

Measurement of Dynamic Young's Modulus for Molybdenum Disilicide/Pentatitanium Trisilicide

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The PUCOT (piezoelectric ultrasonic composite oscillator technique) has been used to measure the temperature dependence of dynamic Young's modulus of molybdenum disilicide ($MoSi_2$) reinforced with 10, 30, and 50% volume fraction of pentatitanium trisilicide (Ti_5Si_3). The temperature ranges were room temperature to 600 °C for the 30 and 50% specimens, and room temperature to 880 °C for the 10% specimens. In all cases, the values of Young's modulus decreased linearly with increasing temperature, although at different rates. A graph of Young's modulus versus composition was used to estimate the value of Young's modulus for $MoSi_2$. The value obtained (385 GPa) was in excellent agreement with the value of 388 GPa quoted in the literature.

Keywords

ceramics, composites, elastic, intermetallics, molybdenum disilicide, pentatitanium trisilicide, temperature, ultrasonics, Young's modulus

1. Introduction

MOLYBDENUM disilicide ($MoSi_2$) is an intermetallic compound that possesses a melting point of 2030 °C, exhibits excellent high-temperature oxidation resistance, and has a brittle-to-ductile transition in the range of 900 to 1000 °C (Ref 1). On its own, $MoSi_2$ does not have sufficient mechanical properties to make it a viable structural material for elevated-temperature applications. In the 1970s, Fitzer et al. (Ref 2) began examining $MoSi_2$ composites reinforced with Al_2O_3, SiC, and niobium wire as a way to improve the mechanical properties of $MoSi_2$.

The present study is concerned with the measurement of dynamic Young's modulus as a function of temperature using an ultrasonic technique on composite specimens consisting of $MoSi_2$ reinforced with up to 50% volume fraction of pentatitanium trisilicide (Ti_5Si_3). The chief advantage of the technique is that the size of the specimens is small (25 mm long). Young's modulus, the ratio of stress to strain in the elastic regime, is of obvious importance in design.

2. Experimental Procedure

2.1 *Specimen Preparation*

Specimens were prepared with 10, 30, and 50% volume fraction of Ti_5Si_3. Cerac-325 mesh Ti_5Si_3 and Alfa-325 mesh $MoSi_2$ powders were mechanically blended in the proper amounts and consolidated by hot pressing at a maximum temperature of 1850 °C. The final specimen size was 1.5 by 2.0 by 25 mm.

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2.2 *Metallographic Examination*

Small pieces of the specimens were mounted in Bakelite (Union Carbide Corp., Danbury, CT) and then polished using cloth polishing disks and various grades of diamond paste. Unetched specimens were examined in an optical microscope, and micrographs were taken at a magnification of 1200×.

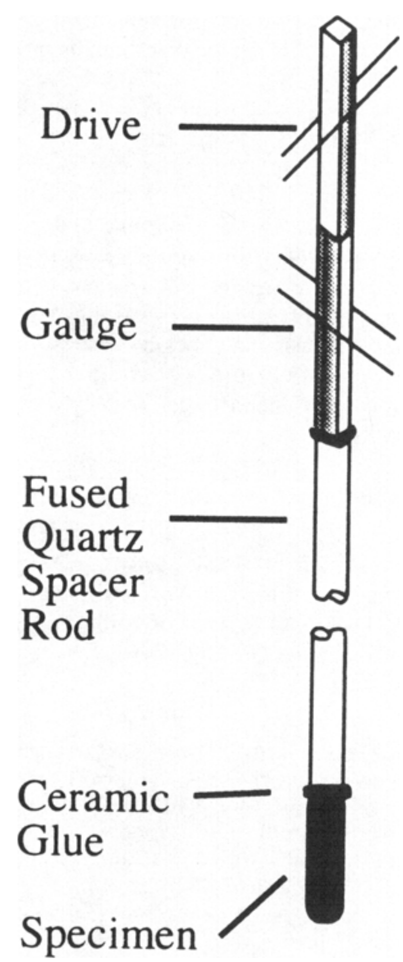


Fig. 1 Four-component PUCOT system

2.3 *Young's Modulus Measurements*

Measurements of dynamic Young's modulus were made at frequencies near 150 kHz using the PUCOT (piezoelectric ultrasonic composite oscillator technique) (Ref 3-5). High-temperature testing followed the guidelines of Harmouche and Wolfenden (Ref 6).

