Irradiation Conditions for Fiber Laser Bonding of HAp-Glass Ceramics with Bovine Cortical Bone

Shigeru TADANO[1*], Satoshi YAMADA[1], Masaru KANAOKA[2]

[1] Division of Human Mechanical Systems and Design, Faculty of Engineering, Hokkaido University, N13 W8, Kita-ku, Sapporo, Hokkaido 060-8628, Japan.
[2] Mitsubishi Electric Corporation, 1-14, Yada-minami, 5-chome, Higashi-ku, Nagoya, Aichi 461-8670, Japan.

*Corresponding Author:
Shigeru TADANO, PhD
Professor, Division of Human Mechanical Systems and Design, Faculty of Engineering, Hokkaido University
N13 W8, Kita-ku, Sapporo, Hokkaido 060-8628, Japan
Tel/Fax: +81-11-7066405
E-mail: tadano@eng.hokudai.ac.jp

Abstract:

BACKGROUND: Orthopedic implants are widely used to repair bones and to replace articulated joint surfaces. It is quite important to develop an instantaneous technique for direct bonding of bone and implant materials.

OBJECTIVE: The aim of the study was to develop a laser bonding technique for bone and ceramics as an implant material.

METHODS: Ceramic specimens (10 mm diameter and 1 mm thickness) were sintered with hydroxyapatite and MgO-Al_2O_3-SiO_2-glass powders mixed in 40:60 wt% proportions. A small hole was bored at the center of the ceramic specimen. The ceramic specimen was positioned onto the bovine bone specimen and a 5 mm diameter area of the ceramic specimen was irradiated using a fiber laser beam (1070-1080 nm wavelength).

RESULTS: The bone and ceramic specimens could be bonded strongly under the irradiation conditions of 400 W laser power and 1.0 s exposure time. The maximum shear strength was 5.3 ± 2.3 N. A bonding substance was generated around the hole in the ceramic specimen; it penetrated deeply into the bone specimen.

CONCLUSIONS: A ceramic specimen was bonded to a bone specimen using a fiber laser instantaneously and the irradiation conditions required to bond these materials were investigated.

(193 words)

Keywords:

Bone, Biomechanics, Fiber laser, Bonding, Ceramics, Hydroxyapatite

1. Introduction

Orthopedic implants are widely used to repair bones and to replace articulated joint surfaces. Implant surgeries are performed only by highly specialized and trained surgeons and require substantial time. It is quite important to develop an instantaneous easy technique for direct bonding of bone and implant materials to reduce the burden on patients. Laser processing is an effective technique for rapid bonding of different materials with a high positional accuracy.

Laser processing has already been used in clinical applications for bone and dental hard tissue surgeries, especially within the context of oral surgery and implant dentistry [1, 2]. The conventional mechanical process using burrs and saws in osteotomy has been replaced with a laser cutting process using carbon dioxide (CO_2) and erbium-doped yttrium aluminum garnet (Er:YAG) lasers. Lasers offer some advantages, such as a lack of contact, short duration, and a minimally invasive process.

The authors first proposed the possibility of bonding bone and ceramic specimens in vitro using laser irradiation [3]. In the previous study, a thin bovine bone specimen placed onto a ceramic specimen, which consisted of tricalcium phosphate

(TCP) and MgO-Al_2O_3-SiO_2-glass powders to prevent cracking and breakage due to the thermal expansion, was irradiated using a CO_2 laser beam as a trial. Both specimens were melted locally and bonded by the consequently generated microporous foam-like substance.

The previous study demonstrated bonding of cortical bone and ceramic specimens through irradiation; however, the shear strength of the bond was quite low, at only up to 0.8 N. For clinical applications, the irradiation of ceramic placed onto bone with the laser beam is required (i.e., conversely to the previous experiments) and the influence of irradiation conditions on the shear strength of bonding must be investigated. As a bioceramic, hydroxyapatite [$Ca_{10}(PO_4)_6(OH)_2$, HAp] has been applied clinically for implant materials because of its excellent biocompatibility [4, 5]. Moreover, the CO_2 laser beam used in the previous method is difficult to propagate over a long range, but the fiber laser is more easily propagated over a long range using fiber cables.

