Microstructural Investigation of α-Si_3N_4 Whiskers by Transmission Electron Microscopy

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Alpha-Si_3N_4 whiskers prepared by the gas-solid reaction have been investigated by transmission electron microscopy (TEM). The correlations among morphology, growth direction and structural defect have been studied. The growth directions of whiskers have been determined accurately by observing the TEM images and corresponding diffraction patterns taken with two different orientations by large angle tilting of the specimens with a double-tilting device in an electron microscope. Defects in whiskers have been observed in detail using various TEM techniques, particularly by high resolution electron microscopy. Two distinct types of the whisker have been found. One is a triangular prism-like shape having the $|10\bar{1}0|$ growth direction, and the other is a thin tape-like shape having the $|10\bar{1}1|$* growth direction. The former includes only a small number of defects, whereas the latter has a lot of planar defects only in one side of whiskers. The planar defects in the latter whisker are found to consist of the $|0001|$ and $|10\bar{1}2|$ planar defects. The most probable displacement vector of the $|0001|$ planar defect is determined to be $a/2\langle 10\bar{1}0\rangle$.*
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Microstructure and Properties of Sintered Silicon Carbides Fabricated by Different Methods

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Studies were made of effects of fabrication methods on the properties and microstructure of sintered silicon carbides. The specimens used in this investigation were three kinds of commercially available SiC bodies which were fabricated by reaction bonding, pressureless sintering and hot-pressing. The hot-pressed SiC contained a small amount of BeO. Measurements were carried out on density, the polytype by X-ray diffraction method and 4-point bend strength. Microstructural observation was also carried out using an optical microscope, a scanning electron microscope (SEM) and a transmission electron microscope (TEM). The results of density measurement showed that the open porosities of three specimens were negligibly small and that the density of the hot pressed SiC had nearly the theoretical density. The measurement of 4-point bend strength indicated that the reaction bonded SiC had the highest value and the hot-pressed SiC the lowest. The analysis of the polytype indicated that all the specimens consisted mainly of α-SiC of 6 H type. In the reaction bonded SiC, about 11% of 3 C type (β-SiC) and 9% of free Si were recognized. The average grain diameter and fracture mode of each specimen were determined from observation with an optical microscope and SEM. In the hot-pressed SiC, the fracture occurred mainly at grain boundaries, whereas it occurred mostly in grains in the reaction bonded and pressureless sintered SiC. A lot of stacking faults were observed in all the specimens with a TEM. In addition, small closed pores were often recognized in the pressureless sintered SiC. In the hot-pressed SiC, a contrast originated from strain field within grains was observed, and dislocations near grain boundaries were a characteristic feature of this material. Small short partial dislocations accompanied by stacking fault were often observed in the reaction bonded SiC.
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Microstructure of $SrTiO_3$ Spherical Fine Particles Prepared by Ultrasonic Spray Pyrolysis of Metal Alkoxide

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Spherical fine particles of $SrTiO_3$ were prepared by ultrasonic spray pyrolysis of a methanol solutoin containing Sr and Ti iso-propoxides. The microstructure and composition of particles were studied by TEM and SEM. TEM observation revealed

that the particles prepared at 1200°C have a hollow around the center of the particle and consist of small primary particles of 0.02-0.05 μm. This primary particle size was comparable to both the particle size calculated from the specific surface area and crystallite size derived from XRD line broadening. While the particles prepared at 600°C contained some unreacted alkoxide and gave a broad XRD pattern and had no distinct particle shape in TEM photograph, particles calcined at 1000°C consisted of well developed crystalline primary particle of 0.02-0.05 μm.

The mechanism of particle formation and microstructure development were discussed based on partial hydrolysis and complex alkoxide polymer formation. [*Received February 18, 1986*]

Properties of Mullite Powder Prepared by Coprecipitation and Microstructure of Fired Bodies

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A mullite powder was prepared from aluminum isopropoxide and methyl silicate by coprecipitation using a dilute ammonium aqueous solution. Properties and microstructure of fired bodies were examined. The results were compared with those from a sol mixture (SM) and oxide mixture (OX). The coprecipitated powder (Cp), the primary particle size of which was about 200 Å, was amorphous by X-ray diffraction analysis. On heating, the amorphous phase changed into spinel phase at about 980°C, and then into mullite phase at about 1250°C. Mullite specimens calcined at 800° or 1000°C and fired at 1780°C for 1 h showed a high bulk density of 3.14 g/cm³ (relative density 99%). Lattice parameters, a_0 and b_0, of mullite decreased with increasing firing temperature from 1500° to 1690°C, but a_0 increased at 1780°C. Cp specimens contained the smallest amount of glass phase in the three specimens fired at 1780°C. [*Received February 14, 1986*]

Formation and Thermal Change of Monoclinic Zirconia Polycrystalline Thin Films by Sol-Gel Process

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A highly dispersed monoclinic zirconia hydrosol of rod-shaped ultrafine particles was prepared by hydrolysis of a concentrated zirconium oxychloride solution. Transparent gel films were formed by drying the sols set in a petri dish of glass. The particles in the films have a tendency to orientate the monoclinic b-axis perpendicularly to the film plane. The orientation increased as gelation was performed more slowly and increased with increasing firing temperature up to 1000°C. The firing shrinkage of films was greater in the thickness direction than in lateral directions. A polycrystalline thin film was obtained as an isolated monograin layer of about 400 Å thick after firing at 1000°C. [*Received February 18, 1986*]

Silicon Nitride Joining with Glass Solder in the System CaO-SiO_2-TiO_2

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Sintered silicon nitride ceramics were joined with CaO-SiO_2-TiO_2 glass solder without applying pressure. The properties of the glass solders and Si_3N_4-glass reactions were studied in detail. The bond strength obtained from the Si_3N_4 joints was strong enough for practical applications. [*Received February 4, 1986*]