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Characterization and Evaluation of Materials for the US-USSR MHD Program

**Quarterly Report for the Period
October-December 1975**

**Prepared for the Energy Research
and Development Administration
under Contract E(45-1):1830**

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CHARACTERIZATION AND EVALUATION OF MATERIALS
FOR THE US-USSR MHD PROGRAM

Quarterly Report for the Period
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J. Lambert Bates (Principal Investigator)

Battelle
Pacific Northwest Laboratories
Richland, Washington 99352

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J. Lambert Bates (Principal Investigator)
G. B. Mellinger
J. L. Daniel
L. R. Bunnell

ABSTRACT

Characterization of electrode and insulator materials is being performed in support of the International US-USSR MHD Power Generation Program. MgO insulators and five zirconia base electrodes containing CeO_2 and Y_2O_3 additions are being examined before and after testing in the USSR and U-02 MHD generator. Characterization includes thermal diffusivity/conductivity, thermal expansion, composition and microstructure. Preliminary microstructural and scanning microscopy evaluation of the 100 hour Phase I Test in USSR U-02 is described.

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OBJECTIVE

The objective of this program is to characterize and evaluate materials for the International US-USSR MHD Program and to provide technical and scientific support to the MHD materials program.

SCOPE

The scope of this program encompasses three areas:

- Provide technical assistance to MHD Division of ERDA and participating contractors with respect to selection, fabrication, and characterization of materials for testing in USSR U-02 MHD generator.
- Assist in the planning, coordination, and monitoring of screening and channel tests and materials characterization relating to USSR U-02 materials testing.
- Characterize and evaluate the behavior of materials before and after testing in the U-02 MHD generator and in other related screening tests. The characterization includes chemical, structural, and thermophysical property measurements.

TECHNICAL PROGRESS

The study of the materials and channel for the U-02 Phase I test has continued.⁽¹⁾ The pretest characterization of the electrode and insulator materials for the U-02 Phase I test in the USSR was completed, i.e. the composition analyses and the thermal expansion. A cursory evaluation was made of the materials from the four hour Phase I proof test conducted at Battelle-Columbus Laboratories. The evaluation of the US channel from the 100 hour test, U-02 Phase I, has been initiated. The results of the above tests are described in this report.

Precharacterization U-02, Phase I, Materials

Composition: The composition of the MgO was determined by emission spectrochemical analysis to a precision $\pm$ factor of 2. The results

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(1) J. Lambert Bates, "Characterization and Evaluation of Materials for the US-USSR MHD Program, "Quarterly Progress Report for the Period July-September 1975, BNWL-B-463, Oct. 1975.

indicate a high purity oxide containing 20 ppm Al, 50 ppm Ca, 50 ppm Si, and 50 ppm V. All other elements were less than detectable concentrations.

The compositions of the electrode materials were determined by x-ray fluorescence, Table 1. These results are considered accurate to $\pm 5\%$.

TABLE 1
COMPOSITION AND DESIGNATION OF ELECTRODE MATERIALS

Electrode	Reference	Mole Percent			
		<u>ZrO₂</u>	<u>Y₂O₃</u>	<u>CeO₂</u>	<u>Ta₂O₅</u>
0.12 Y ₂ O ₃ ·0.88 ZrO ₂	Y-11, Y-13*	88.8	11.2		
0.18 CeO ₂ ·0.82 ZrO ₂	C-2-13, C-2-15*	77.8		22.2	
0.50 CeO ₂ ·0.50 ZrO ₂	C-3-14, C-3-22*	46.2		53.8	
0.75 CeO ₂ ·0.25 ZrO ₂	C-14-16, C-4-19*	19.6		80.4	
0.78 CeO ₂ ·0.20 ZrO ₂ ·0.02 Ta ₂ O ₅	C-5-9*, C-5-11	16.5		82.2	1.3

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*Analytical samples

Thermal Expansion: The thermal expansions of the MgO insulator and reference electrodes were measured from approximately 60 to 1500°C using an aluminum oxide push rod dilatometer. Measurements were made in both argon and air at atmospheric pressure. The MgO samples were approximately 2.5 cm long and electrodes were approximately 1.9 cm long. Heating and cooling rates were linear with temperature rate changes of 4°C per minute.

The expansion was measured with a transducer with a resolution of $\pm 2.5 \times 10^{-5}$ cm. For MgO, which expands on the order of 2% at 1400°C, the precision of the measurement is of the order of 0.05%. With a similar precision for the measurement of the calibration standard, the overall precision is ~0.1%.

The accuracy of the measurements was determined using a tungsten standard (SRM 737) supplied by the U.S. National Bureau of Standards, which is accurate to $\pm 5\%$. Temperatures were measured with using a platinum versus platinum-10% rhodium thermocouple, which had been calibrated against a standard platinum resistance thermometer. The accuracy of the temperature measurement in the system is within 5°C. The overall accuracy of the thermal expansion data is estimated to be better than 5%.