A schematic drawing of the PUCOT is shown in Fig. 1. The assembly consisted of two α-quartz piezoelectric crystals (drive D and gage G) designed to vibrate in the longitudinal mode, a tuned spacer rod of fused quartz (Q), and the specimen (S). The joints between the crystals and between the gage crystal and the spacer rod were made with Loctite (Loctite Corp. North American Group, Rocky Hill, CT) glue; the joint between the spacer rod and the specimen was made with a ceramic glue.

With the specimen and the lower part of the spacer rod in the furnace at the test temperature, the DGQS assembly was driven at resonance by a closed-loop crystal driver and the resonant period was noted. The dynamic Young's modulus was determined from measurements of the masses of the various components, the resonant periods of the DGQ and DGQS assemblies, the mass density of the specimen, and the length of the specimen.

3. Results and Discussion

The microstructures of the three silicide specimens are shown in Fig. 2. In the micrographs, the light-colored background is the dominant $MoSi_2$ phase, and the light and dark gray areas are the Ti_5Si_3 phase. Other dark areas represent some porosity and artifacts caused by extraction of grains during the polishing process. The microstructures of specimens with 30 and 50% Ti_5Si_3 (Fig. 2b and c) appear to be very similar, whereas the microstructure of the 10% Ti_5Si_3 specimen (Fig. 2a) appears to be somewhat coarser.

The temperature dependence of the silicides is shown in Fig. 3. The Young's modulus for the 10% specimen decreased from about 360 GPa at room temperature to about 350 GPa near 900 °C, while the moduli for the 30 and 50% specimens decreased from around 325 GPa at room temperature to about 270 GPa at 600 °C. The data have been analyzed by linear regression; results of the analysis are presented in Table 1.

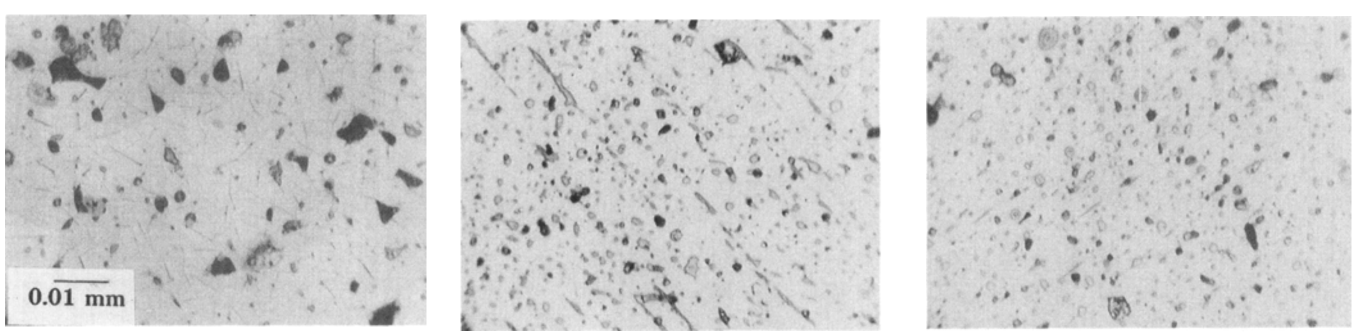
There is a general expectation that Young's modulus decreases as temperature rises, and this is confirmed for the silicides. The 10% specimen retains its stiffness very well at high temperatures. As noted in Table 1, the normalized modulus for this specimen is only 0.61×10^{-4}/K. The normalized moduli for the other two specimens are near 3×10^{-4}/K. The reason for this difference is not yet understood. The difference in grain size apparent in the microstructures shown in Fig. 2 would not be expected to affect the modulus or the temperature dependence of the modulus. A pronounced difference in texture, on the other hand, would. Such a difference in texture is not apparent in the microstructures. This behavior merits further research.

