JOURNAL OF MATERIALS SCIENCE 27 (1992) 2660–2664

Mechanical properties of vanadium beryllide, VBe_{12}

T. G. NIEH, J. WADSWORTH
Lockheed Missiles and Space Company, O/9310, B/204, 3251 Hanover Street, Palo Alto, CA 94304, USA
F. C. GRENSING
Brush Wellman Company, 17876 Sinclair Street, Cleveland, OH 44110, USA
J.-M. YANG
Materials Science and Engineering Department, University of California, Los Angeles, CA 90024, USA

The mechanical properties of VBe_{12}, both at room and elevated temperatures (up to 1200 °C), have been measured. Room-temperature properties, including Young's modulus, flexural strength, and fracture toughness are reported. The material behaved elastically at room temperature but became plastic at temperatures above 1000 °C. Creep properties of VBe_{12} were also studied in temperature ranges from 1000–1200 °C and applied stress ranges from 33–58 MPa. At low strain rates (approximately $< 10^{-5}s^{-1}$), the stress exponent was about 4, suggesting deformation was controlled by dislocation climb. Microstructural examination indicated that fracture was initiated from grain boundaries subjected to tensile stresses. The creep behaviour of VBe_{12} is briefly compared with that of other intermetallics.

1. Introduction

Amongst all of the ordered alloy groups, the beryllides possess a great potential for high-temperature structural applications, particularly at temperatures above 1000 °C, because of their low density, good high-temperature strength, and oxidation resistance [1–3]. Extensive studies on beryllides were undertaken in the early 1960s [4–7], but the research was terminated because of the difficulties of reducing impurity levels and the room-temperature brittleness problems associated with the compounds. Recently, the interest in using beryllides has been revived because the sophisticated structural requirements for some of the proposed advanced flight vehicles, such as NASP, cannot be met by conventional materials. Beryllides, and in particular refractory metal beryllides, are considered to have the best potential to meet the high-temperature properties required for these applications. Unfortunately, almost all refractory metal beryllides have an extremely complex crystal structure. Consequently, conventional dislocation motion is extremely difficult, if not impossible, in refractory metal beryllides at low temperatures. Synchroshear [8] may take place in a complex lattice at low temperatures, but it is energetically highly unfavourable. At elevated temperatures, however, certain diffusional processes prevail, plastic flow can take place, and this should lead to ductile behaviour.

The creep strength is expected from a theoretical viewpoint to be good in intermetallic alloys, especially for materials (like most refractory metal beryllides) which have a complex crystal structure. This is because of the relatively low atomic mobility, even at high temperatures, in these compounds. The creep properties of many intermetallics, e.g. TiAl [9], Ti_3Al [10], NiAl [11, 12], CoAl [12, 13], and Ni_3Al [14], have been characterized. The creep properties of refractory metal beryllides, however, have not yet been studied systematically.

In the present paper, the mechanical properties, both at room and elevated temperatures, of VBe_{12}, will be presented. Vanadium beryllide, VBe_{12}, has a relatively simple structure compared to other refractory metal beryllides. Specifically, VBe_{12} has a $D2_b$ structure; the unit cell of VBe_{12} is composed of 26 atoms arranged in a tetragonal lattice, with vanadium atoms located at the centre and at each corner of the cell.

2. Experimental procedure

Vanadium beryllide powders were prepared by repeated sintering and grinding with elemental vanadium and beryllium powders of an appropriate fraction. The precise processing steps have been previously described [4, 15]. Both vacuum hot pressing (VHP) and hot isostatic pressing (HIP) techniques were used to prepare bulk VBe_{12} from these prealloyed powders. The VHP process was carried out at 1300 °C with an applied pressure of 14 MPa for 2 h, whereas the HIP process was carried out at 1120 °C with an applied pressure of 105 MPa for 3 h. The density of the two materials was measured using a liquid (isopropanol alcohol) immersion technique. The major impurities in the materials, identified by energy dispersive spectroscopy, are iron, nickel and cobalt, which originated from the starting elemental beryllium and vanadium powders. Sheet specimens for mechanical testing were prepared directly from the as-consolidated materials by electric discharge machining and grinding.

