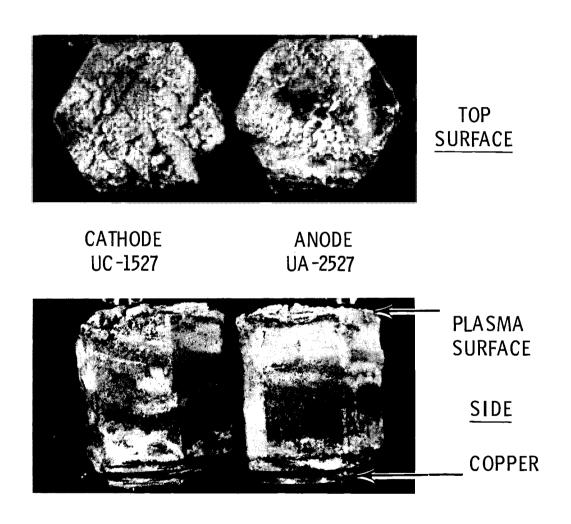
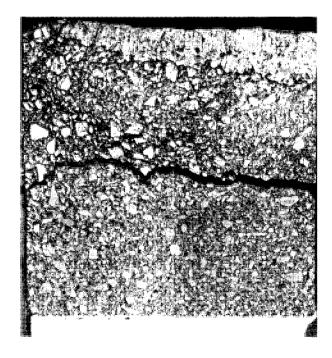
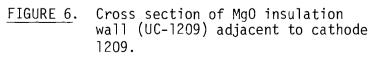


FIGURE 5. Top and side view of MgO blocks from USSR UO-2 Phase III.





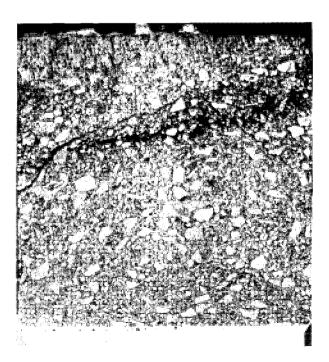


FIGURE 7. Cross section of MgO insulation wall (UC-2209) adjacent to anode 2209.

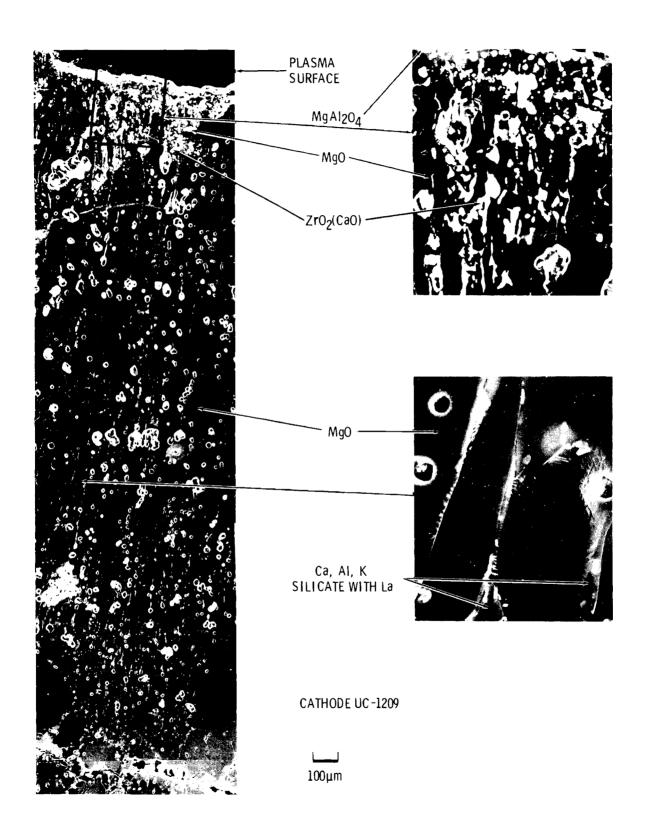
- $\bullet$  The plasma surface was partially coated by the upstream, molten  ${\rm Al_20_3}$  plus  ${\rm Zr0_2}$  .
- $\bullet$  The Al $_2$ O $_3$  coating reacted with the MgO resulting in the formation of reaction layer.
- The high temperature MgO surface below the reaction layer recrystallized forming large columnar grains.
- A crack occurred nearly parallel to the plasma surface, with the distance from the surface varying with the MgO plasma surface temperature. This crack contained  $\rm K_{2}CO_{3}$  which penetrated into the open porosity of the MgO.

The extent of these effects varied depending upon the surface temperature. For example, the thickness of the recrystallized columnar grain layer and the distance from the crack to the surface decreased toward the side of the block covered by the US electrode wall, i.e., to lower surface temperatures. This was especially evident in UC-1209 and UA-2209. Similar effects of lower surface temperatures were observed when comparing the lower temperature UC-1527 and UA-2527 MgO insulator blocks with the higher temperature UC-1209 and UA-2209.

