DOI: 10.1017/S1431927606068942
Microsc Microanal 12(Supp 2), 2006

MICROSTRUCTURE OF TRANSPARENT NANOCRYSTALLINE $MgAl_2O_4$ CERAMICS

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Transparent $MgAl_2O_4$ spinel ceramic has been considered as an important optical material due to its high melting point, good mechanical strength, high resistance against chemical attack and excellent optical properties[1]. It has attracted a growing interest from both defense and civil industries. One problem with transparent ceramics that has been gradually realized for actual applications is its poor mechanical property, i.e. brittleness. However, the traditional toughening methods can not be used since it decreases the transparency. One promising approach is to make nano-ceramic materials which have both the high transparency and good mechanical properties[2].

$MgAl_2O_4$ powders are synthesized with a low-cost high-temperature-calcination method, and transparent $MgAl_2O_4$ nano-ceramics have been sintered using these nanocrystalline powders at relatively low temperatures under high pressure. High purity $NH_4Al(SO_4)_2$•$12H_2O$ and $MgSO_4$•$7H_2O$, with a molar ration of 2:1, were mixed and then put into a muffle oven for calcination at 1150 ºC for several hrs. Sintering experiment of $MgAl_2O_4$ ceramics were performed in a six-pressure-source cubic anvil device at 500~700^{o}C under 2~5 Gpa. The microstructure of powders with different calcinations time is shown in Figure 1. It can be seen that with the increasing of calcinations time, the powders crystallized into larger particles with uniform size of about 35 nanometers. The ceramic sintering experiments show that the powder calcined for 4 or 6 hrs is easier to be sintered to transparent nano-ceramics.

Fig. 2 shows the typical optical appearance and transmission spectrum of $MgAl_2O_4$ ceramic specimens prepared at different conditions. All of the samples sintered under 500 ºC are opaque. From 540 to 700 ºC, the sintered samples are transparent with a light brown color. Above 700 ºC, the influence of carbon diffusion becomes significant, resulting in a sharp decrease of transparency. Two absorption peaks emerge at 1300 and 1800 nm, respectively. These peaks have not been observed in conventional transparent ceramics, indicating a novel optical absorption mechanism from nano-structure. Fig.3 shows the TEM images of the highly transparent nano-ceramic prepared at 620 ºC/3.7GPa. Based on the TEM images, the nano-ceramic has a fine-grained, crystalline structure with dense grain boundaries and a few nano-sized pores mainly exist in triple junctions of the grains. For the nano-grained polycrystalline ceramic, both lower grain scattering and lower pore scattering can be responsible for the high transparency.

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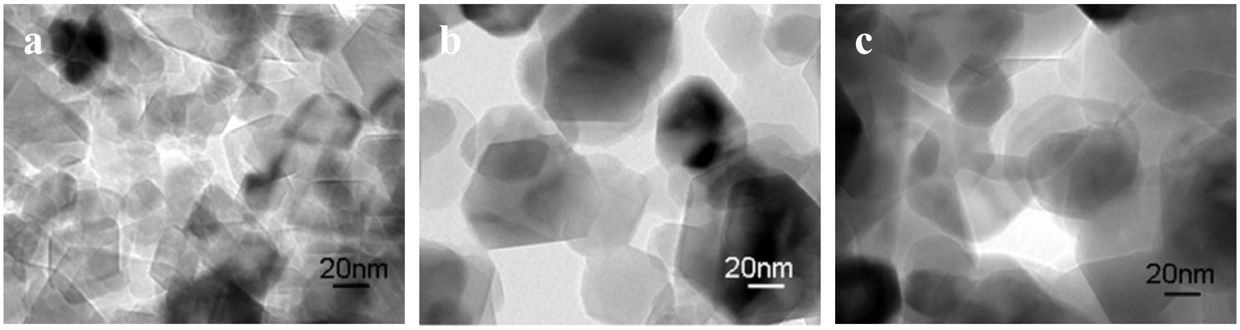

Fig. 1 TEM images of nano-sized $MgAl_2O_4$ spinel powders calcined at 1150°C for 2 hrs (a), 4 hrs (b) and 6 hrs (c).