High-temperature creep tests in air were conducted at temperatures of 1000–1200 °C and at stresses from 33–78 MPa. Specimens (50 mm × 4 mm × 3 mm) were loaded in a four-point bending fixture of hot-pressed SiC with inner spans of 20 and 40 mm, respectively. Constant load was applied using a lever system and a dead weight acting on the upper push rod. Creep curves (i.e. plastic strain versus time) were monitored using a linear variable differential transducer (LVDT). The steady-state creep rates were directly calculated from the creep curve. Because of the limited travel distance, a maximum strain of only 2% can be measured. This strain is normally sufficient to provide steady-state creep data. After testing, the specimens were examined using optical and scanning electron microscopy to characterize the creep fracture surface as well as the initiation and propagation of cracks during creep.

3. Results and discussion

The microstructures of the HIP and VHP VBe_{12} are shown in Figs 1 and 2, respectively. The grains are virtually equiaxed in both materials. The grain size in the VHP sample is uniform and has an average value of 22 μm. The grain size in the HIP sample, however, is noted to be bimodal: grain-size distributions have average sizes of about 5 and 20 μm, respectively. The bimodal grain-size distribution in the HIP material is probably an indication of incomplete recrystallization, resulting from the relatively low consolidation temperature. The presence of voids and impurities (visible as bright specks) in these two materials is evident in Figs 1 and 2. In addition, the pores are generally larger and the porosity content is generally higher in the HIP sample than in the VHP sample.

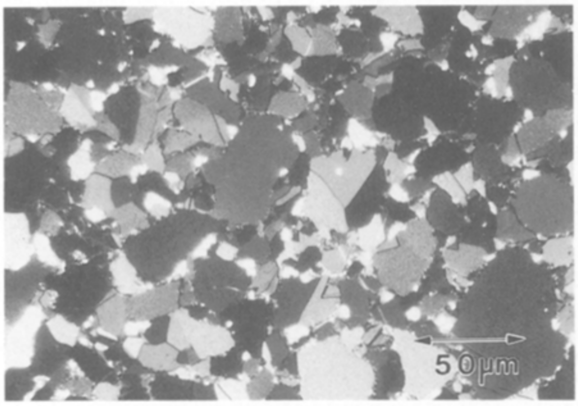

Figure 1 Microstructure of VBe_{12} prepared by hot isostatic pressing at 1120 °C and 105 MPa for 3h.

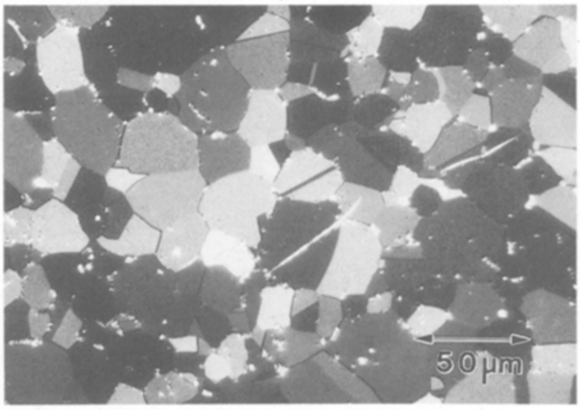

Figure 2 Microstructure of VBe_{12} prepared by vacuum hot pressing at 1300 °C and 14 MPa for 2h.

These observations are in good agreement with the experimental densities of 2.310 and 2.380 Mg m^{-3} for the HIP and VHP materials, respectively. These two density values correspond to 97% and 100% theoretical. (Note: the densities of the major impurities are generally higher than that of VBe_{12}. This explains why the density value of the VHP material can be 100% theoretical, even though it contains voids.) The low density in the HIP sample is attributed to the low consolidation temperature compared to the VHP material.

3.1. Room-temperature properties

A four-point bend test was conducted on a VBe_{12} sample prepared by vacuum hot pressing. The deflection curve from this test is given in Fig 3. The sample behaved in a brittle manner and did not exhibit any plasticity. The Young's modulus and flexural strength were calculated to be approximately 300 GPa and 140 MPa, respectively; these values are consistent with those obtained by Booker *et al.* [15]. The fracture surface of the sample is shown in Fig. 4. It is evident that the sample failed intergranularly, which suggests that fracture of the sample was limited by the strength of grain boundaries. The preferential grain-boundary fracture path may be attributed to the presence of either voids or impurities, such as oxides or other brittle inclusions (marked by arrows in Fig. 4), at grain boundaries.