The thermal expansion data are illustrated in Figures 1 - 6. Differences were observed in the thermal expansions (α) measured in argon and air, with the values of α larger in argon than in air. The magnitude of these differences varied between materials being smallest for the Y₂O₃-ZrO₂ (Y-11) electrode and MgO insulator and greatest for the CeO₂-ZrO₂ electrodes. The samples which showed different expansion in air and argon also experienced changes in sample length during the measurements to 1400-1500°C (Table 2). In general, those samples which had larger α values in argon, also showed the largest increases in length. In addition, the $\Delta L/L$ values for the as-received electrodes were consistently higher during the cooling cycle.

These changes in α during heating in argon suggest possible stoichiometry or phase changes resulting from the lower oxygen pressures. These changes appear to be reversible as seen in the results for MgO, Figure 1, and for the 0.75 CeO₂-0.25 ZrO₂ (C-4-16), Figure 5, in which the thermal expansion returned to the original air values after being first heated in argon and then in air.

The average thermal expansions from 100 to 1400°C calculated from $\Delta L/L$ data are summarized in Table 3. The α values for measurements made in argon are values obtained only on cooling since the values on heating can represent possible changes in sample composition or stoichiometry.

It is interesting to note that for the insulator and all electrodes, the thermal expansion values, $\alpha_{100-1400}$, are larger in argon by a factor of approximately 1:1 than in air. In addition the α values for CeO₂-ZrO₂ electrodes increase with increasing CeO₂ concentration.

Figure 1. Thermal expansion of MgO (M-1).

Figure 2. Thermal Expansion of 0.12 $Y_2O_3 \cdot 0.88 ZrO_2$ (Y-11).

Figure 3. Thermal expansion of $0.18 \text{ CeO}_2 \cdot 0.82 \text{ ZrO}_2$ (C-2-13).

Figure 4. Thermal expansion of $0.5 \text{ CeO}_2 \cdot 0.5 \text{ ZrO}_2$ (C-3-14).

Figure 5. Thermal expansion of $0.75 \text{ CeO}_2 \cdot 0.75 \text{ ZrO}_2$ (C-4-16).

Figure 6. Thermal expansion of $0.80 \text{ CeO}_2 \cdot 0.18 \text{ ZrO}_2 \cdot 0.02 \text{ Ta}_2\text{O}_5$. (C-5-11)

TABLE 2

DIMENSIONAL CHANGES IN THERMAL EXPANSION
OF INSULATOR AND ELECTRODES

Sample	Atmosphere	Length (cm) ^(a)		
		Before	After	Change
MgO (M-1)	Argon ^(b)	2.5334	2.5362	+0.0028
	Argon ^(b)	2.5362	2.5357	-0.0005
	Air ^(b)	2.5359	2.5362	+0.0003
Y-11	Argon	1.8160	1.8763	+0.0003
	Air	1.8628	1.8628	0.0000
C-2-13	Argon	1.9136	1.9131	-0.0005
	Air	1.9167	1.9157	-0.0010
C-3-14	Argon	1.9126	1.9162	+0.0036
	Air	1.9215	1.9210	-0.0005
C-4-16	Argon ^(c)	1.9000	1.9075	+0.0075
	Air ^(c)	1.9075	1.9022	-0.0053
	Air	1.9055	1.9055	0.0000
C-5-11	Argon	1.9126	1.9182	+0.0056
	Air	1.9053	1.9058	+0.0005

(a) Measurements accurate to 0.0005 cm.

(b) All measurements made on same MgO sample in order of their listing.

(c) Both measurements made on same sample, first in argon followed by measurement in air.

TABLE 3

THERMAL EXPANSION OF MgO AND ELECTRODES
(100 to 1500°C)

	<u>$\alpha/^\circ\text{C} (10^{-6}) (100-1500^\circ\text{C})$</u>	
	<u>Argon</u>	<u>Air</u>
MgO (M-1)	15.6 15.5	14.0
0.12 $\text{Y}_2\text{O}_3 \cdot 0.88 \text{ZrO}_2$ (Y-11)	12.3	10.9
0.18 $\text{CeO}_2 \cdot 0.82 \text{ZrO}_2$ (C-2-13)	13.1	11.5
0.5 $\text{CeO}_2 \cdot 0.5 \text{ZrO}_2$ (C-3-14)	13.6	12.2
0.75 $\text{CeO}_2 \cdot 0.25 \text{ZrO}_2$ (C-4-16)	14.5	13.2
0.80 $\text{CeO}_2 \cdot 0.18 \text{ZrO}_2 \cdot 0.02 \text{Ta}_2\text{O}_5$ (C-5-11)	14.9	13.6

