53. Evaluation for the bonded region of a direct bonded Ti:sapphire crystal

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Direct bonding without the use of adhesives was demonstrated on Ti:sapphire laser crystals with a bonding surface of 12 mm x 6 mm and the bonded region was evaluated from the macroscopic to the atomic level by three different methods. Wavefront distortion caused by the bonded region of 10 mm x 5 mm was estimated at 0.031 wavelengths (λ) at 633 nm. Micro defect measurements by a laser tomography method showed that the number of micro defects on the bonded region were much smaller than that of the intrinsic ones inside the crystal. From a magnified inspection, atoms in the bonded region were well arranged with the same regularity as inside the crystal. In addition, we discussed reasons of ion distribution around the bonded interface measured by quantitative EDX analysis for three elements composed of Ti:sapphire crystal.

Keywords: Direct bonding, Laser crystals, Ti:sapphire, Laser tomography, Transmitted wavefront, TEM, EDX

1. INTRODUCTION

To overcome the requirement for the enlargement of laser crystals with high optical quality that are used in CPA laser systems, we have studied a new method, direct bonding of laser crystals without using adhesives. In the development, evaluation of bonded interface from the macroscopic to the atomic level is essential for the use of bonded crystals in laser systems. In this report, several kinds of bonded interface in Ti:sapphire crystals are presented. The measurements were carried out by three different methods: (1) transmitted wavefront distortion measurements by a Fizeau interferometer; (2) micro defects measurements by a laser tomography method; (3) magnified inspections by a transmission electron microscope (TEM) equipped with an energy dispersive X-ray (EDX) analyzer.

3. EXPERIMENTS

3.1. Transmitted wavefront distortion

Figure 1 shows an experimental set up for measurements of transmitted wavefront distortion of the specimen by a Fizeau interferometer (Zygo: GPI-XP). In order to devise accurate measurements with the system, the bonded crystal was located on the narrow space between transmission and reference flat mirrors to reduce the interference fringe motion by the fluctuation of airflow. Since the measured wavefront data in this system implies the roughness of the polished surfaces and distortion of the bonded region as well as inhomogeneity inside the specimen, other single Ti:sapphire crystals, having the same surface flatness of over 0.1 λ at 633 nm on both Brewster's ends and a different length of 8 mm and 18 mm, were also measured to estimate the crystal inhomogeneity. The estimated inhomogeneity was 0.037 λ/cm at 633 nm from the measurements of the other crystals, and it was assumed that the transmitted wavefront of the single crystal having with the same length as the bonded one is 0.121 λ at 633 nm. And, the transmitted wavefront of the bonded crystal was 0.152 λ at 633 nm. As a result, the P-V wavefront distortion at the bonded region was 0.031 λ at 633 nm.

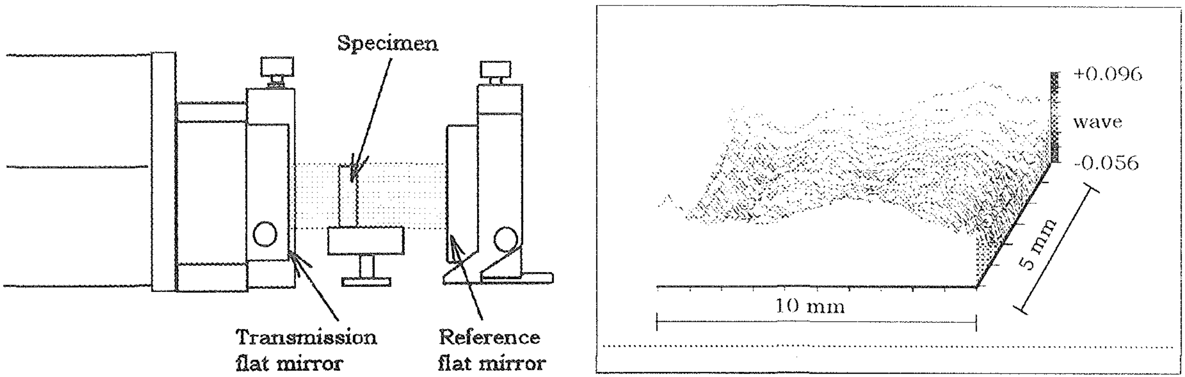

Figure 1 Experimental set up and the transmitted wavefront pattern measured in the area of 10 x 5 mm.

