QUARTERLY REPORT

# THE PEST PHENOMENON IN INTERMETALLICS

NASW-1403

by

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## Introduction and Summary

Under NASW-1167 we established that single crystals of molybdenum disilicide are subject to delayed failure or static fatigue when subjected to a bending stress at 500°C in air.[1] The fracture surfaces observed after failure are covered with the powdery yellow oxide, a mixture of $SiO_2$ and $MoO_3$, that has come to be associated with pest failure in polycrystals.[2]

Previous results were interpreted on the basis of the Charles-Hillig theory for static fatigue in brittle materials. Reasonable values of the parameters involved, the activation volume for pest failure and the interfacial energy between $MoSi_2$ and the pest oxide, were obtained. However, the data were of low precision due to the large variation in the base level strengths of the single crystal specimens tested. Base level strengths of nine single crystal samples of $MoSi_2$ in 4-point bending at 510°C in vacuum varied from 4,230 psi to 147,500 psi. A weighted average of the nine values, 53,000 $\pm$ 29,000 psi, was used in analyzing the delayed failure results obtained in air.

In order to correlate delayed failure measurements in the pest range with classical chemical kinetic measurements, static fatigue curves must be obtained as a function of temperature. It seemed clear that variations in base level strength of the kind described above could completely mask the temperature affects we are looking for. We therefore devoted the first quarter of this year to preparing enough single crystal molybdenum disilicide of well-controlled base level strength for all of the planned experiments. By careful attention to the materials preparation parameters, we have succeeded in obtaining a number of single crystal boules of molybdenum disilicide sufficient for at least 50 static fatigue measurements. The base level strength measured on four (4) samples cut at random was found to be 51,000 $\pm$ 3500 psi.

Work has continued on simultaneous weight-change-oxygen consumption measurements to obtain the kinetic data that we will hopefully be able to correlate with static fatigue measurements.

## EXPERIMENTAL

### Material Preparation

As in previous work, single crystal rods of molybdenum disilicide were prepared by zone melting of sintered compacts. The starting material was 200 mesh molybdenum disilicide of 99.5% purity purchased from Sylvania. This was mixed with 4% by weight of excess silicon to compensate for evaporation during synthesis.

Improvements were made in several aspects of the materials preparation technique. The powder is now sintered to about 85% of theoretical density in a boron nitride die in contrast to 60-70% in previous work. The die is heated to 1300°C for 3 hours in a flowing argon atmosphere. The sintered rod, about 7 inches long and 3/8 inch in diameter, is then zone melted in a flowing 95% argon--5% hydrogen atmosphere, by passing a slowly moving induction coil up along its length. In order to avoid thermal cracking, a split molybdenum radiation shield which is inserted into a ceramic tube moves along the length of the single crystal behind the induction coil. Further improvement was effected by seeding the crystals of molybdenum disilicide with a single crystal seed which is free from macrocracks.

Bar specimens 1/2 - 3/4 inch x 1/8 x 1/16 were cut from the zone melted rods for the bend tests. The bar specimens were ground to size on a 320 grit diamond wheel, then lapped and polished on 600 grit silicon carbide and 6 micron diamond, respectively. The polished specimens were then examined for macrocracks under an optical microscope.

### Base Level Strength Test

The rectangular bar specimens discussed above were tested in bending under four-point loading. The bend test apparatus consists of a 304 stainless steel jig with aluminum oxide (Lucalox) knife edges and 1/8 inch between the upper knife edges. The jig was mounted in an Instron and enclosed in a tungsten resistance furnace.

Bend strengths were obtained on four bar specimens obtained from a single crystal rod, designated #1052. The strength measurements were made at 500°C and a pressure of $2 \times 10^{-5}$ torr. The crosshead speed was 0.05 cm/min. Results are summarized in Table I.

TABLE I

Base Level Strengths of Single Crystal $MoSi_2$

| Sample No. | (psi) |
|---|---|
| 1052-M-1 | 47,000 |
| 1052-M-2 | 50,200 |
| 1052-M-3 | 56,700 |
| 1052-T-1 | 50,000 |

Plans for the Next Quarter

Continuation of the present work will be in the direction of obtaining fatigue curves based on the recently formed single crystals of molybdenum disilicide. For a specified temperature, both the bars required for delayed fracture and those required for the base level strengths will be obtained from the same single crystal boule. The temperatures to be investigated will be within and above the pesting range.

(1) Final Report, The Pest Phenomenon in Intermetallics, NASW-1167, May, 1966. A. D. Little, Inc.

(2) R. W. Bartlett and P. R. Gage, ASD-TDR-63-753, Part II, July 1964.