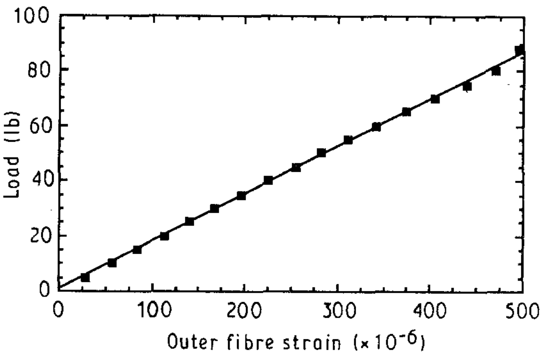

Figure 3 Deflection curve from four-point bend test of VBe_{12}. The elastic modulus is about 300 GPa. (1 lb = 0.45 kg)

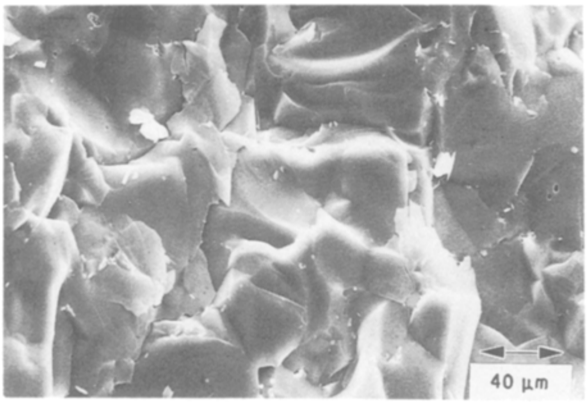

Figure 4 Fracture surface of VBe_{12} at room temperature, showing an intergranular fracture mode.

Microhardness measurements were carried out on VBe_{12}. The hardness value is about 11.4 ± 0.8 GPa. A typical microhardness indent on VBe_{12} is shown in Fig. 5. Radial microcracks were readily observed near the four corners of the indent. It is pointed out that these microcracks propagate transgranularly, which is in contrast to the intergranular crack propagation in the bend specimen shown in Fig. 4. This is probably because of the fact that fracture is less sensitive to voids and impurities in compression than it is in tension. The flexural strength measured from the four-point bend test, therefore, represents a lower bound value. High-purity VBe_{12} would be expected to fracture transgranularly and exhibit a flexural strength which is higher than 140 MPa.

The fracture toughness of VBe_{12} can be estimated from the length of the radial microcracks shown in Fig. 5 using a fracture micromechanics approach. Specifically, using the formula [16]

$$K_c = \xi_v \left[\frac{E}{H}\right]^{1/2} \left[\frac{P}{C_o^{3/2}}\right]$$

where K_c is the fracture toughness, ξ_v is a constant, E is the modulus, H is the hardness, P is the load, and C_o is the crack length, the fracture toughness of VBe_{12} is calculated to be about 0.84 MPa $m^{1/2}$. This value is close to that found in many glasses, but is lower than that of some ceramics (e.g. 2.1 MPa $m^{1/2}$ for Al_2O_3 [17]). The low fracture toughness in VBe_{12} is somewhat expected because it is a highly ordered compound, similar to many ceramics.

It is of interest to note that Lewis [7] has observed plastic slip line formation on $NbBe_{12}$ during microhardness tests at − 196°C. Lewis proposed that this was a result of synchroshear, from a model originally proposed by Kronberg [8]. VBe_{12} has the same crystal structure and a similar melting point to $NbBe_{12}$. No slip lines were found near the microhardness indent of VBe_{12}, however, despite the fact that VBe_{12} has the same crystal structure and a similar melting point as $NbBe_{12}$. Furthermore, to confirm Lewis' result, an $NbBe_{12}$ sample was also prepared, but no slip line was observed near the microhardness indents on this sample.

