HIGH TEMPERATURE PROTECTIVE COATINGS FOR REFRACTORY METALS

by

J. Rexer

PROGRESS REPORT NO. 6

Prepared Under Contract No. NASw-1405

UNION CARBIDE CORPORATION
CARBON PRODUCTS DIVISION
PARMA, OHIO

for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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I. INTRODUCTION

This research, performed under Contract NASw-1405, is a continuation of work initiated under Contract NASw-1030[1]. 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.

This report summarizes the research effort for the period 21 July 1967 to 21 October 1967. Complete descriptions of the materials used, methods of sample preparation, and apparatus were given in previous quarterly reports.

II. SUMMARY

During the present report period, construction of the microbend tester was completed and the performance of the device was checked using small strips of steel. All modifications that were deemed necessary have been completed. The flexural strength values obtained for Union Carbide Corporation grade ATJ graphite were compared with the values obtained on standard size samples by our Physical Testing Group. Tests were also conducted on polished strips of as-received molybdenum and tungsten and heat treated specimens of the same material. Two molybdenum-iridium diffusion samples were hot-pressed.

III. PROGRESS

A. Composites Mechanical Behavior

Construction of the microbend tester, shown in Figure 1, was completed. Twelve samples of Union Carbide Corporation Grade ATJ graphite were tested using the microbend tester, and eleven samples, machined from the same large piece of ATJ graphite, were tested by a standard three point loading technique by our Physical Testing Group. The results are given in Table I. The average flexural strength determined using the microbend tester, 5500 psi., compared well with the average strength of 6000 psi obtained by physical testing. The maximum deviation from the average strength for the subsize samples was ± 1000 psi, whereas that resulting from samples which were physically tested was ± 2200 psi. The difference between the two test values may be caused primarily by differences in instrumentation. The standard test can determine the applied load only within ± one pound, whereas the spring balance of the microbend tester can be read within ± 1/8 pound.

Subsize specimens of tungsten and molybdenum were examined with the microbend tester. The results are given in Table II. Strips of these metals, approximately one inch long, were cut from the as-received sheet with a water cooled abrasive wheel. One surface of each strip was polished and etched to reveal the grain structure. Two of the tungsten samples failed in shear (delaminated) at strengths of 362,000 and 345,000 psi; in a third sample, the load reached a maximum value of 11.25 lbs (corresponding to a flexural strength of 264,000 psi) and then decreased with no observable indications of either a shear or tensile break. One tungsten specimen was heated at 1500°C for 1-1/2 hours under vacuum, furnace cooled, and tested. This sample fractured at a breaking strength of 190,000 psi.

The four molybdenum specimens which were not heat treated prior to testing did not fracture. However, with all samples, the applied load reached a maximum, corresponding to strengths between 184,000 and 208,000 psi, and then decreased as bending was continued. The molybdenum specimen heat treated at 1550°C for one hour was bent to 90 degrees with a maximum applied load of 4 lbs. (maximum strength of 104,000 psi).

