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Formation Mechanism of Boron Rich Compounds Nanoparticles in Induction Thermal Plasma Processing

誘導結合型熱プラズマにおける多ホウ化物ナノ粒子の生成機構

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Induction thermal plasma was used to prepare nano-sized boron rich compounds and the mechanism of nanoparticle formation was investigated by estimating the homogeneous nucleation rate. In order to synthesize AlB_{12} and YB_{66}, the premixed feed powders of Al, B and YB_4 were injected into the thermal plasma in different compositions. High boron contents and thermal conductive helium were favorable to enhance AlB_{12} and YB_{66} yields in the final product due to a high boiling point of boron.

1. Introduction

Boron rich compounds are attractive materials because of their high melting point, hardness, high electrical conductivity and high corrosion resistance to molten material. Therefore, their nanoparticles have received great attention for the applications of polishing material, neutron shield material and catalyst of fuel cell. Some reports about the preparation of boride nanoparticles have been published previously; YB_{66} nanoparticles were prepared by plasma chemical process using starting powders of YB_4 and boron [1].

Induction thermal plasmas have been widely used for powder treatment of the nanoparticles synthesis. Induction thermal plasma has some advantages of high enthalpy to enhance reaction kinetics, high chemical reactivity, large volume with low velocity, oxidation, and reduction atmospheres in accordance with required chemical reactions and rapid quenching to produce chemically non-equilibrium materials.

The purpose of this paper is to prepare nano-sized boron rich compounds and to analyze the formation mechanism of boron rich compounds.

2. Experimental procedure

Figure 1 shows a diagrammatic illustration of experimental apparatus. The set-up consists of a plasma torch, a reaction chamber and a power supply (4 MHz). Plasma operating condition were as follows; plate power of 30 kW, operating pressure at atmosphere, sheath plasma gas mixture of Ar-He, inner plasma gas of He, and carrier gas of Ar. Raw powder material used in this study are boron (average particle size: 45 μm), YB_4 (9.2 μm), and aluminum (15 μm). The composition ratio of the feeding powders was changed in the experiments to find an optimal condition.

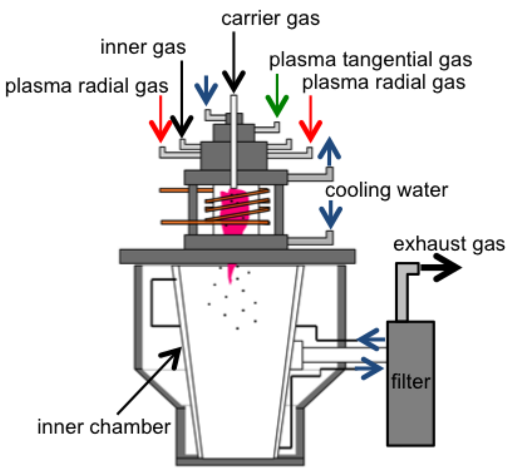

Fig. 1. Schematic diagram of experimental set-up.

Phase and size distribution of the prepared nanoparticles were analyzed by x-ray powder diffraction (XRD) and transmission electron microscopy (TEM).

3. Experimental results and discussion

3.1 *YB_4 and boron system*

The XRD patterns of the prepared nanoparticles for YB_4-B system are shown in Fig. 2. YB_{66} and YB_4 are identified from the XRD spectrum. The successful preparation of yttrium boride was attributed to evaporation enhancement of raw materials in thermally conductive helium as the inner gas.

3.2 *Aluminum and boron system*

The XRD patterns of the prepared nanoparticles for Al-B system are shown in Fig. 3. AlB_{12}, AlB_{10}, boron, and aluminum are observed TEM photograph of the prepared nanoparticles for Al-B system is shown in Fig. 4. The particle distribution of Fig. 5 evaluated from the TEM presents the average particles size of 20.8 nm with geometrical standard deviation of 1.03.

3.3 *Homogeneous nucleation temperature*

The nucleation rate expression proposed by Girshick *et al.* [2] was used for investigation of the formation mechanism. Particle formation can be observed when the nucleation rate exceeds 1.0 cm^{-3} s^{-1}. Therefore, the corresponding value of saturation ratio is defined as the critical saturation ratio [3].

The nucleation temperature at the critical saturation ratio is presented in Fig. 6 according to the raw materials. The nucleation temperature of boron is higher than those of aluminum and yttrium, accordingly boron is nucleated in first. After the boron nucleation, AlB_{12} and YB_{66} are formed by condensation of aluminum and yttrium to the boron nuclei, because the boiling points of aluminum and yttrium is higher than boron nucleation temperature.