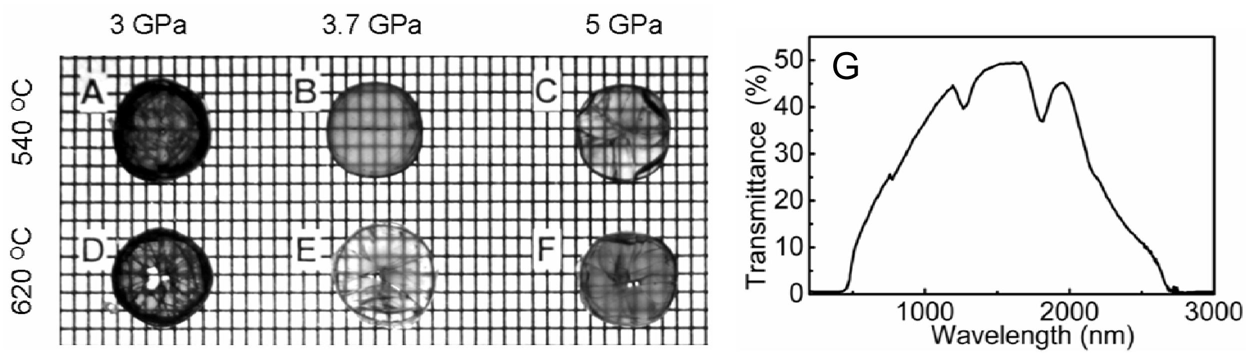

Fig. 2. Optical micrographs showing the appearance and transparency of $MgAl_2O_4$ nano-ceramics prepared at 540ºC/3GPa (A), 540ºC/3.7GPa (B), 540ºC/5GPa (C), 620ºC/3GPa (D), 620ºC/3.7GPa (E), 620ºC/5GPa (F), respectively. (G) is a transmission spectrum of sample B.

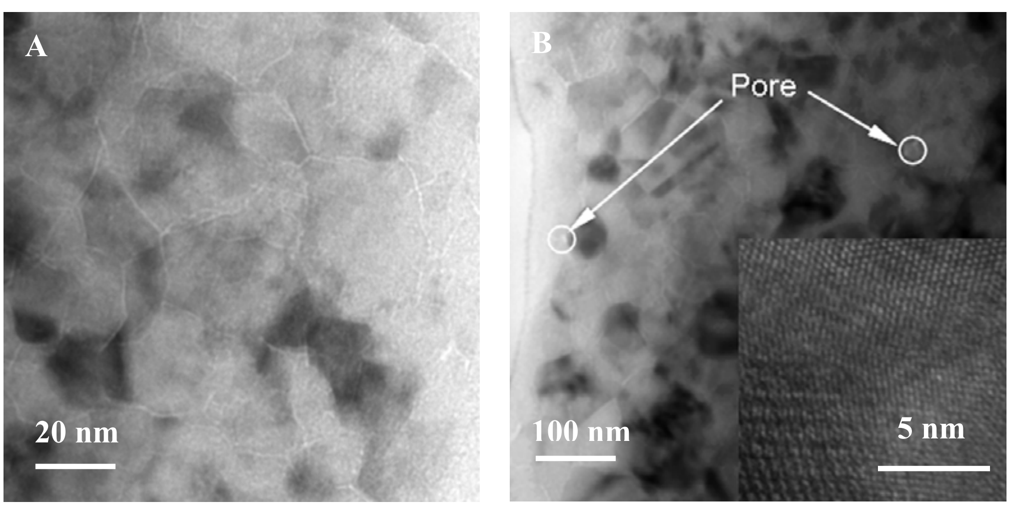

Fig. 3 TEM images of transparent $MgAl_2O_4$ ceramic prepared at 620ºC/3.7 Gpa. A shows the nanoparticles; B shows the grain boundaries (inset) and nano-sized pores.

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