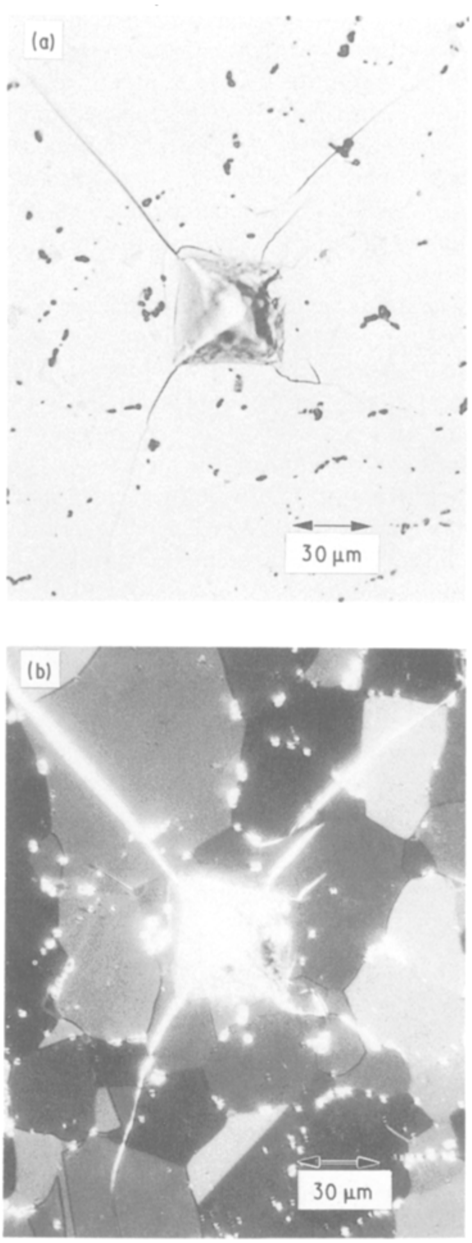

Figure 5 Microhardness indents: (a) bright-field image, and (b) image under polarized light, on VBe_{12} showing transgranular fracture. Fracture toughness is calculated to be 0.84 MPa $m^{1/2}$.

3.2. Creep properties

VBe_{12} is readily observed to flow plastically at temperatures above 1000 °C, a temperature that corresponds to 0.64 T_m, where T_m is the absolute melting point of VBe_{12}. This is consistent with the fact that Nieh and Wadsworth [18] have pointed out that the ductile-to-brittle transition temperature for refractory metal beryllides is expected to be about 0.5 T_m. For VBe_{12}, this value is about 750 °C. A specimen creep deformed at 1150 °C is shown in Fig. 6. The outer fibre strain for this deformed sample is approximately 2% which is the practical deflection limit of the current creep apparatus.

The creep curve for VBe_{12} at 1150 °C and 33 MPa is shown in Fig. 7. The general features in this curve are typical of the other test conditions. The curve in Fig. 7 exhibits a short primary stage, followed by a relatively long apparent steady-state creep. The primary stage is

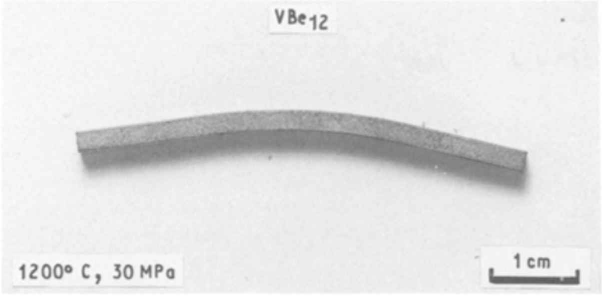

Figure 6 VBe_{12} specimen creep deformed at 1200 °C. The outer fibre strain is about 2%.

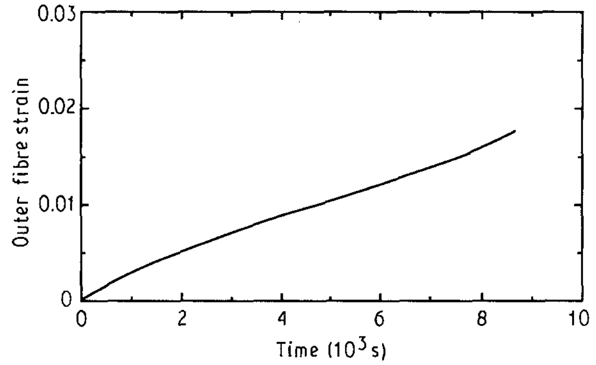

Figure 7 Creep curve of VBe_{12}, performed at 1150 °C with an applied stress of 33 MPa, showing the primary and secondary stages.