We can compare the normalized moduli measured here with the values compiled by Wachtman (Ref 7) for some polycrystalline ceramics and by Friedel (Ref 8) for pure elements (mostly metals). For ceramics the normalized modulus is near 1×10^{-4}/K, but that for metals is in the range of 4×10^{-4}/K to 14×10^{-4}/K. Thus, the normalized modulus measured here for the 10% specimen agrees with the values quoted by Wachtman, while the normalized moduli for the 30 and 50% specimens fall near the low end of the range quoted by Friedel. These trends reflect the lower rate of decreasing modulus with rising temperature for ceramics compared with metals, consistent with the influence of strong covalent and ionic interatomic bonding in ceramics.

The composition dependence of Young's modulus at 22 °C for the silicides is shown in Fig. 4. A polynomial curve of de-

Table 1 Linear regression analysis of the temperature dependence of dynamic Young's modulus (*E*) for the silicides
The equation used is $E = E(0) - (dE/dT)(T)$, with E and $E(0)$ in GPa and T in °C. $E(0)$ is the value of E at 0 °C, and dE/dT is the slope of the straight line in Fig. 1.

Volume fraction of Ti_5Si_3, %	$E(0)$, GPa	dE/dT, GPa/K	Correlation coefficient	Normalized modulus $(dE/dT)/E(0)$, K^{-1}
10	357.8	0.0218	0.447	0.61×10^{-4}
30	320.5	0.0970	0.829	3.02×10^{-4}
50	301.4	0.1819	0.906	2.27×10^{-4}



(a) (b) (c)

Fig. 2 Microstructure of the silicides with 10% (a), 30% (b), and 50% (c) volume fraction of Ti_5Si_3

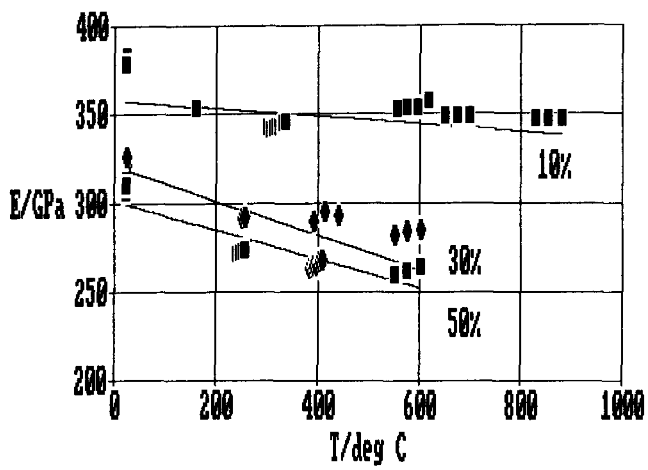


Fig. 3 Temperature dependence of dynamic Young's modulus for the silicides

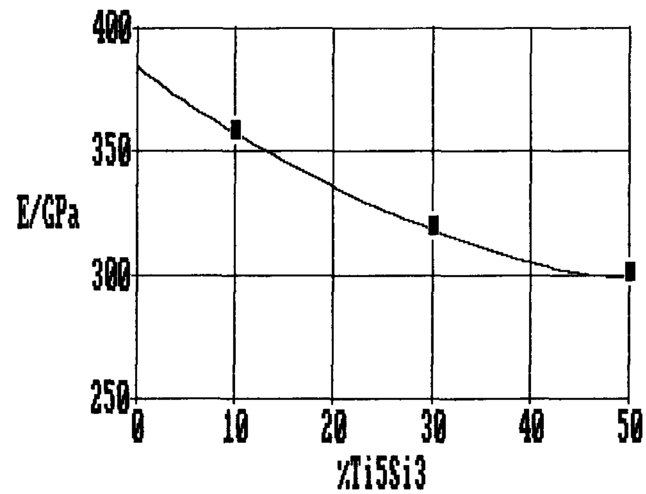


Fig. 4 Composition dependence of dynamic Young's modulus for the silicides at room temperature

