

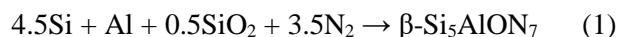
SPARK PLASMA SINTERING OF β -SIALON–BN CERAMIC COMPOSITES*

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Solid solutions of β - Si_3N_4 of general formula $\text{Si}_{6-z}\text{Al}_z\text{O}_z\text{N}_{8-z}$ ($z = 0.0\text{--}4.2$) are known for their excellent hardness, strength, and wear/corrosion resistance, which explains their wide use in various engineering applications [1]. Hexagonal BN exhibit good dielectric properties combined with high thermal conductivity and corrosion resistance at exceeding low wettability with the melts [2]. Addition of h-BN to ceramic composites greatly improves their thermal shock resistance, machinability, and to decrease friction [3-4]. SiAlON–BN composites are highly promising for metallurgical applications such as nozzles and pipes for metal pouring, annular breakers, crucibles, thermocouple casing, etc. In this context, it seemed interesting to develop of new simple and efficient methods for it's fabrication. In this work, we studied the prospects for combining of two advanced techniques: the combustion synthesis (CS) of raw powders with their subsequent spark plasma sintering (SPS). CS is a convenient technique for production of α - and β -SiAlON powders of varied composition, particle size, and morphology [5]. As compared to conventional hot pressing, SPS ensures higher heating rates and short dwell times (several minutes) and to date has been recognized as an effective tool for consolidation of materials [6].

Infiltration- assisted CS of β - Si_5AlON_7 and h-BN powders in nitrogen gas was carried out by the following schemes:



Green mixtures also contained some amount of diluents, β - Si_5AlON_7 and h-BN respectively, in order to improve extent of conversion. Combustion was performed in a 2-L reactor at $P(\text{N}_2) = 2\text{--}4$ MPa for synthesis β - Si_5AlON_7 fine powders with a mean particle size of $0.6\text{--}1.0$ μm and at $P(\text{N}_2) = 8\text{--}10$ MPa for synthesis β - Si_5AlON_7 coarse powders with a mean particle size of $3.5\text{--}4.0$ μm and flaky h-BN particles. Aliquot amounts of combustion synthesized raw powders were intermixed in a high-energy planetary steel-ball mill (800 rpm, ball/mill ratio 10 : 1, $\tau = 5$ min). Then milled powders were sintered in a Labox 625 SPS facility under vacuum (below 10 Pa). The heating rate was 50 deg/min. The sintered compacts were heated from room temperature to 600°C without applied load and then to 1550–1800°C at a compressive stress of 50 MPa. The compacts were held at a desired temperature (T_{max}) for 5 min.

For pure β - Si_5AlON_7 powders, the densification intensified markedly at $T > 1400^\circ\text{C}$. That just corresponded formation of the liquid eutectics caused by surface oxide impurities. The degree of dispersion was main factor determining the shrinkage rate. For fine β - Si_5AlON_7 high-density ceramics was achieved at $T_{\text{max}} = 1550^\circ\text{C}$. While relative density (ρ_{rel}) of coarse β - Si_5AlON_7 sintered at same condition was about 0.75–0.77. The addition of h-BN improves the compactibility. Under a compressive stress of 50 MPa at 600°C, the initial value of ρ_{rel} exceeds 0.8 for the compact containing 30 wt % h-BN, and 0.5–0.6 for that of pure β - Si_5AlON_7 . As the result, high-density structure of sintered β - Si_5AlON_7 -BN composites was achieved at $T_{\text{max}} = 1550^\circ\text{C}$ for all used raw powders. It is important sintered composites had different structure and strength. The flexural strength (σ_f) of fine-grained composites with homogenous distribution of flaky h-BN particles was in the range 450–600 MPa. A marked decrease in σ_f (200–250 MPa) was registered for coarse-grained composites where small flaky h-BN particles were mainly distributed over the surface of larger β - Si_5AlON_7 ones.

REFERENCES

- [1] T. Ekstrom, and M. Nygren, "Sialon ceramics", J. Am. Ceram. Soc., vol. 75, no. 2, pp. 259–276, 1992.
- [2] R. Haubner, M. Wilhelm, R. Weissenbacher, and B. Lux, "Boron nitrides: Properties, synthesis, and applications", Struct. Bonding, vol. 102, no. 1, pp. 1–45, 2002.
- [3] R. Shuba, and I.-W. Chen, "Machinable α -SiAlON/BN composites", J. Am. Ceram. Soc., vol. 89, no. 7, pp. 2147–2153, 2006.
- [4] D. Wei, Q. Meng, and D. Jia, "Mechanical and tribological properties of hot-pressed h-BN/ Si_3N_4 ceramic composites", Ceram. Int., vol. 32, no. 5, pp. 549–554, 2006.
- [5] G. Liu, K.X. Chen, and J.T. Li, "Combustion synthesis of SiAlON ceramic powders: A review", Mater. Manuf. Processes, vol. 28, no. 2, pp. 113–125, 2013.
- [6] M. Tokita, "Recent and future progress on advanced ceramics sintering by spark plasma sintering", Nanotechnol. Russ., vol. 10, nos. 3–4, pp. 261–267, 2015.

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