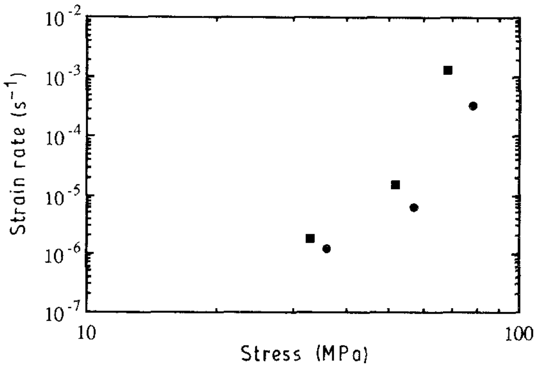

Figure 8 A log $\dot{\varepsilon}$ versus σ plot for VBe_{12}. The stress exponent increases with increasing temperature: (●) 1100 °C, (■) 1150 °C. In the low strain rate regime, the stress exponent is about 4.

usually less than 0.25% of plastic strain. The steady-state creep rates obtained from the present study at various stresses and temperatures are summarized in Table I. Steady-state creep rate consistently increases with increasing temperature and applied stress.

The steady-state creep rate of VBe_{12} as a function of applied stress at 1100 and 1150 °C is shown in Fig. 8. Despite the limited data, several important features are worth pointing out in Fig. 8. The stress exponent, n, is noted to increase with increasing strain rate (or applied stress) and there appears to exist a transition in deformation behaviour at strain rates higher than $10^{-5}\ s^{-1}$ (or at applied stresses higher than 50 MPa). Specifically, the n values are estimated to be about 4 and 10 in the low and high strain rate regimes, respectively. This increase in the n value may be associated with the power-law breakdown in the high strain rate regime. It is of interest to note that the n value in the low strain rate regime is similar to those obtained from other intermetallics, such as TiAl [9], Ti_3Al [10], NiAl [11, 12], and CoAl [12, 13], creep deformed under similar test conditions. Specifically, the n = 4, 4.5, 6, and 4.7 for TiAl [9], Ti_3Al [10], NiAl [11], and CoAl [13], respectively. The above result indicates that, in spite of the fact that VBe_{12} has a more complex crystal structure compared with various aluminides, creep mechanisms for these intermetallics are similar. Namely, deformation is mainly controlled by dislocation climb. This is also consistent with the fact that, despite the differences in grain sizes and void content in the VHP and HIP samples, creep data measured from the two materials are quite similar. The presence of grain-boundary impurities and voids may affect the rupture strain, but creep is essentially controlled by bulk deformation.

TABLE I Steady-state creep rate of VBe_{12}

Material	Temperature (°C)	Stress (MPa)	Steady-state creep rate (s^{-1})
VHP	1000	58	3.1×10^{-7}
VHP	1100	36	1.2×10^{-6}
HIP	1100	57	6.0×10^{-6}
HIP	1100	78	3.3×10^{-6}
HIP	1150	33	1.8×10^{-6}
HIP	1150	52	1.5×10^{-5}
HIP	1150	68	1.3×10^{-3}
VHP	1200	38	1.4×10^{-5}

The temperature dependence of creep can be determined by activation energy analysis. The activation energy for creep can be obtained from an Arrhenius plot, using data from Fig. 8 and Table I. Although the present experimental data are limited, the activation energy in the low strain rate regime is estimated at 35 MPa to be about 270 kJ mol^{-1}. This activation energy value is impossible to compare with literature values because diffusion data for beryllides are not available. It is, however, interesting to note that this value is in close proximity to the activation energies for creep in TiAl (~ 300 kJ mol^{-1}) [9], Ti_3Al (~ 223 kJ mol^{-1}) [10], NiAl (~ 300 kJ mol^{-1}) [11], and CoAl (~ 300–350 kJ mol^{-1}) [13].

The cross-sectional microstructure of a sample creep tested to a strain of 2% is shown in Fig. 9. Despite the fact that the sample is still in the steady state, a major intergranular crack can already be observed. This intergranular crack is initiated at the tensile side surface of the specimen and propagates toward the centre. A few secondary cracks are also developed. It is noted that no evidence of grain-boundary sliding was observed, which confirms that, in the present study, creep of VBe_{12} is primarily dominated by bulk deformation. Namely, the measured stress exponent is always higher than 4. The grain morphology, however, remains equiaxed because of the relatively small plastic strain (~ 2%) in the test sample.

