HIGH TEMPERATURE PROTECTIVE COATINGS FOR REFRACTORY METALS

by

J. Rexer

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for

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I. INTRODUCTION

This research, performed under Contract NASw-1405, is a continuation of work initiated under Contract NASw-1030[1]. After the first year, Contract NASw-1405 was extended to April 21, 1968 with slight modifications in the program objectives. The major objectives of the present program are 1) to measure the change in thickness of an iridium coating on the refractory metals tungsten, molybdenum, and niobium due to interdiffusion and intermetallic compound formation and 2) to examine microscopically with a microbend tester, the mechanical behavior of iridium-coated and heat-treated specimens.

The effort during the first year under Contract NASw-1405 indicated that sufficient work had been performed on the fused salt electrodeposition of iridium to evaluate its potential as a method of cladding refractory metals. The plating parameters and apparatus made possible the consistent fused salt electrodeposition of coherent and adherent iridium coatings on tungsten and molybdenum. Since niobium and tantalum react chemically with the fused salt electrolyte, these metals could not be consistently electroplated with an adherent iridium coating without the prior application of an unreactive intermediate coating such as nickel. The amount of iridium depleted through intermetallic compound formation can best be determined directly from measurements of the thickness of iridium remaining after annealing rather than from measurements of changes in the thickness of the total reaction zone. Although some success was achieved in developing the polishing and etching techniques needed to reveal the reaction zone interfacial boundaries, the total reaction zone thickness is still difficult to determine because of irregularities in these boundaries. The changes in the program objectives were a direct consequence of the above findings.

This report discusses the research effort for the period 21 April 1967 to 21 July 1967.

II. SUMMARY

~~During the present report period~~, a spring-loaded microbend tester was designed and is being constructed. The microbend tester will be mounted on a microscope platform permitting side-of-beam observations of metallographically polished surfaces during deformation. The applied load will be measured by means of a spring-loaded balance having a marker recording the maximum applied load. Flexural strengths will be calculated from the data obtained with iridium coated and heat-treated samples.

A previous malfunction of the temperature control unit resulted in extensive damage to the heat-treating apparatus. This apparatus and the control unit have been repaired. The best control settings for the controller were established permitting set-point control of the temperature of the tungsten crucible within the range 1100° to 2100°C. In addition, a calibration curve was obtained for the new thermocouple, and the radial temperature distribution within the tungsten crucible was established at various temperature settings.

III. PROGRESS

A. Composites Mechanical Behavior

A microbend tester was constructed under Contract No. NASw-1030[1] so that we might obtain more knowledge concerning the mechanical behavior of iridium-coated and heat-treated composites; the basic design of the microbend tester follows that proposed by Flinn and Trojan[2]. The microbend tester permitted microscopic observation of the side of the beam while a bending stress was applied. Three-point loading was accomplished by turning a thumbscrew attached to two outer loading pins while the center pin was held stationary. Consequently, neither the load being applied nor the maximum load at fracture could be measured. For the present program, a similar device is being constructed which will permit the load to be applied to two outer loading pins through the plunger of a spring balance. The spring balance is calibrated to within 1/8 lb. and has a marker to measure the maximum applied load. In other respects, the new microbend tester is not significantly different from the previous one.

B. The Effect of Heat Treatment on Coating Thickness

The apparatus for heat treating iridium coated samples and the electronic temperature controller have been repaired. The various adjustments of the controller were systematically tuned for set-point control of the temperature of the tungsten crucible. Optimum control settings were established for temperature variations of five degrees of the desired temperatures, at 100°C intervals, within the range 1100° to 2100°C. A W-5% R/W-26% Re thermocouple, positioned inside of the tungsten crucible, was calibrated against a calibrated micro-optical pyrometer. The calibration curve is given in Figure 1. The temperature plotted is the average of five determinations at each thermocouple output setting. In addition, the radial temperature distribution within the tungsten crucible was established at several temperatures through the use of a crucible lid containing five 1/16 inch diameter holes. In the accompanying table, hole number one was located in the center of the lid and the remaining four holes were uniformly distributed between the inner crucible surface and the center hole. No temperature gradients which could be determined by the optical pyrometer were observed.

TABLE I

Temperature Gradients Within the Tungsten Crucible

Temp. at * Hole #1, °C	Temp. at Hole #2, °C	Temp. at Hole #3, °C	Temp. at Hole #4, °C	Temp. at Hole #5, °C	Ave. Temp. °C
1288 ± 10	1278 ± 10	1278 ± 10	1288 ± 10	1280 ± 10	1282
1497 ± 10	1491 ± 10	1500 ± 10	1503 ± 10	1497 ± 10	1498
1683 ± 12	1675 ± 12	1700 ± 12	1696 ± 12	1689 ± 12	1689
1900 ± 15	1885 ± 15	1870 ± 15	1900 ± 15	1879 ± 15	1887

* Each recorded temperature is the average of 10 trials.