gree two has been fitted to the data. This curve fitting yields a value of 384.5 GPa for the modulus of pure $MoSi_2$. Our value of modulus is in excellent agreement with the value of 387.5 GPa quoted by Srinivasan and Schwartz (Ref 9). Further, for $MoSi_2$ the values of modulus/density (in units of GPa/(g/cm^3)) found by Fleischer (Ref 10) and determined from the data of Srinivasan and Schwartz (Ref 9) are 70 and 64.4, respectively. With an extrapolated value for density, our value of modulus/density of 63.7 is in good agreement with the other two values.

4. Conclusions

- The Young's modulus for the 10% specimen decreased from about 360 GPa at room temperature to about 350 GPa near 900 °C, while the moduli for the 30 and 50% specimens decreased from around 325 GPa at room temperature to about 270 GPa at 600 °C.
- Linear regression analysis of the temperature dependence of dynamic Young's modulus (E) for the silicides yielded an equation of the form: $E = E(0) - (dE/dT)(T)$, where E and $E(0)$ are in gigapascals and T is in degrees Celsius. $E(0)$ is the value of E at 0 °C, and dE/dT is the slope of the E versus T graph. The values of $E(0)$ and dE/dT for the three specimens are: 10%, 357.8 GPa and 0.0218 GPa/K (T up to 900 °C); 30%, 320.5 GPa and 0.0970 GPa/K (T up to 600 °C); and 50%, 301.4 GPa and 0.0819 GPa/K (T up to 600 °C).
- The values determined for the normalized modulus $[(dE/dT)/E(0)]$ for the 10, 30, and 50% specimens were 0.61×10^{-4}, 3.02×10^{-4}, and 2.72×10^{-4}/K, respectively. These values are consistent with the low values reported for ceramics in the literature.
- From the composition dependence of Young's modulus at 22 °C for the silicides, a value of 384.5 GPa for the modulus of pure $MoSi_2$ was obtained. This value of modulus is in excellent agreement with the value of 387.5 GPa given in Ref 9. For $MoSi_2$, the values of modulus/density (in units of GPa/(g/cm^3)) from the literature (70 and 64.4) are in good agreement with our value of 63.7.

References

1. J.J. Petrovic and R.E. Honnell, *Mater. Res. Soc. Bull.*, Vol 18, 1993, p 35
2. E. Fitzer, D. Rubisch, J. Schlichting, and I. Sewdas, *Spec. Ceram.*, Vol 8, 1973
3. J. Marx, *Rev. Sci. Instrum.*, Vol 22, 1951, p 503
4. W.H. Robinson and A. Edgar, *IEEE Trans. Sonics Ultrasonics*, Vol SU-21, 1974, p 98
5. M.R. Harmouche and A. Wolfenden, *J. Test. Eval.*, Vol 13, 1985, p 424
6. M.R. Harmouche and A. Wolfenden, *J. Test. Eval.*, Vol 15, 1987, p 101
7. J.B. Wachtman, Jr., Ed., *Mechanical and Thermal Properties of Ceramics*, Special Publication 303, National Bureau of Standards, May 1969, p 158
8. J. Friedel, *Dislocations*, Pergamon Press, 1964, p 454-457
9. S.R. Srinivasan and R. Schwartz, *J. Mater. Res.*, Vol 7, 1992, p 1610
10. R.L. Fleischer, *J. Mater. Sci.*, Vol 22, 1987, p 2281