3.2. Micro defects measurements

Figure 2 shows a schematic layout for optical scattering measurements of the bonded Ti:sapphire by a laser tomography system (Mitsui: MO-421). Laser tomography has been used as one of the attractive non-destructive methods to detect micro defects in crystals by means of optical scattering.[1,2] The system consists of an optical unit and a data analyzer. In the optical unit, the bonded specimen located on a 3-D motion variable stage was irradiated by a probe laser of the 2nd harmonic of CW Nd:YAG. The focused probe laser beam on the specimen was about 10μm in diameter. Optical scattering signals generated from defects inside the specimen were vertically detected by a CCD camera through an optical microscope. A color glass filter was inserted between the CCD camera and the microscope to avoid the effect of fluorescence in the scattering measurements.

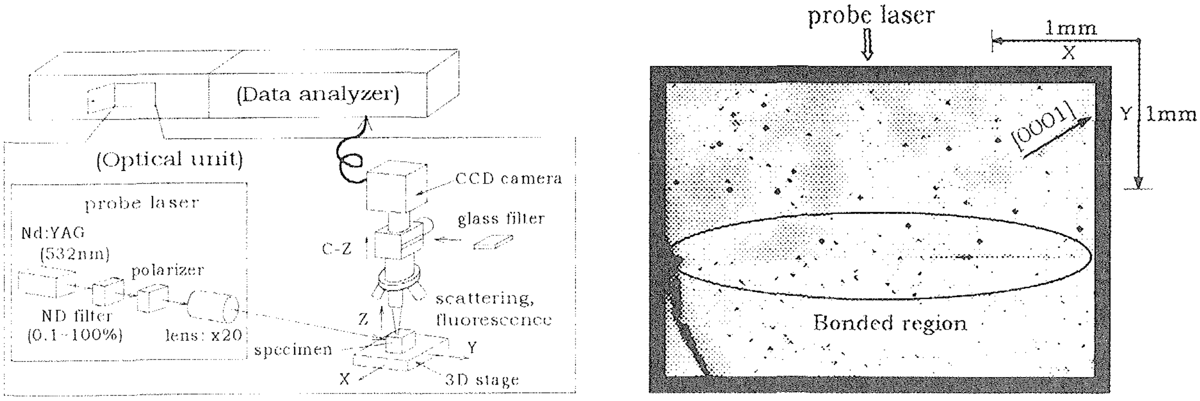

Figure 2 Laser tomography method for scattering measurements around the bonded region.

Detection of the signal was synchronized with the step motion in X direction of the stage and each set of data was transferred to the data analyzer where the whole image of micro defects within a viewing area is framed from the stored data. In Fig. 2, small spots appearing horizontally inside the oval shows the micro defects existing on the bonded surface, and the other spots spread through the whole image were intrinsic defects formed in the crystal growth. It can be seen that the number of micro defects on the bonded surface are much smaller than that of the intrinsic ones, which suggests that the optical loss at the bonded region of Ti:sapphire was insignificant and the bonded crystal was applicable for laser oscillation.[3]

