MICROSTRUCTURE OF TRANSPARENT NANOCRYSTALLINE MgAl₂O₄ CERAMICS


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Transparent MgAl₂O₄ 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. 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 high transparency and good mechanical properties.

MgAl₂O₄ powders are synthesized with a low-cost high-temperature-calcination method, and transparent MgAl₂O₄ nano-ceramics have been sintered using these nanocrystalline powders at relatively low temperatures under high pressure. High purity NH₄Al(SO₄)₂•12H₂O and MgSO₄•7H₂O, with a molar ration of 2:1, were mixed and then put into a muffle oven for calcination at 1150 °C for several hrs. Sintering experiment of MgAl₂O₄ ceramics were performed in a six-pressure-source cubic anvil device at 500~700 oC 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 MgAl₂O₄ ceramic specimens prepared at different conditions. All of the samples sintered under 500 °C are opaque. From 540 to 700 °C, the sintered samples are transparent with a light brown color. Above 700 °C, 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 °C/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.

Fig. 1 TEM images of nano-sized MgAl₂O₄ 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 MgAl₂O₄ 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 MgAl₂O₄ ceramic prepared at 620ºC/3.7 Gpa. A shows the nanoparticles; B shows the grain boundaries (inset) and nano-sized pores.