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研究速報・Short Communication

Reaction between Al_2O_3 and Silicon in Nitrogen Atmosphere

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窒素雰囲気中における Al_2O_3 とシリコンとの反応

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Mixed powder of Al_2O_3 (particle size : ca. 0.1μ , purity in ig. loss free state $> 99.9\%$, ig. loss : 0.5%) and silicon (particle size : ca. 10μ , purity $> 99.9\%$) in equal weight was pressed in 1 g disk ($15\phi \times 4 \text{ mm}$) and reacted at 1410°C for 10 h in nitrogen atmosphere of 1 atm. The reaction product was consisted of $\beta\text{-Si}_3\text{N}_4$, $\alpha\text{-Al}_2\text{O}_3$, AlN and $\alpha\text{-Si}_3\text{N}_4$. Free silicon was not detected in the reaction product, but weight increment observed after the reaction was lower than the theoretical value, 34% expected from simple nitridation of silicon.

By annealing the sample at 1730 and 1780°C in Si_3N_4 crucible by r.f. heating with molybdenum susceptor under nitrogen atmosphere of 1 atm, results as shown in **Table 1** were obtained. Those data clearly show following phase change being occurred during the initial reaction at 1410°C and the annealing stage at $1730 \sim 1780^\circ\text{C}$.

Those reactions are quite similar to that between silicon and $\text{SiO}_2^{4)}$, and the decomposition of $\text{Si}_2\text{N}_2\text{O}^{5)}$. The weight loss, 45.4% observed at 1780°C for 8 hr heating, just coincides with the value calculated for oxygen free state according to first reaction. Similar consideration may be applied to the other systems of oxide- Si_3N_4 mixture or metal-silicon-oxynitride type compounds or solid solutions, especially in the process of pressureless sintering.

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Table 1. Results observed after the reaction.

Temperature in (°C)	Time in (h)	Weight gain in (%)	Linear shrinkage in (%)	Phases present
1410	10	19.2	0	$\beta\text{-Si}_3\text{N}_4 > \alpha\text{-Al}_2\text{O}_3 > \text{AlN} \sim \alpha\text{-Si}_3\text{N}_4$
1730	2	-12.5	2.6	β' -phase* $> \text{AlN}$ base polytype**
1730	4	-28.7	3.1	$\beta\text{-Si}_3\text{N}_4 \sim d\text{-AlN}^{**} > \text{AlN}$ base polytype
1730	8	-40.2	4.1	$\text{AlN} > \beta\text{-Si}_3\text{N}_4 > \text{AlN}$ base polytype
1780	2	-33.6	4.4	$d\text{-AlN} \sim \beta\text{-Si}_3\text{N}_4 > \text{AlN}$ base polytype
1780	4	-44.9	6.7	$d\text{-AlN} > \beta\text{-Si}_3\text{N}_4 > \text{AlN}$ base polytype
1780	8	-45.4	9.7	$d\text{-AlN} > \beta\text{-Si}_3\text{N}_4 > \text{AlN}$ base polytype

* β' -phase is a solid solution having $\beta\text{-Si}_3\text{N}_4$ type structure²⁾.

** Detailed description was given by K.H. Jack³⁾, $d\text{-AlN}$ indicates faulty AlN.