# **Strength and Creep in Boron Carbide (B<sub>4</sub>C) and Aluminum Dodecaboride (***a***-AlB<sub>12</sub>)**

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Some thermomechanical properties of boron carbide and aluminum dodecaboride are presented. The dependence of the bending strength on the temperature of  $B_4C$  and  $AIB_1$ , in the ductile rupture region was studied. It was detected that the ductile rupture of  $B_4C$  starts at temperatures above 1500 $\degree$ C while that of  $\text{AlB}_{12}$  begins at 1400°C. Total deformation, however, did not exceed 2% even at 1800 $^{\circ}$ C. The investigation of the creep of B<sub>4</sub>C and  $\text{AlB}_{12}$  showed that the rate  $d\epsilon/dt$  of steady creep depends on the applied stress and the temperature:  $d\epsilon/dt = A\sigma^n \exp(-E/RT)$ . Besides, creep in boron carbide at stress values lower than 90 MPa occurred by the vacancy-diffusive mechanism, for which  $n = 1$ . At stress values over 90 MPa,  $n = 3$  and the creep mechanism changes to dislocation creep. A similar phenomenon is observed in the case of aluminum dodecaboride; however, the replacement of the vacancy-diffusive mechanism by dislocation creep takes place at stress values of 80 MPa. © 2000 Academic Press

*Key Words:* boron carbide; aluminum dodecaboride; mechanical properties; bending strength; creep rate.

# INTRODUCTION

Refractory boron-rich compounds of boron carbide and aluminum dodecaboride are widely used at high temperatures (1000–2000 $^{\circ}$ C) in various advanced technical fields [\(1\)](#page-2-0) and the investigation of the thermomechanical properties of these materials at high temperatures ( $T > 0.5T_{\text{melt}}$ ) is of interest.

This paper contains the results of the investigation of strength and creep in boron carbide and aluminum dodecaboride at temperatures above  $1000^{\circ}$ C. To our knowledge, this is the first investigation of this kind.

# EXPERIMENTAL

Strength tests were carried out by a three-point bending technique with a deformation rate of 0.2 mm/min. The size of the samples was  $2 \times 2 \times 20$  mm.

Creep was studied on prismatic samples  $(3 \times 3 \times 12 \text{ mm})$ under compression and the 1246P-2/2500 (USSR) device was used in the experiments. The temperature was measured and controlled with an optical pyrometer and tungsten-rhenium thermocouples. Deformation was measured with the cathetometer. A detailed description of the experimental technique is reported in [\(2](#page-2-0), [3\)](#page-2-0).

The samples of boron carbide  $(B_4 C)$  and aluminum dodecaboride ( $\alpha$ -AlB<sub>12</sub>) were produced by hot pressing [\(4, 5\)](#page-2-0) and their characteristics are reported in [Table 1](#page-1-0).

# RESULTS AND DISCUSSION

[Figure 1](#page-1-0) presents the temperature dependence of bending strength in boron carbide and aluminum dodecaboride samples. The behavior with three regions has a "classical" shape specific to ceramic materials. At temperatures below  $1000^{\circ}$ C, the temperature increase does not lead to a considerable change of the fracture strength. In this region the fracture is brittle, owing to expansion of pre-existing defects (cracks, pores). Above  $1000^{\circ}$ C, with the development of microductility the strength increases and reaches a maximum at the temperature of brittle-ductile transition as a result of thermally activated relaxation of the local peak stresses near the structural defects. Further temperature increase causes progression of macroductility and a monotonic decrease of yield stress and strength [\(6, 7\)](#page-2-0).

The results of creep in boron carbide and aluminum dodecaboride are respectively presented in [Figs. 2](#page-1-0) and [3.](#page-1-0) The boron carbide creep is characterized by a short (10 minutes) transitional creep and low values (up to 0.3%) of instantaneous deformation.

The aluminum dodecaboride creep also has a short (30 minutes) transitional creep and a prolonged steady-state section. The samples were tested up to 7% of deformation, since deviations from linearity in the steady-state creep region were observed owing to an increase in the sample cross section. The main contribution to the total deformation is made by steady creep; however, instantaneous deformation and deformation at transition creep markedly

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increase when the sample density decreases (up to 1% and 2.5% respectively at a density of 1.99  $g/cm^3$ ).

[Figures 4](#page-2-0) and [5](#page-2-0) illustrate the steady creep rate versus applied stress and temperature. The steady creep rate (*d*e/*dt*) in both boron carbide and aluminum dodecaboride is described as

$$
d\varepsilon/dt = A\sigma^n - \exp(-E/RT), \qquad [1]
$$

where *A* is a constant,  $\sigma$  the applied stress, *E* the creep activation energy,  $R$  the gas constant, and  $T$  the temperature.

An analysis of the results [\(Figs. 4 a](#page-2-0)nd [5\)](#page-2-0) shows that boron carbide creep occurs by the vacancy-diffusive mechanism with an exponent of  $n = 1$  at stress values below 90 MPa. At stresses above 90 MPa the creep mechanism changes and dislocation creep with an exponent of  $n = 3$  is dominant [\(Fig. 4\)](#page-2-0). The creep activation energy in both cases is the same and equal to 385 kJ/mole [\(Fig. 5\)](#page-2-0).

