

Selected Abstracts from *Yogyo-Kyokai-Shi*

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Preparation of $\text{Pb}(\text{Zr},\text{Ti})\text{O}_3$ by Oxalate Method in Ethanol Solution (Part 1)

Investigation of Preparation Procedures

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$\text{Pb}(\text{Zr}_{x,3}\text{Ti}_{1-x,3})\text{O}_3$ (PZT) was prepared by calcining the oxalates which were co-precipitated from a reaction of metallic components in small amount of water with oxalic acid in large amount of ethanol. In the co-precipitation experiments, mixing procedures of two kinds of solutions and effects of additional titration with ammonia solution after oxalation were investigated. It was found that the addition of an aqueous solution containing metallic components to the ethanol solution of oxalic acid and subsequent addition of an ammonium solution were necessary in order to obtain highly dispersed fine-particulated PZT powders which have good crystallinity. These effects were confirmed by X-ray diffractometry, SEM observation, and measurements of particle size distribution. Calcination of thus obtained oxalate at 800°C for 2 h produced a mixed phase of PZT's of rhombohedral and tetragonal symmetries. The particle size was around 0.2 μm. On the other hand, the calcination at 1100°C produced the tetragonal phase, of which the c/a value was 1.029, and particle size was around 1 μm. [Received May 20, 1985]

Sinterability of $\text{SiO}_2\text{-Al}_2\text{O}_3$ Powders Prepared by Spray Pyrolysis

— Effect of Chemical Composition —

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The $\text{SiO}_2\text{-Al}_2\text{O}_3$ powders containing 68 to 78 wt% Al_2O_3 were synthesized by spray pyrolysis technique to study their sinterability at 1630°-1650°C. The prepared amorphous powders were transformed directly to crystalline mullite by calcination above 980°C. When heat-treated at temperatures above 1500°C, corundum was detected in the powder composed of 78 wt% Al_2O_3 . Densification of the silica-rich powder was accelerated by the formation of glassy phase, and the bulk

density of alumina-rich powder also increased with the increase of alumina content. While, in the $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ region, poor densification was observed. The shape of mullite grain in the sintered body changed from needle-like to roundish in compliance with the increase in alumina content, and corundum particles were observed along the grain-boundary in the sintered specimen of 78 wt% Al_2O_3 content. Glassy phase which consists merely of silicon and oxygen was observed along the grain-boundaries of the 70 wt% Al_2O_3 sintered body. [Received May 27, 1985]

Nitridation of Silicon Compact in a Gas Mixture of Nitrogen and Hydrogen

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Silicon powder compacts formed by slip casting were reaction-sintered at various heating rates in a mixed nitrogen-hydrogen atmosphere containing 0 to 80 vol % hydrogen. The rate of gas consumption measured in the temperature from 1050° to 1400°C gave two peaks. With increasing hydrogen content the nitriding fraction increased a maximum value at 40 vol % hydrogen. The hydrogen content also influenced the bending-rupture strength of sintered bodies, and the maximum bending-rupture strength was obtained at about 25 vol % hydrogen. The nitridation was almost independent of the heating rate. [Received August 12, 1985]

Effects of TiO_2 on Sintering of Alumina Ceramics

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The effects of TiO_2 addition on the sintering process and microstructure of alumina ceramics were studied. In air, the solid solubility of TiO_2 in $\alpha\text{-Al}_2\text{O}_3$ was too small to be determined by the lattice parameter shift of $\alpha\text{-Al}_2\text{O}_3$. Then, the relative amounts of titanium compounds remaining in fired bodies were measured by X-ray diffractometry using a step-scanning technique which can detect less than 0.1 wt% rutile or Al_2TiO_5 in $\alpha\text{-Al}_2\text{O}_3$, and were compared with the amount of TiO_2 . The solid solution of TiO_2 in $\alpha\text{-Al}_2\text{O}_3$ was found above 1150°C, and the solubility was estimated to be 0.27 wt% at the temperature range from 1300° to 1700°C. Beyond the solubility limit, excess TiO_2 coexisted with $\alpha\text{-Al}_2\text{O}_3$ as rutile below 1350°C and as Al_2TiO_5 above 1450°C. The sintering of $\alpha\text{-Al}_2\text{O}_3$ was markedly promoted when TiO_2 was added up to the solubility limit and the fired density higher than 97% of the theoretical was obtained at 1400°C. The addition of TiO_2 also promoted the grain growth of $\alpha\text{-Al}_2\text{O}_3$. But beyond the solubility limit, the grain size decreased with an increase of Al_2TiO_5 . Therefore it is inferred that Al_2TiO_5 existing as a second phase retards the grain growth of $\alpha\text{-Al}_2\text{O}_3$. The lattice parameters of Al_2TiO_5 in fired bodies considerably differed from those of a single crystal. It is explained as due to the difference of thermal expansion coefficient between Al_2TiO_5 and $\alpha\text{-Al}_2\text{O}_3$. [Received August 13, 1985]

High-Pressure Self-Combustion Sintering of SiC from Fine Mixed Powders of Silicon and Carbon

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A dense $\beta\text{-SiC}$ sintered body was fabricated using a new process, high-pressure self-combustion sintering (HPCS). By using this process, SiC can be simultaneously synthesized and sintered directly from mixed reactants of silicon and carbon without any sintering aids by initiating the exothermic reaction under high pressure. The conversion ratio to SiC was improved over 99% and the mean grain diameter in the compact could be reduced below 1 μm when submicron fine powders of silicon and carbon were used as starting elements. The relative density, Vickers microhardness and fracture toughness were 92-93% of theoretical, 27 GN/m² and 4.5 MN/m^{3/2} respectively. [Received August 21, 1985]