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Heat treatment of transparent Yb:YAG and YAG ceramics and its influence on laser performance

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ABSTRACT

Composite transparent ceramic materials are promising for improving the performance of high–average-power lasers. A combination of room-temperature bonding *via* surface treatment by a fast atom beam and diffusion bonding *via* heating, which effectively controls the ion diffusion distance near the interface, makes the laser materials suitable for a variety of oscillator/amplifier. During the heat treatment of yttrium aluminum garnet (YAG) ceramics, the Si ions in the solid solution of the sintering aid incorporated within the grains were seen to segregate at the grain boundary, resulting in an increase of scattering sites. The number density and size of the scattering sites strongly depended on the post-heating temperature rather than the heating time. Specifically, heating at 1300 °C did not affect the transmittance of the YAG ceramic, whereas both the size and number of scattering sites substantially increased with a heat treatment at 1400 °C. The laser oscillation experiment using cryogenically-cooled Yb:YAG ceramics exhibited heating temperature dependence of the slope efficiency owing to the increasing scattering loss.

1. Introduction

Composite ceramics are a very useful technology to realize efficient operation of high-repetition-rate, high-average-power solid state lasers owing to the freedom that composites provide in the amplifier design [1,2]. Further, many studies have reported the use of composite ceramics such as a thin Yb-doped ceramic bonded on a non-doped bulk supporting ceramic for efficient cooling [3,4], a high-power Nd:YAG (yttrium aluminum garnet) laser rod with non-doped end caps for realizing uniform pumping region [5,6], an Nd-doped core surrounded by non-doped YAG for reducing the thermal lens effect [7], an Nd:YAG ceramic rod with non-doped YAG cladding for reducing thermal birefringence and the lens effect [8], an Nd:YAG rod with Sm:YAG cladding and a square Yb:YAG with Cr^{4+}:YAG cladding for the suppression of parasitic oscillations [9,10], a Yb:YAG/Cr^{4+}:YAG composite disk for Q-switch operation [11] and large ceramic materials fabricated from smaller pieces using compositing techniques. In contrast to the conventional optical bonding of precisely polished surfaces of glass or crystals, such composite techniques produce strong bonding of laser materials that do not detach under relatively low thermal stress.

Two methods have been reported to improve the bonding strength of composite YAG ceramics [12–17]. Yagi et al. have reported a two-step sintering method [12], wherein two primary bodies were first pre-sintered in vacuum at 1400 °C to a density of 85%–90% of the bulk YAG ceramics, whereupon the two surfaces polished to a roughness of λ/10 (λ = 633 nm) were placed in contact and sintered in a manner similar to that of YAG ceramic fabrication. Because the grains near the contact interface grow and merge across the interface while avoiding any degradation of the optical property, a substantial bonding strength enhancement can be obtained compared with that of the usual diffusion bonding at relatively low temperatures. The measured diffusion length of Nd^{3+} ions was 18 μm for the Nd:YAG/YAG composite ceramic, which was five times greater than that for diffusion bonding of the Nd:YAG crystal and a non-doped YAG crystal. Sato et al. have reported another approach for composite ceramics [13] where powdered composite materials with different Nd dopant densities were individually pressed, stacked into one piece by cold isostatic pressing, and then sintered to form the composite ceramic. Using this method, Nd:YAG

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ceramics were produced possessing a density gradient of doped Nd ions.

The composite ceramics fabricated by these two methods have been customized by manufacturers for individual specialized use, but to extend the composite technology to a wide range of applications it is desirable to improve the bonding strength and to control the ion diffusion distance across the composite interface. For this purpose, we have demonstrated a YAG composite ceramics based on the combination of room-temperature bonding *via* surface treatment by a fast atom beam (FAB) and the usual diffusion bonding with heating. In this paper, we report an increased light scattering in this YAG composite that is dependent on the heating condition, as well as demonstrate the influence of the scattering sites on the laser oscillation performance.

2. Bonding process and measurement of light scattering

The samples bonded herein were Yb:YAG and non-doped YAG ceramics (commercially-available products fabricated by Konoshima Chemical Co. Ltd., Japan), where the Yb doping density was 9.8 at%. The size of ceramic disk was 17.6 mm in diameter and 3.5 mm in thickness. One of the disk surfaces was ultra-precisely polished for bonding, while the other surface was optically polished for observation. The surface flatness and root-mean-square (rms) roughness were ~14 nm PV (*via* interferometric measurement) and ~10 nm rms (*via* atomic force microscope measurement), respectively. The FAB equipment used was an Ar-FAB (Ayumi Industry Co. Ltd., Japan) [18,19] possessing two atom beam sources, where each beam was individually used for surface activation of samples set in parallel with each other at a distance of 5 cm. After evacuation of the Ar-FAB to $\sim 1 \times 10^{-5}$ Pa, the sample surfaces were irradiated by the Ar atom beam for 5 min with an acceleration voltage of 1 kV. The two samples were then bonded at room temperature with a load of 5 kgf·cm for 5 min. After the room-temperature bonding process, the samples were heat-treated in vacuum at 2×10^{-3} Pa in an electric furnace with heating conditions (temperature/period) of 1100 °C/50 h, 1400 °C/10 h, 1400 °C/50 h and 1600 °C/50 h.

