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GAS PRESSURE SINTERING OF β -SIALON WITH Z=3

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| 16. Abstract An experiment was conducted on beta-sialon in atmospheric pressure, using a temperature of 2000°C and 4 MPa nitrogen atmosphere. <p style="text-align: center;">ORIGINAL PAGE IS OF POOR QUALITY</p> | | | |
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GAS PRESSURE SINTERING OF β -SIALON WITH $z=3$

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Silicon nitride, an end component of β -sialon, decomposes by heat of approximately 1900 °C in 0.1 MPa nitrogen atmosphere. In order to inhibit the decomposition, it is required to apply approximately 3 MPa nitrogen pressure at the temperature of 2000 °C. If β -sialon may be fired at the temperature higher than 2000 °C, it will be possible to sinter it without producing the low melting temperature phases such as X phase, and compacts with high resistability against high temperatures may be obtained. */523

(2)

Mitomo has conducted a sintering experiment using β -sialon ($\text{Si}_{6-z}\text{Al}_z\text{O}_z\text{N}_{8-z}$; $z=0-4.2$) in atmosphere pressure, specifically with $z=1$ at the temperature of 1900 °C and 2 Mpa nitrogen pressure, and he obtained compacts of the density 2.99 g/cm³. There has been no reported sintering experiments involving temperatures above 2000 °C, however. We therefore conducted an experiment on β -sialon in the atmosphere pressure, applying the temperature of 2000 °C and 4 MPa nitrogen atmosphere. This paper is a report of our experiment.

In our experiment, $z=3$ was selected for β -sialon compositions, and the compositions containing 0, 2, and 4 equivalent weight % of more oxygen were set respectively as $z=3(0)$, $3(+2)$, and $3(+4)$.

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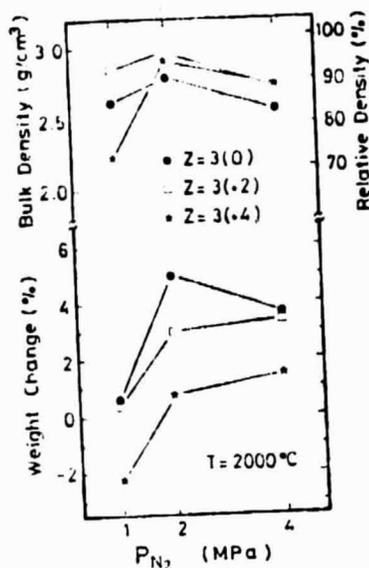


Fig. 1. Effect of nitrogen gas pressure on density and weight change of β -sialon with $z=3$ at 2000°C .

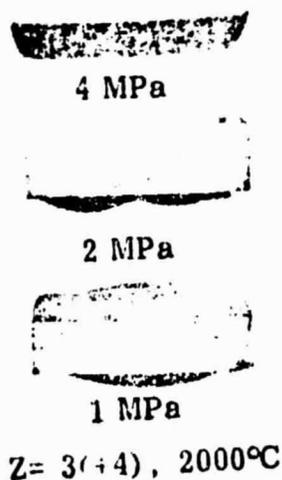


Fig. 2. Side views of β -sialon with $z=3(+4)$ sintered under nitrogen gas pressure of 1, 2 and 4 MPa at 2000°C .

The raw materials that were used in the experiment were Si_3N_4 (TS-7, average granule grade $0.6 \mu\text{m}$) manufactured by Toyo Soda Mfg. Co. Ltd., AlN (average granule grade, $3.0 \mu\text{m}$) manufactured by Toshiba Ceramics Co. Ltd., and Al_2O_3 (RA-40, average granule grade $0.83 \mu\text{m}$) manufactured by Iwata-ni Kagaku Kokyo Co. Ltd. These raw materials were measured at the compositions of $z=3(0)$, $3(+2)$, and $3(+4)$. Then, they were mixed with ethanol as milled by a Si_3N_4 ball for two days, suction filtered, and vacuum dried. The mixed materials, approximately 11 g, were pressure formed by 30×30 mm metal press with the pressure of 98 MPa. Then, they were treated with the stable water pressure of 490 MPa. Thus, powders of approximate density 2.0 g/cm^3 were obtained.

The powders were put in a graphite crucible coated with BN in the order of $z=3(0)$, $3(+2)$, $3(+4)$ from the bottom, around which a granulated composition $z=3(+4)$ powder was filled. This graphite crucible was set in an atmospheric pressure furnace (graphite

heat generator, manufactured by Tooa Shinku Giken KK.) and was fired at 2000 °C in the nitrogen atmospheric pressure of 1, 2, and 4 MPa. The initial pressure of the nitrogen gas was set so that it would reach the expected level at 1100 °C. As for heating, approximately 50 minutes was spent up to 1100 °C, then the temperature was raised at 10 °C/min up to 2000 °C, where the temperature was maintained for 60 minutes before cooling.

The fired compacts were weighed, the surface phase was identified by X-ray diffraction, and the density was measured by water substitution method.

Figure 1 shows the change of compact density and weight. The density, of all $z=3$ (0), 3(+2) and 3(+4) compacts, was the highest when the pressure was 2 MPa, among which $z=3(=2)$ showed the highest density, 2.96 g/cm³ (96.4%). The weight showed a clear increase from 1 MPa to 2 MPa, while the increase between 2 MPa and 4 MPa was slightly decreased.

The surface of the $z=3(0)$ compact was identified as β -sialon and 15R-sialon phases on the top and 15R-sialon on the bottom at 1 MPa, β -sialon phase on the top and β -sialon and 15R-sialon on the bottom at 2 MPa, β -sialon phase on both top and bottom at 4 MPa. As for the compacts of $z=3(+2)$, both the top and bottom contained both β -sialon and 15R-sialon phases at 1 MPa, while a slight amount of X phase was observed along with β -sialon at 2 and 4 MPa. As for $z=3(=4)$, measuring was not conducted on the compact of 1 MPa because it was swollen (Figure 2), and both the top and bottom of 2 and 4 MPa compacts were identified

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as β -sialon. Additionally, some of the compacts contained the phases that were not identified.

There was no swelling observed on the compacts of $z=3(0)$, while slight swelling was observed on the bottom of the compacts of $z=3(+2)$ at 1 and 2 MPa. As for $z=3(+4)$, the swelling was well developed on both the top and bottom at 1 MPa, slight swelling was observed on the bottom at 2 MPa, and no swelling at 4 MPa. (Figure 2) This swelling is considered to have developed by the gas generated by the thermal decomposition. Because this thermal decomposition was inhibited by the increase of the nitrogen gas pressure, it indicates that the atmospheric pressure sintering method is effective for β -sialon sintering.

We plan to report the details of the result of this β -sialon atmospheric pressure sintering in the coming future.

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