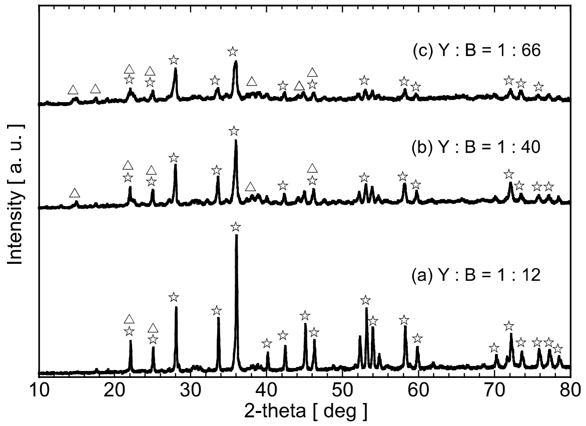

Fig. 2. XRD spectrum chart of prepared particles for YB_4-B system; Composition ratio of Y and B: (a) 1:12, (b) 1:40, (c) 1:66 (☆ : YB_4, △:YB_{66}, □:B).

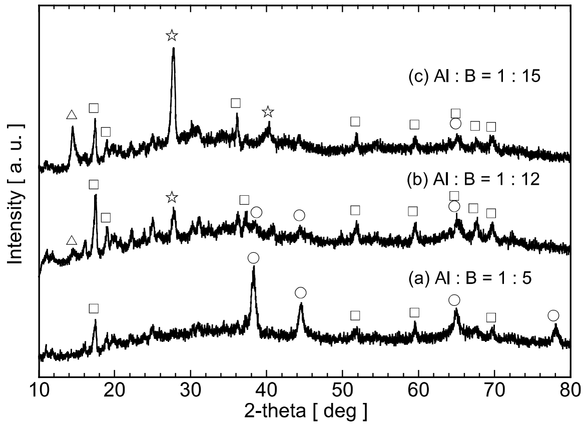

Fig. 3. XRD spectrum chart of prepared particles for Al-B system; Composition ratio of Al and B: (a) 1:5, (b) 1:12, (c) 1:15 (☆ : AlB_{12}, △:AlB_{10}, □:B, ○:Al).

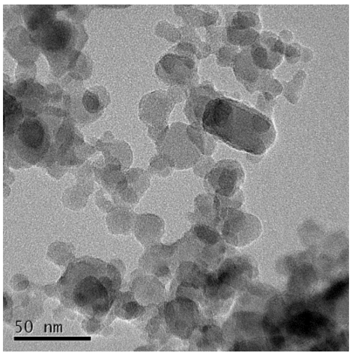

Fig. 4. TEM photograph of nanoparticles: Al:B = 1:15.

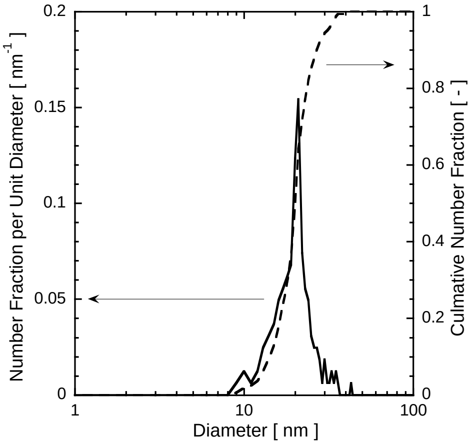

Fig. 5. Particle size distribution of nanoparticles: Al:B = 1:15.

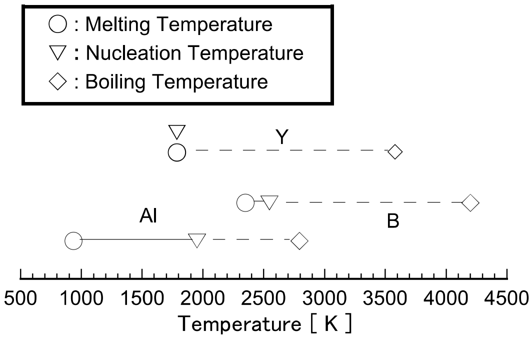

Fig. 6. Nucleation, boiling and melting temperature.

4. Conclusion

Nano-sized YB_{66} and AlB_{12} are successfully synthesized by the induction thermal plasma. Injection of helium as the inner gas promotes the yield of boron rich nanoparticles. High boron contents in the raw material were favorable to enhance YB_{66} and AlB_{12} in the final product.

References

[1] J.Y. Huang, et al.: J Mater Sci. **33** (1998) 4141.

[2] S.L. Girshick, et al.: Aerosol Sci and Technol. **13** (1990) 465.

[3] S.K. Friedlander: Smoke, Dust and Haze, Wiley-Intersience, (1977) 241.