Post Characterization of BCL Phase I Proof Test

A proof test of a 10 electrode pair channel was conducted at Battelle Columbus Laboratories to determine material and design performance of Westinghouse's U-02 Phase I, MHD channel.⁽²⁾ The test was performed to simulate the gas and thermal environment in the USSR U-02 MHD channel. The electrodes were electrically driven with an external power supply. The channel operated for a total exposure of approximately four hours (two hours with potassium seed) including one very rapid shutdown (five minutes from maximum temperature to room temperature). Details of this test have been described elsewhere.⁽²⁾

Cross sections of the proof test channel electrodes and insulators were obtained from the National Bureau of Standards. These cross sections were vacuum impregnated in resin and polished for ceramographic examination. All cutting and polishing was conducted in nonaqueous liquids. The microstructures of these electrode pairs are shown in Figures 7 - 11.

-
- (2) W. E. Young, Progress Report for September 1975, "Magneto-hydrodynamic Investigation for Coal Fired Open Cycle Systems," Report No. 25, September 1, 1975 to October 1, 1975, Westinghouse Research Laboratories, Pittsburgh, Pa., October 15, 1975, pp A-1 to A-17.

CATHODE (1207)

2.5 mm

ANODE (2207)

Figure 7. Microstructure of $0.12\text{Y}_2\text{O}_3 \cdot 0.88 \text{ZrO}_2$ Electrode Pair
After BCL Proof Test (as polished)

CATHODE (1309)

2.5 mm

ANODE (2309)

Figure 8. Microstructure of $0.18 \text{ CeO}_2 \cdot 0.82 \text{ ZrO}_2$ Electrode Pair
After BCL Proof Test (as polished)

CATHODE (1311)

2.5 mm

ANODE (2311)

Figure 9. Microstructure of $0.5 \text{ CeO}_2 \cdot 0.5 \text{ ZrO}_2$ Electrode Pair After BCL Proof Test (as polished)²

CATHODE (413)

2.5 mm

ANODE (2413)

Figure 10. Microstructure of 0.75 CeO₂·0.25 ZrO₂ Pair After BCL Proof Test (as polished)

CATHODE (1415)

2.5 mm

ANODE (2415)

Figure 11. Microstructure of $0.80 \text{ CeO}_2 \cdot 0.02 \text{ Ta}_2\text{O}_5 \cdot 0.18 \text{ ZrO}_2$
After BCL Proof Test (as polished)

MgO Insulators: The insulators of all the electrode pairs exhibited a similar appearance after the test. The MgO had cracked, with the cracks appearing to radiate from the corners of the machined cavity adjacent to the bottom of the electrode. The MgO had areas of light brown-grey which appeared concentrated near the above cracks. In general, this coloration extended from these cracks into the colder parts of the MgO insulator.

The surface of the MgO exposed to the plasma had recrystallized. This was more pronounced on the upstream side of the electrode and on the cathodes when compared to the anode of a similar electrode pair. Recrystallization was greatest on the upstream end of the proof test channel.

Electrodes

Electrode Pair: 0.12Y₂O₃·0.88ZrO₂ (1207,2207)* The oxide near the surface of the cathode had recrystallized and exhibited possible evidence of melting on the surface, (Figure 7). The upstream side of the electrodes had eroded to an ~45° angle and this area was black in color, with the darkening extending a short way into the electrode. The cathode appeared light pink in areas and the light grey mottling observed in the reference electrode was not observed. In contrast the anode appeared similar in coloration to the reference material. Cracking and surface changes were less in the anode than in the cathode. The Pt current lead-out had separated from part of the cathode.

Electrode Pair: 0.18CeO₂·0.88ZrO₂ (1309,2309) Extensive porosity and recrystallization was observed near the plasma surface of the cathode and anode with the upstream corner of the electrode eroded with evidence of enhanced crystallization (up to ~0.1 cm deep) and darkening, (Figure 8). The dark mottling observed in the reference and anode was less than that seen in the cathode, especially near the surface. The cathode was not cracked but did exhibit more pullout during surface preparation than the anode. Both anode and cathode separated from the cement. The Pt current lead-out separated from part of the cathode.

Electrode Pair: 0.5CeO₂·0.5ZrO₂ (1311,2311) The surface of both the cathode and anode had recrystallized, with the grains appearing bonded together to a depth approaching 0.17 cm, (Figure 9). This effect was greater in the cathode than in the anode. The upstream corner of the electrodes had eroded to an ~45° angle and was black at the surface with the darkening penetrating into the electrode. The electrodes had experienced some cracking and pullout, being more pronounced in the cathode. The electrodes had separated from the cement with some separation of the platinum.

*Cathode designations are 1000 and Anode 2000.

