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Microstructure of composite YAG crystal/ceramics

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Abstract

Transparent composite YAG crystal/ceramics were synthesized by solid-state reaction method using high-purity Y_2O_3, Al_2O_3 powders as raw Q1 materials. The mixed slurry was dried, sieved, and cold-isostatically pressed with Nd:YAG crystal under a pressure of 250 MPa. The mixed powder compacts were sintered at 1780 °C for 10 h under vacuum and annealed at 1450 °C for 20 h in air. The microstructure of YAG crystal/ceramics ceramics was studied with SEM and EPMA, which showed there was an intermediate layer between Nd:YAG crystal and YAG ceramics. HRTEM image and corresponding SAED patterns studied showed that the intermediate layer was the YAG ceramics grain that grew along Nd:YAG crystal orientation and has become one part of crystal.

Keywords: YAG; Transparent ceramics; Composite ceramics; Microstructure

1. Introduction

Recent developments of laser diodes (LD) pumped solid-state lasers have stimulated the development of high gain mediums [1–3]. Among the high gain mediums, the neodymium (Nd)-doped yttrium aluminum garnet (YAG) is one of the most available laser materials. Neodymium-doped $Y_3Al_5O_{12}$ (Nd:YAG) crystals are widely used as the pumping medium in solid-state lasers [4]. YAG is a garnet and crystallizes in the cubic system. Nd:YAG has the formula $\{Nd_xY_{1-x}\}_3Al_5O_{12}$, where Nd^{3+} substitutes for eight-coordinate Y^{3+} as shown in Fig. 1 [5].

Since 1984, polycrystalline ceramic laser materials have attracted much attention [6–9] because the optical quality has been improved greatly and highly efficient laser output could be obtained. The efficiencies are comparable or superior to those of single crystals. It is well-known heat effect that has bad impact on the laser efficiency and beam quality of high power and large energy solid state lasers, composite structure materials such as bonded crystal and composite ceramics could resolve the heat effect efficiently. As a result, the preparation and performance study of composite structure materials has become a hot research topic [10].

The aim of this paper is to report the results of the synthesis and properties of YAG crystal/ceramics composite transparent ceramics using commercial Y_2O_3 and Al_2O_3 powders by a high energy ball-milling method. Emphasis is laid on the microstructures of grain and intermediate layer of YAG crystal/ceramics ceramics.

2. Ceramic fabrication

High-purity powders of Al_2O_3, and Y_2O_3 were used as starting materials. The starting powders were weighed to result in a chemical composition of YAG and mixed by ball-milling in anhydrous alcohol for 10 h, with an addition of 0.5 wt.% tetraethyl orthosilicate (TEOS) as sintering aid separately. The mixtures were dried, sieved, dry-pressed with Nd:YAG crystal (here we use Nd:YAG not YAG in order to distinguish from YAG ceramics) under 100 MPa into Ø 20 mm disks and finally one piece of YAG crystal/ceramics was cold-isostatically pressed under 250 MPa.

The compacted YAG crystal/ceramics disks were sintered at 1780 °C under vacuum and then annealed at 1450 °C for 20 h in air. Mirror-polished samples on both surfaces were used to measure optical transmittance and absorbed spectrum. The microstructure of YAG crystal/ceramics composite ceramics

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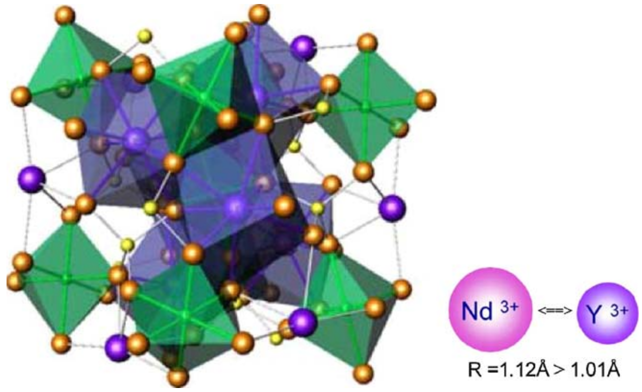

Fig. 1. Nd:YAG atomic structure.

