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High-Temperature Evaluation of Mo-Tm₂O₃ Cermet

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Edward S. Keddy

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HIGH-TEMPERATURE EVALUATION OF Mo-Tm₂O₃ CERMET

by

Edward S. Keddy

ABSTRACT

Molybdenum and tungsten-coated 60:40 v/o Mo-Tm₂O₃ cermet samples, evaluated at 1700°C for 1000 h, displayed no dimensional changes and suffered only minor weight losses. These losses, which are due to Tm₂O₃ surface evaporation, might be prevented by thicker coatings.

INTRODUCTION

A 60:40 v/o Mo-Tm₂O₃ cermet was evaluated at 1700°C for 1000 h to determine the high-temperature characteristics of the cermet and thus to establish criteria for future testing and application. Of particular interest were dimensional changes, mass loss by migration and evaporation, and structural integrity during thermal cycling. In addition, various fabrication techniques were checked out to establish the best method of construction.

CONSTRUCTION OF TEST SAMPLES

The test samples were fabricated into short cylinders by pressing and sintering a mixture of powders, consisting of 64.02 w/o Mo and 35.98 w/o Tm₂O₃, into 60:40 v/o Mo-Tm₂O₃ cermets of nominally 0.250 in. o.d. with a length of ~ 1.0 in. Both ends of the cylindrical samples were rounded on the edges to a radius of 0.0625 in. A 0.125-in.-diam hole was eloxed at one end to a depth of 0.313 in. to accommodate a 0.0625-in.-diam molybdenum stem for handling and mounting the samples in the test apparatus. Instead of being brazed into the hole, the stem was slip-fitted in place and was held by a vapor-deposited coating.

Three samples (1M, 2M, and 3M) were coated with molybdenum by vapor-deposition to a nominal thickness of 0.003 in; and three samples (8W, 9W, and 10W) were coated similarly with tungsten.

TEST ENVIRONMENT

A high-frequency induction system was selected to evaluate these cermets. By using a specially designed concentrator with seven separate heating zones within the same field, as many as seven cermet pins could be evaluated simultaneously. The samples were under continuous observation through the top of the concentrator. Operating temperatures were adjusted and set by use of an optical pyrometer. The cermet pins were held in place by their stems which extended out of the heating zone into a support stand. An ion pump was used in the vacuum system and maintained a pressure of ~ 10⁻⁶ mm Hg throughout the evaluation.

METHOD OF EVALUATION

Molybdenum-Coated Samples

The first test involved the three molybdenum-coated samples. Sample 1M was polished to a bright finish prior to high-firing but lost the stem during this procedure. In addition, the coating on one of the rounded ends was completely polished off. Sample 2M was therefore polished with special care; however, the stem became separated again and the rounded edges lost some of the molybdenum coating. Sample 3M was also polished to a bright finish, but extreme care was taken to leave the rounded corners unpolished and thus to retain the coating. The three samples were placed into the induction-heating system and were fired to 1700°C (corrected).

The first firing cycle, planned for 100 h, was interrupted after 88 h because a heavy residue had collected on the cold wall of the heating zone by that time. This residue consisted mainly of thulium; it was heavily concentrated in areas where the coating on the pins had been removed during polishing. The samples were cycled four more times intentionally for weight and dimensional measurements, and were cycled at least six more times due to maintenance problems with parts associated with the vacuum system. The samples remained in the induction-heating system throughout this time.

The molybdenum coating began to spall after 125 h at 1700°C, which made accurate dimensional measurements almost impossible because the change, if there was one, remained within the 0.001 in. of the actual coating thickness. Any drastic dimensional change could still have been detected, but there was none on the three molybdenum-coated samples.

Samples 1M and 2M were submitted, respectively, to chemical analysis and metallographic examination for comparison with an as-pressed and sintered standard taken from the original batch of pressings.

Because of evaporation losses suffered by the first three samples, the second batch of cermet specimens was coated with tungsten to a thickness of 0.003 in. At this time it was not known whether the loss could be ascribed to the fact that the vapor pressure of molybdenum at 1700°C is on the order of 10^{-7} mm Hg. It was thought that the tungsten coating with a vapor pressure of $< 10^{-8}$ mm Hg at 1700°C might restrict the evaporation loss rate of the cermet if the loss was indeed mainly molybdenum.