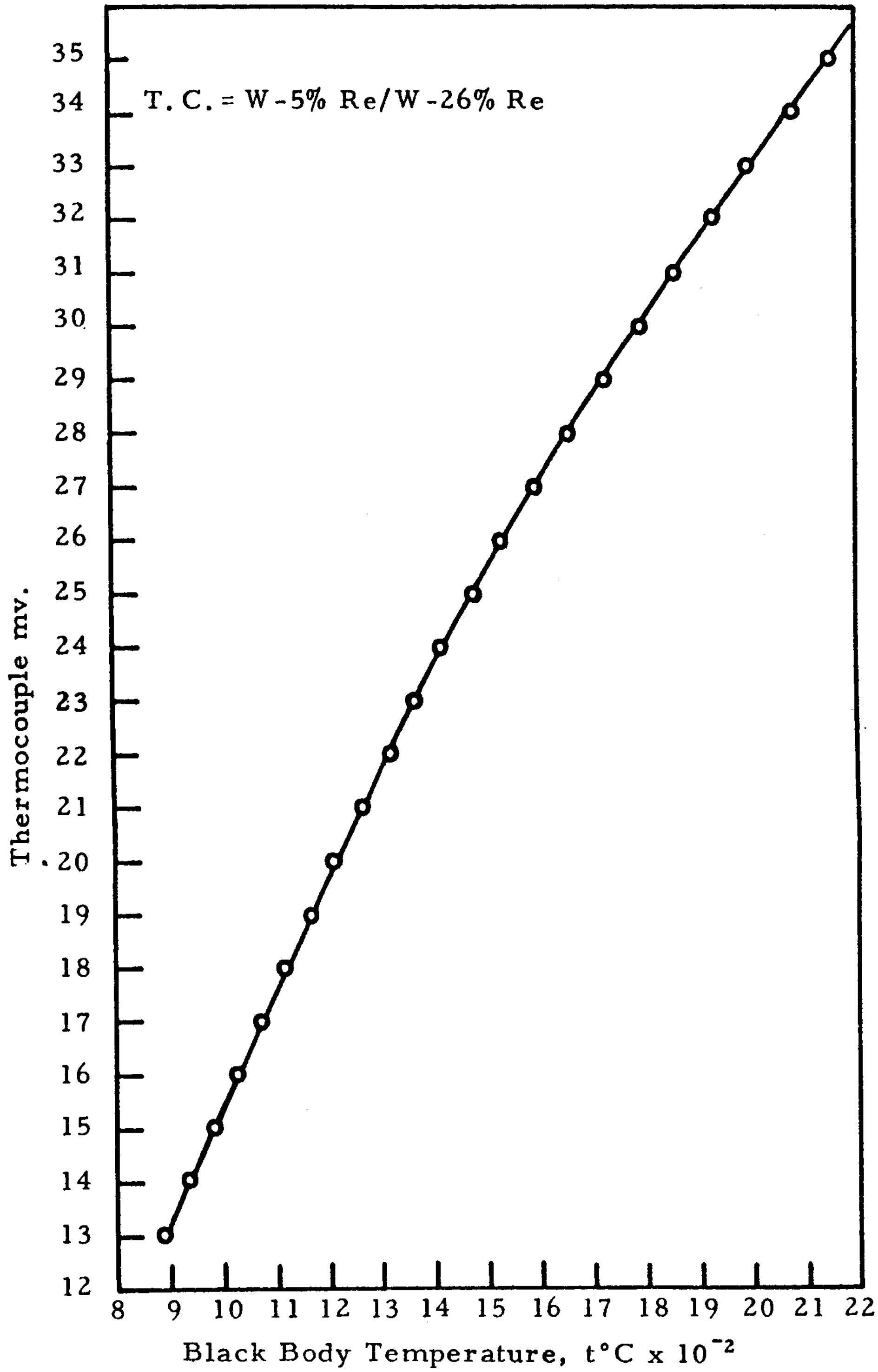

Figure 1. Thermocouple Calibration Curve

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1. Criscione, J.M., Rexer, J., and Fenish, R.G., "High Temperature Protective Coatings for Refractory Metals", under Contract NASw-1030.

2. Flinn, R.A., and Trojan, P.K., "Examination of Microstructure Under Varying Stress", Metal Progress, July, 1955.

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