3.3. Magnified inspections in the atomic level

The bonded region was also inspected by a field emission TEM (Hitachi: HF-2000) equipped with an EDX (Kevex: Sigma-EDX) at a magnification power of 4-million. In the measurements, additional fabrication using argon ion milling was required to prepare a specimen extracted from the bonded Ti:sapphire that was already used for the impact fracture test. Thickness of the specimen was reduced to around 70 nm in the bonded area. A probe electron beam accelerated by 200 kV was used for TEM measurements. In the quantitative analysis of elements by EDX with K_α lines, three elements of oxygen, aluminum and titanium were selected and detected in each 5 nm of diameter around the bonded region. Specific X-rays of these elements are 0.525 keV for O, 1.486 keV for Al and 4.508 keV for Ti, respectively. Fig. 3 shows the magnified image of bonded region. Although obscure parts of 1 nm size slightly appeared along the bonded interface, the atoms in the bonded region were well arranged with the same regularity as inside the crystal. It seemed that the obscure parts were based on the irregularity of the polished surfaces of the Ti:sapphire crystals. In the bonding process, most of the irregularity would be reduced by elastic deformation and diffusion of the atoms during the long heat treatment while the rest of large irregularity could be less deformed by these effects.

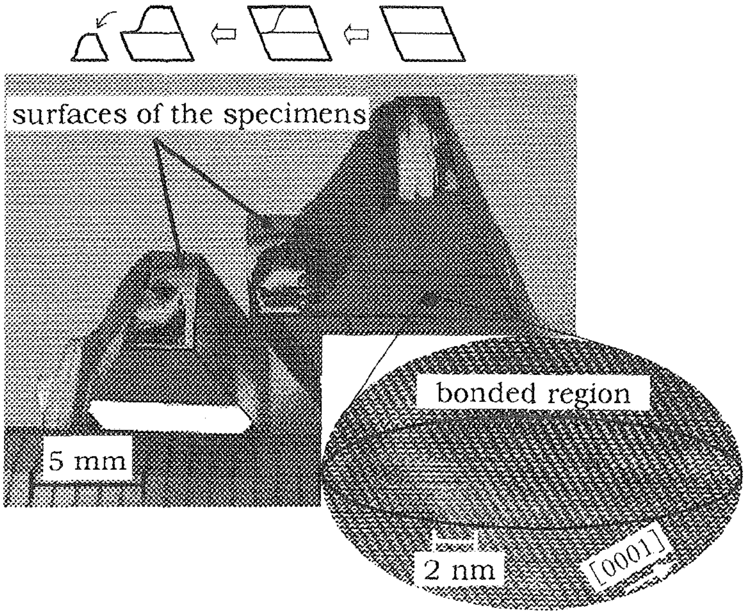

Figure 3 Magnified image of bonded Ti:sapphire.

4. RESULTS AND DISCUSSION

The detection limit of quantitative analysis by EDX measurement is about 0.1-wt. %. On the other hand, the measured specimen has only 0.15-wt. % of titanium oxide (Ti_2O_3) in sapphire. Therefore, it would be difficult to clearly detect Ti if the prescribed doped concentration is correct and the laser crystal has been grown homogeneously. Certainly, the measured X-ray intensities of Ti were quite small and the same as the noise level in most parts of the crystal. However, larger intensities of Ti were measured along the bonded interface, shown in Fig. 4. The Ti concentration at the interface was nearly four times higher than other parts of the crystal on average, while the variation of the concentration of Al and O shows the same tendency along the direction of distance from the interface, and the highest concentration in both elements was found at 5 nm from the interface.

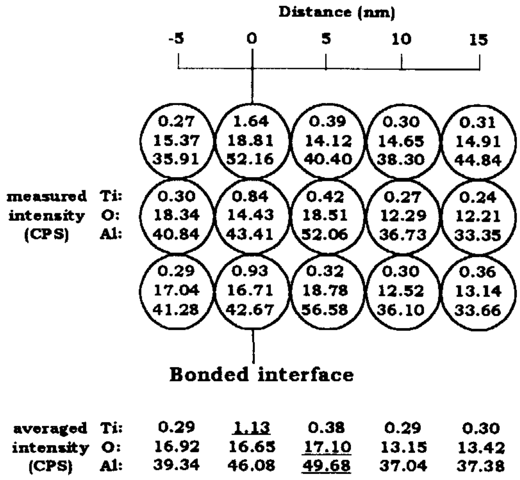

Figure 4 Measured intensities of three elements at each position.