Tungsten-Coated Samples

Samples 8W, 9W, and 10W were introduced into the test system after the first three molybdenum-coated samples had been running for 125 h at temperature. Sample 8W had been polished with great care to avoid breaks in the coating. However, microscopic examination revealed small voids that could have penetrated to the cermet substrate, especially at the rounded edges of the sample. Samples 9W and 10W were also polished but the rounded edges were not touched.

Microscopic examination during the second test cycle, after 113 h, indicated that all three tungsten-coated samples had voids in the rounded edges as well as along the sides of the overcoating. The samples were returned to the induction system and the evaluation was continued: they were thermally cycled and their weight and dimensions were periodically measured. After 1000 h at 1700°C, these samples were compared to the first three units as to weight loss and dimensional change. The tungsten coating had not spalled.

DISCUSSION OF RESULTS

Dimensional Changes

Measurements on the diameter of the cylindrical samples were taken periodically throughout the heating cycle. No overall dimensional change was detected on Samples 1M, 2M, and 3M although some loss in cermet diameter might have occurred, considering that the coating had a maximum thickness of 0.005 in. and accurate measurements were difficult. An inspired guess would place the average loss in cermet diameter at -0.001 in. Samples 8W, 9W, and 10W did not indicate any dimensional change at 1700°C. -- For the purpose of this evaluation a dimensional change of ± 0.0005 in. on the average is considered nondetectable because of instrumentation limitations; the extremes of the original average were in this range.

Weight Loss by Evaporation

The weight losses of each sample after 1000 h at 1700°C are presented in Table I.

TABLE I. WEIGHT LOSS COMPARISON

Sample No.	Weight, g		Weight Loss, g
	Original	Final	
1M	6.9504	6.8263	0.1241
2M	6.8727	6.7945	0.0782
3M	8.0828	7.9765	0.1063
8W	9.4349	9.3863	0.0986
9W	9.6656	9.5676	0.0980
10W	9.7284	9.6295	0.1012

Note that the weight losses of the samples during the 1700°C evaluation amounted to ~ 0.100 g, regardless whether the samples had been coated with

molybdenum or tungsten. All coatings had a thickness of 0.003 in. and all contained voids.

Chemical Analysis

A sample from the molybdenum-coated batch and a sample from the tungsten-coated batch were analyzed to determine their composition by weight of thulium and molybdenum. Comparative data are presented in Table II.

TABLE II. CHEMICAL ANALYSIS OF 60:40 v/o Mo-Tm₂O₃

	Content, w/o	
	Molybdenum	Thulium
Theoretical Comp.	64.02	35.98
Standard	64.01	34.7
Sample 1M	63.2	29.3
Sample 9W	68.1	27.3

The data thus far show a higher loss of Tm₂O₃ than of molybdenum. This would indicate that the vapor pressure of Tm₂O₃ exceeded that of molybdenum at 1700°C.

Metallographic Analysis

Sample 1M was sectioned and compared for metallographic analysis (Fig. 1). No basic structural change in the composite was detected when compared to the as-received cermet. Only some surface depletion of Tm₂O₃ was noted, which would account for the weight loss.

CONCLUSIONS

The following conclusions are drawn from these experiments:

- Increasing the operating temperature beyond 1700°C would increase the weight loss for samples with 0.003-in.-thick coatings.
- The loss of Tm₂O₃ from the surface of the cermet indicates that the vapor pressure of Tm₂O₃ exceeds that of molybdenum at this temperature.
- The weight loss of the cermet could possibly be restricted by an overcoating which would prevent the Tm₂O₃ from evaporating from the surface; this surface evaporation would otherwise create voids through which the remaining Tm₂O₃ could migrate out of the solution. Thicker coatings might be considered.

100X

500X

Fig. 1. Sections of molybdenum-coated Sample 1M after 1000 h at 1700°C in vacuo.