A similar phenomenon is observed in aluminum dodecaboride; however, the replacement of the vacancydiffusive mechanism by dislocation creep takes place at



FIG. 2. Boron carbide creep under 100 MPa; (1)  $1500^{\circ}$ C, (2)  $1650^{\circ}$ C,  $(3) 1800^{\circ}$ C.

stress values of 80 MPa [\(Fig. 4\)](#page-2-0). The creep activation energy in aluminum dodecaboride also does not depend on applied stress and is equal to 460 kJ/mole [\(Fig. 5\).](#page-2-0)

As previously mentioned [\(8](#page-2-0), [9\)](#page-2-0), the materials in the dislocation creep region are subdivided into two classes: for one, exponent  $n = 3$  in Eq. (1), for the other,  $n = 5$ . In  $Ref. (10)$  $Ref. (10)$ , it is supposed that here the difference in the types of interatomic bonds is predominant, and compounds with covalent bonds (both boron carbide and aluminum dodecaboride belong to this class) have exponent  $n = 3$ .

The creep rate versus temperature for applied stress values of 50 and 100 MPa give parallel lines [\(Fig. 5\)](#page-2-0), which indicate that both creep mechanisms (the vacancy-diffusive and dislocation mechanisms) have the same nature. The creep in ceramic materials is controlled by the diffusion of the slowest atom [\(2\)](#page-2-0). In the case of boron carbide, carbon atoms are the slowest ones, because the diffusion rate of boron into carbon was higher than the diffusion rate of carbon into boron [\(11\).](#page-2-0) A good agreement of the creep



**FIG. 1.** High-temperature bending stress: (1) boron carbide  $(B_4C)$ ; (2) aluminum dodecaboride ( $\alpha$ -AlB<sub>12</sub>), density 2.37 g/cm<sup>3</sup>; (3) aluminum dodecaboride ( $\alpha$ -AlB<sub>12</sub>), density 2.22 g/cm<sup>3</sup>; (4) aluminum dodecaboride  $(\alpha$ -AlB<sub>12</sub>), density 1.99 g/cm<sup>3</sup>.



FIG. 3. Aluminum dodecaboride creep: (1) 50 MPa, 1500°C, density 2.37 g/cm<sup>3</sup>; (2) 100 MPa, 1500°C, density 2.37 g/cm<sup>3</sup>; (3) 100 MPa, 1650°C, density 2.37 g/cm<sup>3</sup>; (4) 50 MPa, 1500°C, density 2.22 g/cm<sup>3</sup>; (5) 50 MPa, density  $1.99$  g/cm<sup>3</sup>.

<span id="page-1-0"></span>TABLE 1 Characteristics of the Samples Used in the Investigations

<span id="page-2-0"></span>

FIG. 4. Steady creep rate versus a stress: (1) boron carbide, at  $1650^{\circ}$ C; (2) boron carbide, at  $1800^{\circ}$ C; (3) Aluminum dodecaboride, density 2.37 g/cm<sup>3</sup>, at 1500°C; (4) aluminum dodecaboride, density 2.37 g/cm<sup>3</sup>, at  $1650^{\circ}$ C; (5) aluminum dodecaboride, density 2.22 g/cm<sup>3</sup>, at  $1650^{\circ}$ C; (5) aluminum dodecaboride, density 1.99 g/cm<sup>3</sup>, at 1650°C.

activation energy (385 kJ/mole) in boron carbide with the activation energy of carbon self-diffusion,  $-382 \text{ kJ/mole}$ (12), supports this statement. In the case of aluminum dodecaboride, we can presume the steady creep rate to be controlled by the boron atom diffusion.

# **CONCLUSION**

The ductile rupture of  $B_4C$  starts at temperatures above  $200C_1 + 1$ 1500 $\degree$ C while that of AlB<sub>12</sub> begins at 1400 $\degree$ C. Total deformation, however, did not exceed  $2\%$  even at  $1800^{\circ}$ C. The steady creep rate *d*e/*dt* depends on the applied stress and the temperature. Creep in boron carbide at stress values lower than 90 MPa occurred by the vacancy-diffusive mechanism. At stress values over 90 MPa the creep mechanism changes to dislocation creep. A similar phenomenon is observed in the case of aluminum dodecaboride; however, the replacement of the vacancy-diffusive mechanism by dislocation creep takes place at stress values of 80 MPa.



FIG. 5. Steady creep rate versus a temperature: (1) boron carbide, under 50 MPa; (2) boron carbide, under 100 MPa; (3) aluminum dodecaboride, density  $2.37$  g/cm<sup>3</sup>, under 50 MPa; (4) aluminum dodecaboride, density 2.37 g/cm<sup>3</sup>, under 100 MPa; (5) aluminum dodecaboride, density  $2.22$  g/cm<sup>3</sup>, under 50 MPa; (6) aluminum dodecaboride, density 1.99  $g/cm^3$ , under 50 MPa.

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