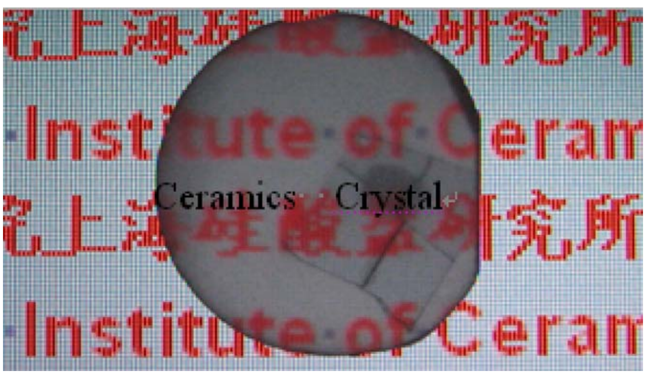

Fig. 2. Photographs of composite YAG crystal/ceramics.

was studied using electron probe micro-analysis (EPMA), energy dispersive analysis system of X-ray (EDX) and high resolution transmission electron microscopy (HRTEM).

3. Results and discussion

Fig. 2 shows the transparent mirror-polished YAG crystal/ceramics sample sintered at 1780 °C under vacuum and then annealed at 1450 °C for 20 h in air. The Nd:YAG crystal is surrounded by YAG ceramics. The Nd:YAG crystal is split into four pieces because during the sintering period there is a constriction in YAG ceramics while no constriction in YAG crystal. If the crystal is on one face of the ceramics the force of constriction will pull the crystal crush. Putting the crystal at the center of the ceramics can avoid crush.

Table 1
Quantitative analysis of No. 1 and No. 2 areas by energy spectrum.

Weight %	Y	Al	Nd	O
No. 1	51.56 ± 0.95	22.40 ± 0.51	0	26.03 ± 1.04
No. 2	50.27 ± 0.99	22.54 ± 0.53	2.50 ± 0.50	24.70 ± 1.07

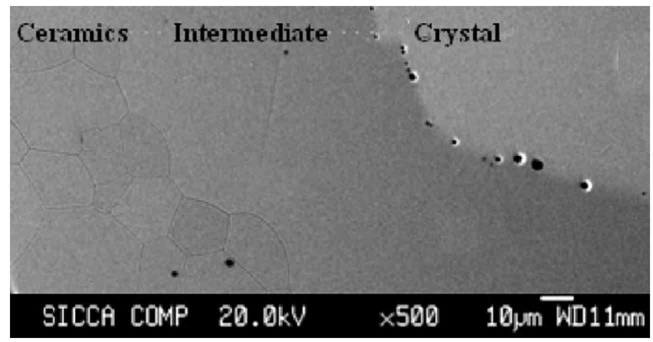

Fig. 4. EPMA micrographs of thermal etched surfaces of composite YAG ceramics/crystal.

Fig. 3 shows the EPMA microstructure of mirror-polished surface of the composite YAG ceramics/crystal specimen and EDX spectra analysis. From it we can see there is a string of hole at the interface between crystal and ceramics. During the sintering period the crystal acts as a seed and the ceramics around it grows along the crystal so the growth speed is very fast and the air can hardly be exhausted.

In order to confirm the interface between crystal and ceramics EDX spectra analysis was used. We select two areas: one is at the left of the hole strings and the other is at the right. Table 1 gives us the quantitative analysis of the two areas by energy spectrum. According to the data from Table 1 we can see that there is no Nd ion in area No. 1 which is YAG ceramics area while there are some Nd ions in area No. 2 which is Nd:YAG crystal. This indicates the hole strings are the boundary of

