at% in excess of the stoichiometric composition WB, was pretreated at 500°C for 60 min in a stream of hydrogen and subsequently treated at 1400°C for 60 min in a stream of argon. The crystallinity and the amount of WB increased with increasing both heat treatment time and temperature. The atomic ratio B/W remained constant after the heat treatment below 1500°C. Sintering of the synthesized powder at 4 GPa and 1500°C for 15 min resulted in the density of 13.3 g/cm³ and microhardness of 1400 kg/mm². For 5 at% titanium addition, higher density (14.0 g/cm³) and improved microhardness (2800 kg/mm²) were attained by sintering at a lower temperature of 1300°C at 4 GPa. The densification of reaction-bonded WB is promoted possibly by the formation reaction of TiB_2 from free amorphous boron and added titanium. Similar behavior was observed with the zirconium addition also. [*Received July 15, 1986*]

Preparation of Ultra-Fine Spherical Particles of γ-Al_2O_3 by Burning of Aluminum Powder

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A new method for preparing ultra-fine alumina powders consisting of spherical particles based on combustion has been developed. A mixture of fuel gas, H_2+CH_4, and aluminum powder with particle size of several micrometers is burnt in a conventional burner for glass blowing. Examination of obtained products by electron microscopy and X-ray diffraction showed that they were alumina with two types of structures, θ- and δ-Al_2O_3. The present note describes an economical and efficient method for the production of alumina spherical particles. [*Received July 11, 1986*]

Preparation of Silicon Oxynitride Fiber and Its Mechanical Properties

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The nitridation of polycarbosilane started at 400° to 500°C in the heat treatment of polycarbosilane in NH_3 gas and was almost completed at about 800°C. Above 1000°C, the obtained nitride changed from amorphous to crystalline state, and at 1400°C, the crystalline phase was α-Si_3N_4. On the basis of the nitridation of polycarbosilane, oxidation-cured polycarbosilane fibers were heated in NH_3 gas between 1000° and 1400°C to obtain silicon oxynitride fibers. The silicon oxynitride fibers were in the amorphous state and transparent to visible light. The tensile strength and Young's modulus of the fibers obtained at 1300°C were 1.80±0.37 GPa and 139±17 GPa, respectively. The fibers obtained at 1400°C were heat-treated between 1000° and 1400°C in Ar gas and in air to examine the heat resistance of the fibers. The fibers retained their tensile strength and Young's modulus up to 1400°C in Ar gas and up to 1200°C in air. [*Received July 15, 1986*]

Microstructural Change of Polycrystalline Ba-Ferrite ($BaO \cdot 6Fe_2O_3$) by Hot-Extrusion Processing

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The hot-working processes such as rolling, forging, extrusion etc. are widely used for metallic materials to make their shape and improve their properties. For ceramics, these processes are seldom carried out because of the lack of their plastic property. However, it is also well known that ceramics can deform plastically at high temperature. In this study we tried to examine the possibility of the hot-extrusion process for polycrystalline Ba-ferrite ($BaO \cdot 6Fe_2O_3$) that is easier to deform plastically than other oxide ceramics. Polycrystalline Ba-ferrite samples capsulated in stainless steel (SUS 304) were pre-heated

from 1200° to 1430°C and then extruded by pressing the stem to the bullet at a maximum pressure of 800 MPa. After extrusion, the body became dense and the grains became fine and orientated. The analysis by SEM and X-ray diffraction patterns showed that the polycrystalline grains had the tendency to orient their c-plane to the pressing direction and in a concentric circle. These results are discussed in view of strain rate, applied stress and slip plane. Besides, the control of the heat-transfer from the billet to the container was very important to carry out the hot-extrusion process smoothly.

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Synthesis of AlN by the Nitridation of the Floating Al Particles in N_2 Gas

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The nitriding reaction of floating Al particles in N_2 gas was studied at 1350°-1550°C. The hollow spherical and fibrous AlN's formed were examined by X-ray diffraction and scanning electron microscopy. The nitriding reaction of floating Al particles in N_2 gas proceeded in the following four steps : (i) floating Al particles melted and became spherical by surface tension, (ii) up to 1350°C the nitriding reaction was controlled by the diffusion of nitrogen through the AlN surface layer, (iii) cracks propagated in the AlN layer at 1400°-1550°C. The nitriding reaction rapidly proceeded with the eruption of internally melted Al, (iv) the eruption of Al also caused the nitriding reaction between vaporized Al and N_2 gas to form AlN, resulting in hollow spherical AlN and fibrous AlN. The synthesized AlN was white, indicative of high purity. Since the particle size of floating Al was determined by controlling the N_2 gas velocity, the classification of Al particles was possible. The mean size and size distribution of hollow spherical AlN particles formed were controlled within the range 4 to 12 μm. The hollow spherical AlN particles were so brittle that they were easily crushed to fine particles even by fingers. Thus obtained AlN particles were very fine and had narrow size distribution of 0.1-0.2 μm. [*Received July 17, 1986*]

Effects of NaF and NH_3 on Preparation of Si_3N_4 Powders from SiO_2

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Effects of NaF and NH_3 on the preparation of Si_3N_4 powders were investigated by nitridation of SiO_2-C-NaF system in a flow of NH_3/N_2 mixture gas at 1350°-1500°C. Reaction products were α-Si_3N_4 and β-Si_3N_4. A small amount of β-SiC formed at a higher reaction temperature. Also, a trace of Si_2ON_2 formed with an excess amount of NaF. The yield of Si_3N_4 powders increased remarkably with the addition of a small amount of NaF and/or NH_3. The α-phase content in Si_3N_4 powders also increased with the addition of a small amount of NaF, but was not influenced by the addition of NH_3. It was considered that NaF acted as flux and accelerated the reaction of SiO_2 with C, followed by the formation of SiO and Si, and that NH_3 promoted the reduction of SiO_2 to SiO or Si. Addition of excess amounts of NaF and NH_3 promoted the formation of Si_3N_4 whisker. From the thermodynamical consideration it was suggested that Si_3N_4 powders were formed by the following reactions :

$3\,SiO+3\,C+2\,N_2 \rightarrow Si_3N_4+3\,CO$ *and/or* $3\,Si+2\,N_2 \rightarrow Si_3N_4$.

The optimum condition to prepare Si_3N_4 powders was as-follows; SiO_2 : C : NaF = 1 : 20 : (0.01-0.05) (molar ratio), NH_3/N_2 = 50(ml/min)/350 (ml/min), 1350°C, 10 h. Si_3N_4 fine powders with α-phase contents of 94-96% were prepared with more than 95% yield. [*Received July 18, 1986*]