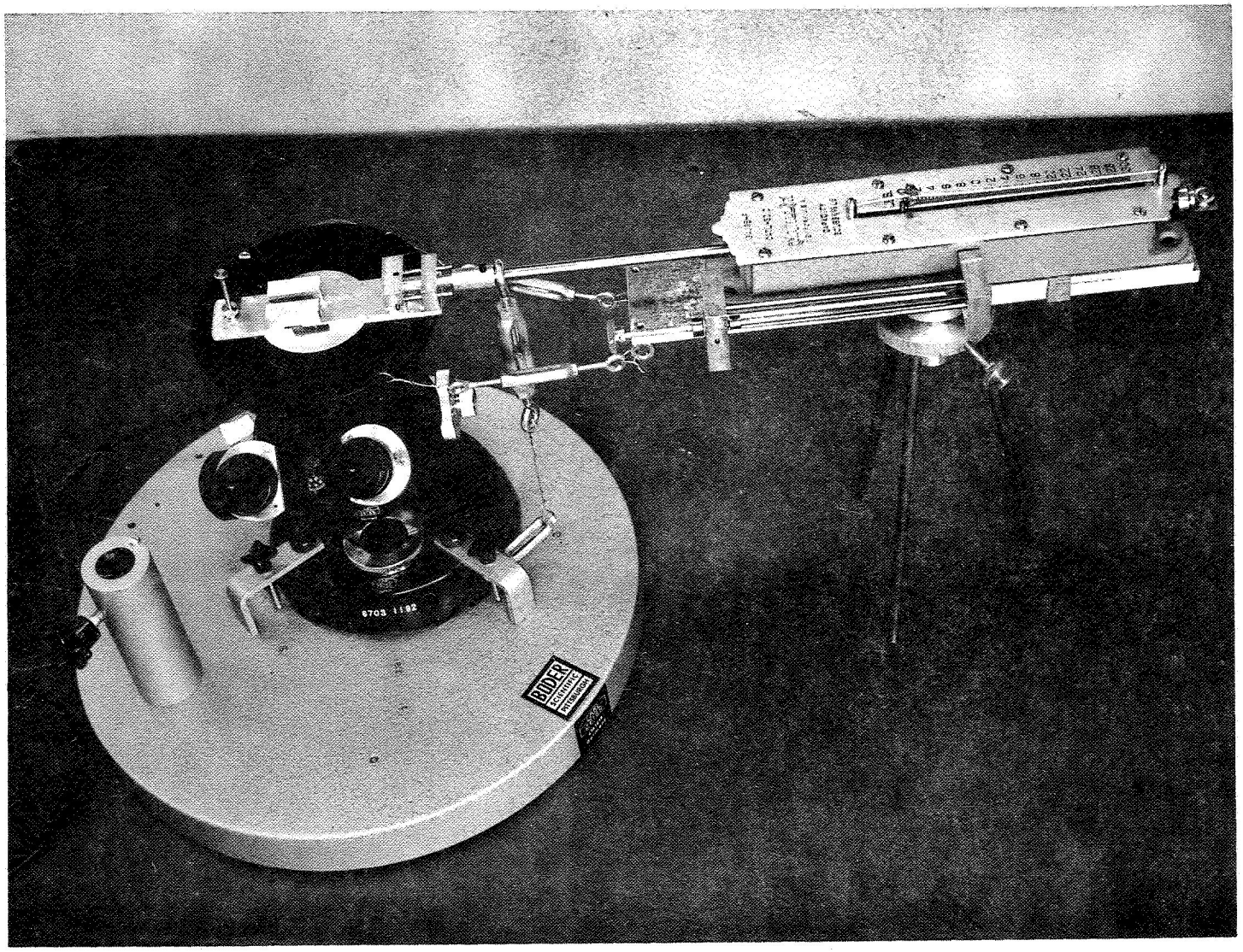

Figure 1. Photograph of Microbend Tester Mounted on Microscope

N-18168

TABLE I

Flexural Test Results for ATJ Graphite

MICROBEND TESTER RESULTS					PHYSICAL TESTING GROUP RESULTS				
Sample	Width Inches	Thickness Inches	Maximum Load, lb.	Strength psi x 10^3	Sample	Width Inches	Thickness Inches	Maximum Load, lb.	Strength psi x 10^3
1	0.0994	0.1005	6-3/4	6.3	1	0.102	0.102	6	5.5
2	0.1009	0.1010	5-1/2	5.2	2	0.100	0.100	6	5.6
3	0.0997	0.1002	6-7/8	6.5	3	0.102	0.099	8	7.3
4	0.1007	0.1012	6-1/4	5.9	4	0.100	0.100	5	4.7
5	0.0955	0.0983	4-1/4	4.5	5	0.101	0.101	9	8.3
6	0.0992	0.1007	5-3/4	5.3	6	0.100	0.100	8	7.5
7	0.1000	0.1000	6	5.6	7	0.099	0.099	6	5.7
8	0.0990	0.0995	5-1/2	5.2	8	0.098	0.100	7	6.7
9	0.0986	0.0990	5-1/2	5.2	9	0.100	0.100	6	5.6
10	0.0996	0.1010	5-3/4	5.3	10	0.100	0.100	7	6.5
11	0.0995	0.1006	5-1/4	4.9	11	0.096	0.100	4	3.9
12	0.0996	0.1003	6-1/2	6.1					
			Average =	5.5				Average =	6.0

TABLE II

Flexural Test Results for Tungsten and Molybdenum

Trial*	Sample	Width, Inches	Thickness Inches	Maximum Applied Load, Lbs.	Flexural Strength psi x 10^5	Comments
1-W	Tungsten	0.1081	0.0216	17	3.62	Failed in shear
2-W	"	0.1092	0.0222	16-1/4	3.45	Failed in shear
3-W	"	0.0981	0.0220	11-1/4	2.64	Load dropped off. No break.
4-W	"	0.0832	0.0220	6-1/2	1.90	Heat treated at 1500°C for 1-1/2 hrs. Clean break.
1-M	Molybdenum	0.1188	0.0215	9-1/2	1.84	Load dropped off. No break.
2-M	"	0.0982	0.0210	4	1.04	Heat treated at 1550°C for 1 hr. No break, 90 degree bend.
3-M	"	0.1128	0.0222	8-1/2	1.70	Load dropped off. No break.
4-M	"	0.1046	0.0212	8-1/8	2.08	" " " " "
5-M	"	0.1014	0.0210	8	1.86	" " " " "

* All samples were tested with a fixture having a 0.75 inch span and three-point loading.

Few data are available on the flexural strengths of either tungsten or molybdenum; consequently, our results cannot be compared with any reference standards. The principal objective of our microbend test measurements on tungsten and molybdenum sheet was to generate these reference values in order to evaluate the effect of iridium coatings on the base metals.

B. The Effect of Heat Treatment on Coating Thickness

Two molybdenum-iridium couples were prepared by hot-pressing sheets of these metals together at approximately 1100°C and 2700 psi for 1-1/2 hours. The bimetallic couples will be sectioned into strips approximately 1/8 inch wide by 3/4 inch long. Some of the strips will be flexed, using the microbend tester, without further heat treatment; others will be heat treated to develop the reaction zone and then flexed.

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REFERENCE

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

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