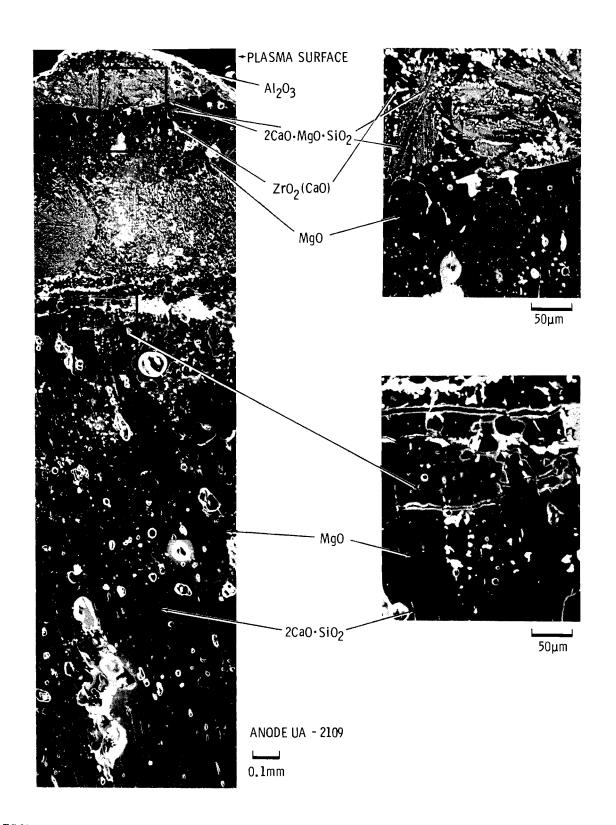
The surface coating on the MgO surface near the entrance to the channel was nearly pure  ${\rm Al}_2{\rm O}_3$  with high porosity and a  ${\rm ZrO}_2({\rm CaO})$  second phase suggesting solidification from a melt. Downstream, the coating also contained increasing amounts of La, Mg, and Zr(Y) from the electrode walls which all were  ${\rm LaCrO}_3$  based materials. This coating was generally retained on the surface as large globules (1-3 mm in diameter), apparently resulting as the molten  ${\rm Al}_2{\rm O}_3{\rm -ZrO}_2$  particles contacted the surface and solidified.

The top surface of the MgO reacted with the coating forming a uniform layer,  $\sim 0.5$  mm thick (Figures 8 and 9). This layer was mostly MgAl $_2$ O $_4$  with some second phase CaO stabilized ZrO $_2$ . It was formed only on the surfaces exposed to the plasma.

Below the reaction layer, the MgO recrystallized extensively forming long columnar grain with lines of pores and second phase stringers at the grain boundaries (Figures 8-9). This recrystallized structure varies up to 3 mm



 $\frac{\text{FIGURE 8.}}{\text{adjacent to cathode 1209 (UC-1209).}} \text{ Microstructure near plasma surface of MgO insulator wall}$ 



thick in the MgO (which was operated at the highest temperatures). The second phase had a composition near CaO·MgO·2SiO<sub>2</sub> with substantial amounts of K, Al, Fe, and La. There is a gradual transition from the columnar grains to large equiaxed grains, then to the original small grain structure which contains K. The formation of the columnar and equiaxed grain structure such as this is generally associated with a significant, high temperatures gradient.

The crack which formed about 4-6 mm below the surface appeared to be the only crack in all the blocks examined. This area was high in potassium which concentrated in the open pores of the MgO structure. The potassium attacks the MgO·CaO·2SiO<sub>2</sub> phase in the small particles which bond the large, dense particles together. The potassium concentration is substantially higher and extends to greater depths below the surface in the MgO blocks adjacent to the cathode wall than in those adjacent to the anode wall. Some growth along the crack did occur suggesting that these fractures were formed near the start of the test and not during shutdown. Generally, below this fracture, the MgO resembles that of the original structure. No microstructural features could be resolved to distinguish between the colored layers.

The tin-lead solder used to bond the MgO to the copper cooling pins was 0.3 mm thick and showed no evidence of degradation.

The performance of the MgO from the USSR UO-2 insulating wall for the Phase III test of a US Channel was good. The molten  $\mathrm{Al}_2\mathrm{O}_3$  from the upstream components of the UO-2 complicated the evaluation since it reacted with the MgO (estimated to be  $\sim 1875\mathrm{K}$ ). Surface temperatures were high enough to cause extensive recrystallization near the surface. The overall resistance to thermal shock was good; however, the formation of a large crack parallel to the surface, with subsequent seed penetration and hydration lead to some surface spalling. The potassium attack on the MgO  $^{\circ}\mathrm{CaO} ^{\circ}\mathrm{2SiO}_2$  phase, which was the binding media between the large, high density grains of the periclase, may be a problem for long-term performance stability.

#### 4.0 WORK ANTICIPATED NEXT MONTH

#### 4.1 ELECTROCHEMICAL TESTING

Electrochemical testing of potential electrode materials is being reduced, and emphasis shifted to fabrication development of electrode materials.

#### 4.2 MATERIALS DEVELOPMENT

The fabrication development of the hafnium-rare earth oxide electrode materials will be accelerated. The goal is to develop fabrication methods which can produce quality electrode bars of sufficient quantity for testing in Westinghouse MHD Test Facility (WESTF). This test (WESTF-42) will evaluate the thermal performance in coal slag and seed without electric current.

#### 4.3 CHARACTERIZATION AND EVALUATION

- 4.3.1 The thermal diffusivity/conductivity of the hafnium-rare earth oxide electrodes will be measured, with data being used for design of WESTF-42. Similar properties will be measured for some  $Y_2O_3$  stabilized  $ZrO_2$  prepared by Westinghouse for testing in WESTF-42.
- 4.3.2. The examination of the RFG refractory from the Montana State University air-preheater test (MSU-#1) will be completed. The results of metallographic, SEM and EDX characterization will be reported and possible corrosion/erosion mechanisms described.
- 4.3.3 The examination of a platinum coated, copper electrode tested in AVCO's Mark VII in coal slag and  $\rm K_2SO_4$  seed will continue.

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