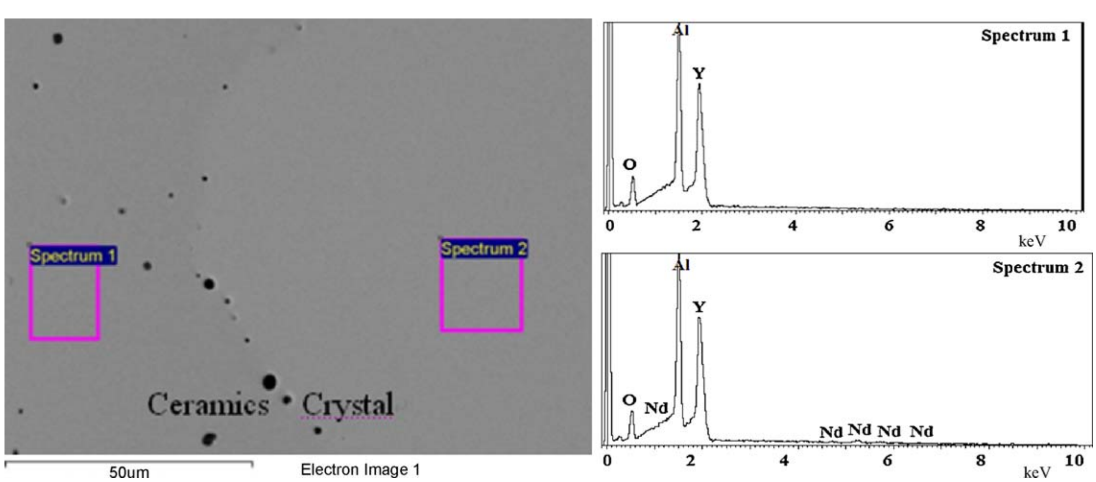

Fig. 3. EDX spectra analysis of composite YAG ceramics/crystal.

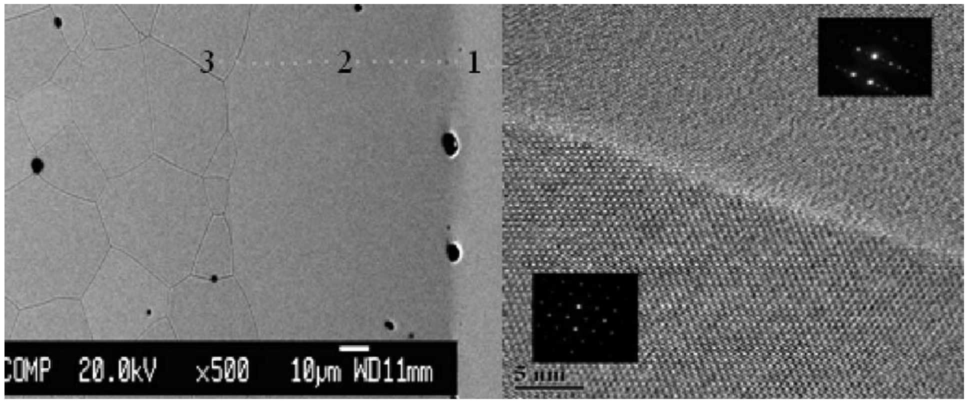

Fig. 5. HRTEM image of Nd:YAG ceramics with corresponding SAED patterns as inset.

crystal and ceramics. The Al and Y contents in the ceramics grains were examined and remained unchanged across the grain boundary, indicative of an even distribution of the elements.

Fig. 4 shows the EPMA micrographs of the mirror-polished and thermal etched surfaces of Nd:YAG/YAG composite transparent ceramics sintered at 1450 °C. It could be seen there was an intermediate layer between YAG ceramics and crystal. The width of the intermediate layer is about 100 μm. There are no interfaces in the intermediate layer as in the ceramics. The structure of the intermediate layer is quite the same as that of the YAG crystal.

As the microstructure shown in Fig. 4 there are some obvious pores in or between the grains. Pores are the main factor in the transparency of ceramics. The grains are all about 20 nm and there is no abnormal grain growth (AGG) in ceramics. AGG is detrimental to the condensation of the ceramics, as they can enwrap pores. It is very important to control the grain size to obtain fully transparent Nd:YAG ceramics. The intermediate layer between YAG ceramics and crystal can be treated as AGG. This is the reason why there are so many pores between the intermediate layer and Nd:YAG crystal.

Fig. 5 shows the HRTEM micrographs and corresponding SAED patterns of the Nd:YAG/YAG composite transparent ceramics sintered at 1450 °C. We examined the SEAD patterns from the Nd:YAG crystal to the intermediate layer and then from the ceramics. It showed the intermediate layer has the same crystal plane index which is (1 1 0). The right figure shows the grain in the intermediate layer which is (1 1 0) and in ceramics which is (4 3 3).

4. Conclusions

YAG crystal/ceramics composite transparent ceramics were fabricated successfully by a solid-state reaction method. The microstructure of YAG crystal/ceramics ceramics was studied with SEM and EPMA, which showed that there was an intermediate layer between Nd:YAG crystal and YAG ceramics. During the sintering period the crystal acts as seed and the ceramics around it grows along the crystal so the growth speed is very fast and the air can hardly be exhausted. HRTEM image and the corresponding SAED patterns were studied and showed the intermediate layer has the same crystal plane index as the Nd:YAG crystal which is (1 1 0). This means the intermediate layer was the YAG ceramics grain that grew along Nd:YAG crystal orientation and has become part of crystal.

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