Fig. 1 shows photographs of the bonded samples (9.8 at%-doped Yb:YAG/non-doped YAG) for different post-heating conditions, wherein all of the samples were completely bonded and thus did not exhibit Newton's rings. The upper images in Fig. 1 were obtained after the post-heat treatment in vacuum, where the Yb ions were reduced to Yb^{2+}.

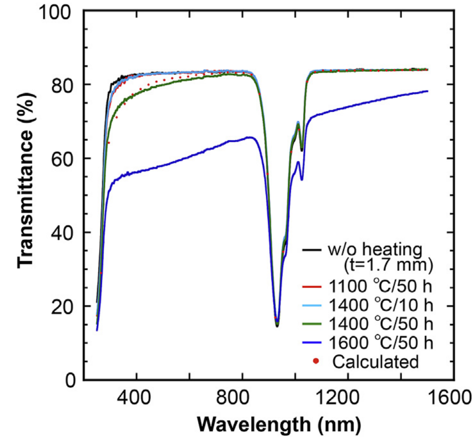

Fig. 2. Transmission spectra of composite ceramics shown in Fig. 1. The curve fit (dotted line) is based on Rayleigh scattering.

These samples were then thermally annealed in atmosphere at 1350 °C for 10 h, permitting the conversion of the Yb ions back to Yb^{3+} (bottom images in Fig. 1). The light scattering does not increase after the annealing process [20]. Fig. 2 shows the linear transmission spectra of the bonded samples measured by a spectrophotometer (HITACHI U-4100). The transmission spectra of samples with post-heating conditions of 1100 °C/50 h and 1400 °C/10 h were almost equivalent to that of the sample with no post-heating, whereas a small reduction of transmittance in a wavelength range below 800 nm was observed with the post-heating condition of 1400 °C/50 h, which implies the creation of small scattering sites. An overlaid dotted line shows a fitting with the transmission reduction inferred using the wavelength-dependent cross section of Rayleigh scattering. When the heating temperature was increased to 1600 °C, the transmission image appears dim (Fig. 1) and the significant degradation of transmittance was obvious in the entire spectral range of 300–1500 nm. From these observations, one can note that the reduction of transmittance is strongly dependent on the heating temperature rather than heating time, and the transmission spectra is likely affected by the size of the scattering sites.

The bonded samples were then cut perpendicular to the contact interface, and the cut surface was optically polished to observe the

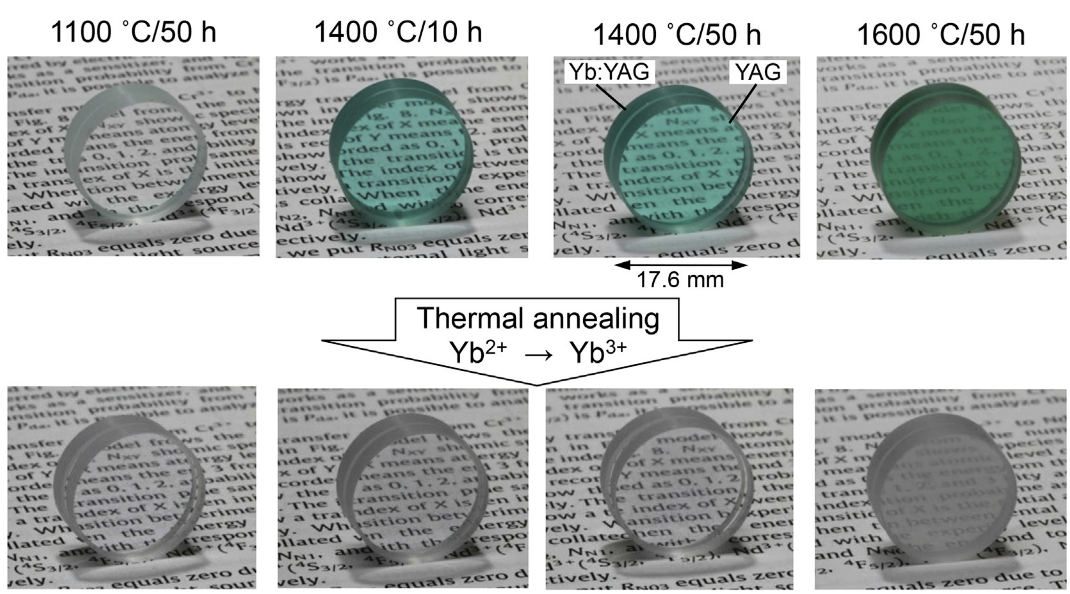

Fig. 1. Composite ceramic samples (Yb:YAG/non-doped YAG) fabricated by two-step bonding process consisting of room-temperature bonding *via* Ar-FAB surface treatment and diffusion bonding *via* post-heating. (Upper) After post-heating treatment in vacuum. (Lower) After further annealing in atmosphere at 1350 °C for 10 h. The size of ceramic disk is 17.6 mm in diameter and 3.5 mm in thickness.