HIGH TEMPERATURE PROTECTIVE COATINGS FOR REFRACTORY METALS

by

J. Rexer

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UNION CARBIDE CORPORATION
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I. INTRODUCTION

The research performed under Contract NASw-1405 is a continuation of work initiated under NASA Contract NASw-1030.(1) The major objectives of this contract are 1) to develop procedures for applying protective coatings of iridium on refractory metals, with special emphasis directed towards optimizing the fused salt electrodeposition of iridium and 2) to study the rates of interdiffusion of iridium with the refractory metals tungsten, molybdenum, and niobium. This report summarizes the research effort for the period 21 October 1966 to 21 January 1967. A complete description of the materials used, methods of sample preparation, and the diffusion and electroplating apparatus were given in the previous report for the period 21 August to 21 October 1966.

II. SUMMARY

During the present report period, the polishing and etching techniques needed to reveal the tungsten-iridium reaction zone boundaries were developed. The total reaction zone thickness was measured for the samples previously heat treated. Additional samples were heat treated, and sections of these samples are in the process of being metallographically polished and etched. Two molybdenum-iridium diffusion couples were hot pressed and sectioned for metallographic examination.

Difficulties were still experienced during attempts to electroplate iridium on niobium and tantalum. A flash coating of nickel was deposited on the substrate materials to reduce the chemical activity of these metals in the presence of the molten cyanide electrolyte; the iridium coating was then deposited over the nickel.

Sections of dual-coated samples are being prepared for microscopic examination, and other sections will be heat treated so that the effect of the nickel inner layer on the systems behavior may be determined.

III. PROGRESS

A. Diffusion Studies

1. Tungsten-Iridium System: - Efforts were made to reveal the reaction zone of the tungsten-iridium system by using the polishing and etching techniques of Rapperport and Smith.(2) Their etchant proved to be too severe, resulting in preferential attack of the tungsten and tungsten-rich intermediate phase. An electrolytic color etch was developed which tinted the tungsten rich intermediate phase blue and the iridium-rich phase tan to grayish tan. A description of the polishing and etching procedure follows: Specimens mounted in epoxy resin are given a final polish with one-micron diamond paste on a Texmet polishing cloth. The polished samples are then electrolytically etched for approximately ten seconds (a direct current of four volts is used) in a solution containing 300 ml water, 5 ml ammonium hydroxide, 3 gm sodium hydroxide, and 2 gm chromic acid. The preliminary determination of the total reaction zone thickness was made by direct measurement in the microscope by means of a calibrated eyepiece. The results are given in Table I. Photomicrographs are being taken of all heat-treated samples; additional measurements will be made on these samples. Since the data obtained from these samples proved insufficient to reveal the temperature dependence of the reaction zone growth, additional specimens have been heat treated. Sections of the heat-treated samples are being metallographically polished and etched.

2. Molybdenum-Iridium System: - Two molybdenum-iridium diffusion couples were prepared by first metallographically polishing one side of the one-inch square sheets of 0.020-inch thick molybdenum and 0.005-inch thick iridium. Then, with polished surfaces touching, the molybdenum-iridium couples were hot pressed at 1100°C for 45 minutes at 2700 lb/in^2. Sections of the hot-pressed samples will be heat treated to determine the reaction zone growth rates.

TABLE I

DIFFUSION DATA FOR THE TUNGSTEN-IRIDIUM SYSTEM

Sample No.	Optical Pyrometer Temperature °C	Thermocouple mV	Heat Treat Time, hrs.	Total Reaction Zone, Width, μ
HP1-W*				0
HP5-W*				5.6
HP6-W*				5.6
HP7-W*				7.0
HP1-W1300-1	1295 ± 10	22.65 ± 0.01	4.0	4.2
HP1-W1300-2	1295 ± 10	22.65 ± 0.01	16.0	5.6
HP1-W1530-1	1538 ± 10	26.58 ± 0.02	2.0	7.0
HP1-W1530-2	1538 ± 10	26.58 ± 0.02	4.0	9.8
HP1-W1530-3	1538 ± 10	26.58 ± 0.02	8.4	12.6
HP1-W1530-4	1538 ± 10	26.58 ± 0.02	15.8	14.0
HP1-W1530-5	1538 ± 10	26.58 ± 0.02	32.2	21.0
HP5-W1710-1	1705 ± 10	29.15 ± 0.03	2.0	11.2
HP5-W1710-2	1705 ± 10	29.15 ± 0.03	4.0	14.0
HP5-W1710-3	1705 ± 10	29.15 ± 0.03	8.27	18.2
HP5-W1710-4	1705 ± 10	29.15 ± 0.03	16.1	23.8
HP5-W1900-1	1905 ± 10	31.94 ± 0.03	2.0	16.8
HP6-W1900-2	1905 ± 10	31.94 ± 0.03	4.0	19.6
HP6-W1900-3	1905 ± 10	31.94 ± 0.03	7.9	28.0
HP6-W1900-4	1905 ± 10	31.94 ± 0.03	16.3	33.6
HP7-W2125-3	2127 ± 10	34.40 ± 0.02	2.0	16.8

* Sections of the hot-pressed samples

B. Fused-Salt Electroplating of Iridium

Difficulties were still experienced during attempts to electroplate iridium on niobium and tantalum. Failure to obtain coherent and adherent iridium deposits on these substrate metals was not attributed to electrolyte contamination, since the operation of each salt bath was intermittently checked by electroplating onto a molybdenum substrate. The plating difficulties may be due to the ease with which niobium and tantalum react with the molten electrolyte. A flash coating of nickel was deposited on the substrate materials to reduce the chemical activity of these metals in the presence of the molten cyanide electrolyte; the iridium coating was then deposited over the nickel. Visual examination of sections of the dual-coated samples indicates that both coatings are well bonded. These sections are presently being prepared for microscopic examination. Other sections will be heat treated to determine the effect of the nickel inner layer on the behavior of the various systems.

REFERENCES

1. Criscione, J.M., Rexer, J., and Fenish, R.G., "High Temperature Protective Coatings for Refractory Metals," under Contract NASw-1030.
2. Rapperport, E.J., and Smith, M.E., Technical Documentary Report No. WADD-TR-60-132, Part II, September 1962.

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