# Yttrium segregation behavior to necks during sintering in alumina doped with yttria

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Yttrium segregation of $Al_2O_3$ doped with 0.1mol% of $Y_2O_3$ was investigated at the sintering temperature of 1100 and 1200°C in order to examine the segregation behavior of doped yttrium. High angle annular dark-Field scanning transmission electron microscopy (HAADF-STEM) observations confirmed that doped yttrium exists at alumina particle surfaces over the whole at the sintering temperature of 1100°C. At the sintering temperature of 1200°C, yttrium segregates at necks formed during an initial stage of sintering. The segregated yttrium retards shrinkage rate during sintering.

Alumina is widely used as structural ceramic materials due to the high heat resisting property. Mechanical property of alumina sinters at high temperature plays an important role for the practical use. The high-temperature creep resistance in $Al_2O_3$ is known to be highly improved by doping a small amount of yttria. In the fully sintered $Al_2O_3$ doped with yttria, it was already reported that the doped yttria segregates at grain boundaries[1)], and the segregated yttria results in increasing covalent bonding to obtain prominent creep resistance. However, the process of segregation behavior of yttria has not been clarified yet. This study aims to analyze the microstructure and yttria segregation near necks formed at an initial stage of sintering by HAADF-STEM and TEM.

The materials used in this study were 0.1 mol% $Y_2O_3$ doped $Al_2O_3$, which were sintered at 1100 and 1200°C in air. The $Y_2O_3$ was doped as yttrium acetate solution to $Al_2O_3$ raw powder. Sintering was conducted with conventional process of sintering. TEM/HAADF-STEM observations were performed to analyze yttrium segregation behavior in the sintered samples.

Figure 1 shows TEM bright field images taken from 1100°C and 1200°C samples. The grain sizes in respective samples are almost the same, indicating that the grain growth does not occur at the initial sintering stage. However, necks, *i.e.*, contacting areas at adjacent grains, are confirmed to be formed clearly at 1200°C. Fig. 2 are HAADF-STEM micrographs showing the necks of the two samples. It's noted that bright line contrast can be clearly observed with very narrow width only at 1200°C. This fact means that doped yttrium ions segregate to the necks at 1200°C because contrast intensity in HAADF-STEM is approximately proportional to the square of the atomic number. In contrast, such contrast increment can not be seen at 1100°C. Therefore, yttrium segregation can be considered to start between 1100°C and 1200°C. To know decomposition of yttrium acetate used on doping to the raw powder, we carried out in-situ observation for yttrium acetate in TEM. When yttrium acetate was

heated with W-wire in TEM, a crystallization containing yttrium dissolved and spread out about 800°C. Considering the obtained results, doped yttrium existed in the alumina particle surface over the whole upto the sintering temperature of 1100°C, and finally segregates at the necks at least at 1200°C.

Acknowledgement

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References

1) H.Yoshida,et. al., J.Mater.Res.Vol.13,No.9,2597-2601(1998)

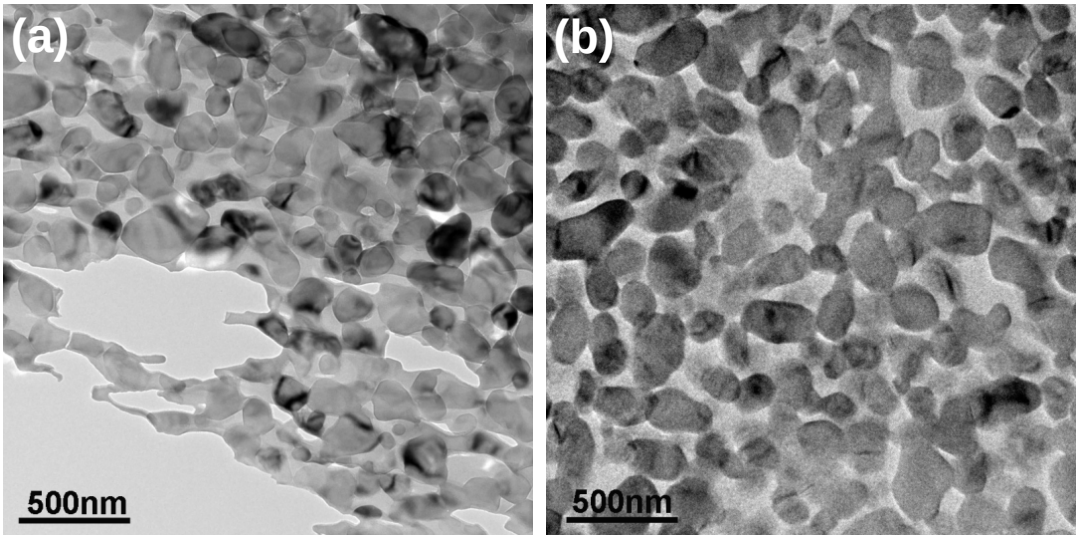


**Fig.1** TEM bright field images 0.1 mol% $Y_2O_3$-doped $Al_2O_3$ sintered at (a) 1100°C, and (b) 1200°C.

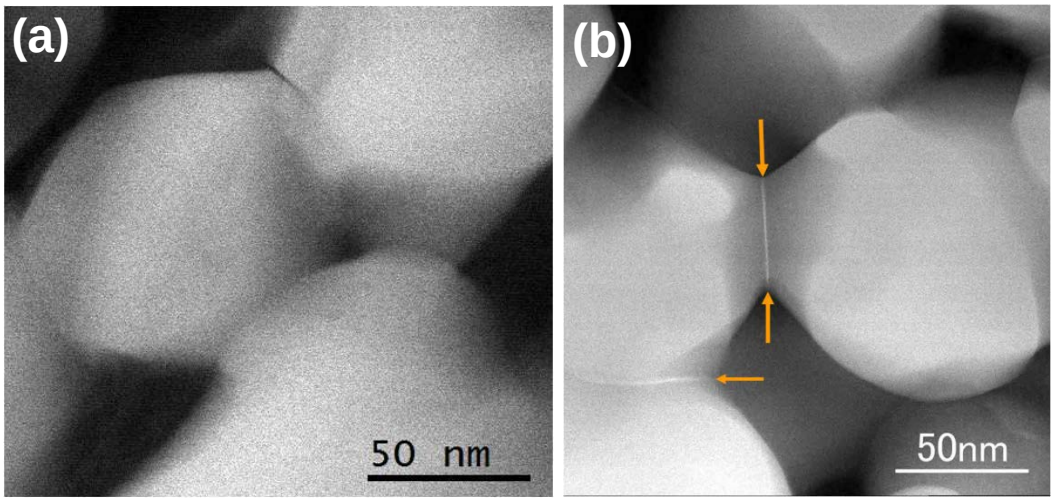


**Fig.2** HAADF-STEM images of 0.1 mol% $Y_2O_3$-doped $Al_2O_3$ sintered at (a) 1100°C, and (b) 1200°C. In (b), the arrows are showing line contrasts.