Concerning the polished surfaces of the specimens before starting the bonding process, we have not performed the same EDX analysis. Although we have this unknown factor, we deduce the reason for Ti precipitation on the bonded interface from the following facts and assumptions:

a. The crystal has quite a small amount of Ti ions, (O : Al : Ti = 1409 : 938 : 1).
b. Heat treatment at 50 h and 1373 K in vacuum was made for the contacted specimens.
c. Fig. 2 shows Ti:sapphire has large amount of intrinsic defects based on impurities in the initial material and/or oxygen vacancies. Under this condition, dislocation of atoms easily occurs inside the crystal.
d. Ion radii of three elements with coordinate numbers of 6 are 0.51 Å for Al^{3+}, 1.46 Å for O^{2-}, 0.76 Å for Ti^{3+} and 0.68 Å for Ti^{4+}, respectively.
e. The chemical binding energy of Al-O is smaller than that of Ti-O (Al-O: 222 kJ/mol, Ti-O: 305 kJ/mol).

Diffusion coefficients (D) of Al and O are on the order of 10^{-20} and 10^{-22} m^2/sec at 1373 K in sapphire, respectively. From these coefficients, the time duration of heat treatment (t) in sec and the equation of $L = (6tD)^{1/2}$, the diffusion length (L) for the atoms are estimated to be 100 nm for Al and 10 nm for O. Although the diffusion coefficient of Ti has not been measured in sapphire, the coefficient of Ti would be less than that of Al since the radius of the Ti ion is larger than that of Al. It is assumed that positively charged ions of Al and Ti can mainly travel through the defects inside the crystal by means of thermal diffusion, while negatively charged O ions stay in a local field, vibrating with high frequency peculiar to the light element. Since the initial contacted interface has an irregular atomic arrangement, the surface energy would be high. To reduce the energy, the diffusion of the atom easily occurred at the interface and positively charged ions were attracted to negative O ions during the heat treatment. Furthermore, thermal expansion by the heat treatment stimulated partial compression and contraction at the irregular atomic arrangement along the interface, which accelerated the Gorsky effect which induces condensation of Ti towards the interface as the impurity ion of sapphire. As the Ti-O bond breaks harder than the Al-O bond, the dislocation of Ti ions bound with O ions would be restricted. On the other hand, Al ions are liable to separate from O ions and travel inside the crystal. As a result, the maximum concentration of Ti appeared at the bonded interface. Then, traveling Al ions are mainly attracted O ions to secure the electric charge compensation, which leaded to the result of the same tendency of concentration variation in both Al and O ions, as shown in Fig. 4.

5. CONCLUSION

The direct bonding of Ti:sapphire laser crystal was demonstrated. The bonded region was evaluated from the macroscopic to the atomic level by three different methods such as transmitted wavefront distortion measurements by the interferometer, micro defect measurements by laser tomography and magnified inspections by TEM equipped with EDX. From the first interferometer experiments, the wavefront distortion caused by the bonded region was estimated at 0.031 λ at 633 nm. The second micro defect measurements showed the number of micro defects on the bonded region were much smaller than that of the intrinsic ones inside the crystal. It suggests the previous results of optical inspection and laser oscillation in ref. 3. From the third inspections, the atoms in the bonded region were well arranged with the same regularity as inside the crystal although obscure parts 1 nm in size appeared slightly along the bonded interface. In the EDX quantitative analysis, it was found that the maximum Ti concentration appeared on the bonded interface and the concentration was nearly four times larger than the other parts of the crystal on average. The mechanism of the Ti ion precipitation was also considered by atomic diffusion and the Gorsky effect on the bonded region during our bonding process.

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