Electrode Pair: 0.75CeO₂·0.25ZrO₂ (1413,2413) The cathode and anode recrystallized below the surface to a depth for ~0.15 cm into a dense layer surrounding large spherical pores, Figure 10. The surface had the appearance of having possibly been molten. These effects were more pronounced on the cathode. The upstream corners had eroded with a black surface which penetrated into the open porosity of the electrode. Some small cracks and pullout was observed in both electrodes, but pullout was more pronounced in the cathode while cracking was greatest in the anode. The electrode had separated from areas of the platinum and from the cement.

Electrode Pair: 0.80CeO₂·0.02Ta₂O₃·0.18ZrO₂ (1415,2415) Recrystallization was substantially greater near the surfaces of these electrodes than on the other electrode pairs, Figure 11. The recrystallization occurred to depths between 0.15 and 0.20 cm and the oxide nearest the surface were columnar type grains containing large spherical pores. This recrystallized area was nearly black compared to the mottled dark green grains in a yellow-green matrix of the original material. The upstream corners were eroded at ~45 degrees with a black coloration that penetrated a short distance into the electrode. The electrodes were cracked with some very small microcracks in the noncrystallized portions of the electrode. The platinum lead-outs had partially separated from the bottom of the electrodes and from the cement.

Post Characterization of U-02 Phase I

The US MHD Phase I channel was successfully tested for 127 hours (including the 98 hours at operating conditions) in the USSR U-02. After the completion of the test on October 1, 1975, the electrode sections were removed from the U-02, visually examined, and within four hours were placed in plastic bags which were filled with inert gas. The electrode walls were delivered to the National Bureau of Standards where extensive visual and photographic observations were made. The electrode walls were separated into their separate sub-modules, and potted in epoxy resin in a vacuum. Each sub-module was cut in ethylene glycol with a diamond wheel perpendicular to the electrode surface and lengthwise parallel to the plasma flow. This produced cross sections of each electrode-insulator. Three slices, each approximately 0.5 cm thick, were cut from one-half of the electrode length. The outside slices from each sub-module were retained at NBS for phase and chemical composition studies; the inside slices were returned to the USSR for post-test analysis and the center slices were provided to Battelle-Pacific Northwest Laboratory for microstructural scanning microscopy and electron microprobe analyses. The samples were sealed in plastic in inert gas for shipment.

At Battelle-Pacific Northwest Laboratories, the electrode-insulator samples were stored in a vacuum desiccator to prevent moisture attack. The cross sections of the electrode pairs were again impregnated with a fluid resin under vacuum. (The impregnation at NBS had not penetrated

into the smaller pores and cracks.) The mounted sections were ground to expose the electrode and insulator and to prepare the samples for final polishing. All of the above preparations were conducted without use of lubricants. The cross sections were then polished with diamond through a number of steps with a final polishing with MgO in a nonaqueous media. All these steps were required to prevent smearing of potassium, hydration of the potassium carbonate, and to minimize pullout.

The sections are presently being examined with optical and scanning microscopy in conjunction with electron microprobe analysis. Included are the following electrode pairs:

<u>Cathode</u>	<u>Anode</u>	<u>Composition</u>
1103	2103	$0.12\text{Y}_2\text{O}_3 \cdot 0.88\text{ZrO}_2$
1206	2206	$0.18\text{CeO}_2 \cdot 0.82\text{ZrO}_2$
1309	2309	$0.50\text{CeO}_2 \cdot 0.50\text{ZrO}_2$
1414	2414	$0.75\text{CeO}_2 \cdot 0.25\text{ZrO}_2$
1517	2517	$0.80\text{CeO}_2 \cdot 0.02\text{Ta}_2\text{O}_5 \cdot 0.18\text{ZrO}_2$

The following general conclusions were made after a cursory optical microscope evaluation of the above electrode pairs. These conclusions are considered preliminary and will undoubtedly be modified after the structural characterization is complete.

- Extensive breakup and bloating of the cathode and insulator surrounding the cathode occurred on all electrode pairs.
- The cathode degradation was substantially greater than the anode.
- The cement used to hold the electrode in the machined cavity in the MgO appears to act as a source of seed concentration.
- The platinum current leads have separated from the electrodes, especially on the cathodes.
- The fracture of the MgO appears similar for all electrode pairs. The cracks appear to initiate and radiate away from the machined corners which hold the electrodes.
- The surfaces of the MgO and electrode materials appear to be recrystallized.

WORK ANTICIPATED DURING NEXT QUARTER

Composition

The impurity levels of the electrode materials will be determined.

Thermal Diffusivity

The thermal conductivity of the electrode materials will be calculated using the measured thermal diffusivity, measured density and calculated heat capacity using measured low temperature values as a reference.

Post Test Characterization

The detailed microscopic and electron probe examination of the materials from the U-02 Phase I test will continue.

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