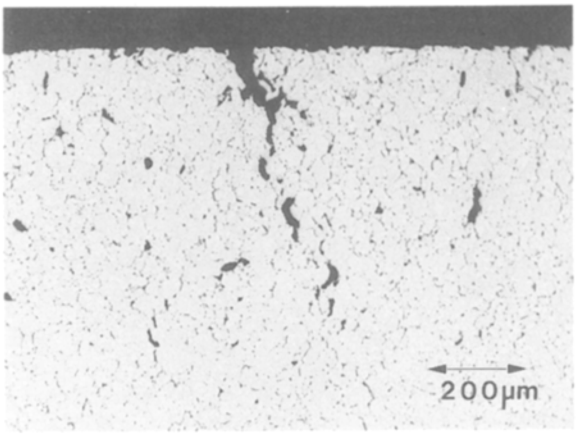

Figure 9 Cross-sectional microstructure from a creep deformed specimen showing intergranular fracture mode.

4. Conclusion

The mechanical properties of VBe_{12} both at room and elevated temperatures (up to 1200 °C) have been measured. The material exhibits brittle behaviour at room temperature but exhibits extensive ductility at temperatures above 1000 °C. The Young's modulus and flexural strength of VBe_{12} are measured to be 300 GPa and 140 MPa, respectively. The fracture toughness of VBe_{12} is calculated to be about 0.84 MPa $m^{1/2}$. Experimental data indicate that, in the low strain rate regime (approximately $< 10^{-5} s^{-1}$), the stress exponent for creep of VBe_{12} was about 4, suggesting that deformation was controlled by dislocation climb. In the high strain rate regime, however, power law breakdown dominates. High-temperature fracture of VBe_{12} initiates from grain boundaries which are subject to tension.

Acknowledgements

This work was supported by the Lockheed Independent Research Program. The authors would like to thank Dr J. S. Lee for his technical contribution.

References

1. T. G. NIEH, J. WADSWORTH and C. T. LIU, in "High Temperature Ordered Intermetallic Alloys III," MRS Symposium Proceedings, Vol. 133, edited by C T. Liu, A. I. Taub, N. S. Stoloff and C. C. Koch (Materials Research Society, Pittsburgh, PA, 1989) p. 743.
2. J. S. LEE and T. G. NIEH, in "Oxidation of Intermetallic Compounds," edited by T. Grobstein and J. Doychak (The Minerals, Metals and Materials Society, Warrendale, PA, 1988) p. 271.
3. J. M. MARDER and A. J. STONEHOUSE, in "2nd International SAMPE Metals and Metals Processing Conference", Vol. 2, "Space Age Metals Technology" edited by F. H. Froes and R. A. Cull (Society for Advancement of Material and Process Engineering, Covina, CA, 1988) p. 357.
4. A. J. STONEHOUSE, R. M. PAINE and W. W. BEAVER, in "Mechanical Properties of Intermetallic Compounds", edited by J. H. Westbrook (Wiley, New York, 1960) p. 297.
5. K. R. FRANCIS, PhD dissertation, University of Arizona (1969).
6. J. R. LEWIS, *J. Metals* **13** (1961) 357.
7. *Idem, ibid.* **13** (1961) 829.
8. M. L. KRONBERG, *Acta Metall.* **5** (1957) 507.
9. P. L. MARTIN, M. G. MENDIRATTA, and H. A. LIPSITT, *Metall. Trans.* **14A** (1983) 2170.
10. M. G. MENDIRATTA and H. A. LIPSITT, *J. Mater. Sci.* **15** (1980) 2985.
11. J. D. WHITTENBERGER, *ibid.* **22** (1987) 394.
12. D. L. YANEY and W. D. NIX, *ibid.* **23** (1988) 3088.
13. J. D. WHITTENBERGER, *J. Mater. Sci. Engng* **73** (1985) 87.
14. J. H. SCHNEIBEL and W. D. PORTER, *J. Mater. Res.* **3** (1988) 403.
15. J. BOOKER, R. M. PAINE and A. J. STONEHOUSE, "Investigation of Intermetallic Compounds for Very High Temperature Applications", The Brush Beryllium Company, USAF Contract 33 (616)–6540, WADD Technical Report 60–889, March (1961).
16. G. R. ANSTIS, P. CHANTIKUL, B. R. LAWN and D. B. MARSHALL, *J. Amer. Ceram. Soc.* **64** (1981) 533.
17. B. R. LAWN, D. B. MARSHALL, P. CHANTIKUL and G. R. ANSTIS, *J. Aust. Ceram. Soc.* **16** (1) (1980) 4.
18. T. G. NIEH and J. WADSWORTH, *Scripta Metall. Mater.* **24** (1990) 1489.

Received 27 July 1990
and accepted 6 February 1991