The aim of the current study is to advance the technique for laser bonding of bone tissues and bioceramics. In this study, ceramic specimen placed onto a bone specimen were bonded to the bone specimen using a fiber laser and the irradiation

conditions needed to bond the materials and to maximize bonding strength were investigated. A bonding substance generated during irradiation was observed by optical microscopy and scanning electron microscopy (SEM) to determine how it bonds the ceramic specimen to the cortical bone. Further, the influence of irradiation conditions on the shear strength of the bonding was investigated.

2. Materials and Methods

2.1 Specimens

Cortical bone specimens (approximately 20 mm long, 20 mm wide, and 4 mm thick) were taken from bovine femoral diaphysis frozen at -35 ℃ until use. The specimens were immersed in a saline solution until just before the laser irradiation experiment.

To prepare the ceramic specimens, HAp powder (HAP-100: Taihei Chemical Industrial, Japan) and MgO-Al_2O_3-SiO_2-glass powder (MgO 22.2 mol%, Al_2O_3 20.3 mol%, and SiO_2 57.6 mol%: Tokan Material Technology, Japan) were mixed in 40:60 wt% proportions and the powders were compression-molded into specimens with a 10

mm diameter and 6 mm thickness. The compact was sintered at 1200 °C for 90 min and subsequently cut into 1 mm thick slices using a slow-speed diamond wheel saw (Model 650: South Bay Technology Inc., USA).

Because the ceramic specimen placed onto a bone specimen were irradiated with a laser beam and it was necessary to locally melt both the ceramic and the cortical bone specimens, a hole was bored at the center of the ceramic specimen to allow the laser beam to pass directly through to the bone specimen. To investigate the effects of the hole size on bonding, the study evaluated three hole sizes: diameters of 1.0, 2.0, and 3.0 mm.

2.2 Bonding process

The ceramic specimen was positioned onto the cortical bone specimen and secured using a steel jig with a large opening. As shown in Fig. 1, a fiber laser beam (1070-1080 nm wavelength) was generated by an ytterbium laser system (YLS-2000: IPG Photonics, USA) and was focused 34 mm above the surface of the ceramic specimen using a lens. A 5 mm diameter area on the surface of the ceramic specimen

was irradiated using the laser beam, as the center of the laser beam corresponded to the center of the hole in the ceramic specimen. Laser irradiation powers of 100, 200, 300, 400, and 500 W were evaluated, with an exposure time of 1.0 s, to determine the influence of irradiation conditions on bonding and shear strength.

2.3 Observation and elemental analysis of bonding area

The specimen was bisected at the laser-irradiated point and the cross section was observed microscopically (VH5000: KEYENCE, Japan). Furthermore, SEM images of the bonding area cross section were obtained (JSM-6460LA: JEOL, Japan). Energy dispersive X-ray spectrometry (EDS) was also performed at regions of the cortical bone specimen, ceramic specimen, and bonding substance in the SEM images by using an EDS system (JED-2300: JEOL, Japan) attached to the scanning electron microscope. The mass fraction of each element was calculated for each of the three regions.

2.4 Fracture test

Share fracture tests were conducted using a material testing machine (Model 3365: Instron, USA) with a 50 N load cell to measure the shear strength of the bonding between the ceramic and the cortical bone specimens. The cortical bone specimen was fixed with a vise (Fig. 2) and the bonded ceramic specimen was subjected to a force applied using a steel plate until it was removed. The resistance force was recorded using a load cell, with the maximum resistance force indicating the shear strength of the bonding.

3. Results

Figure 3 shows the overhead view of irradiated point, which in this case was a 2.0 mm diameter hole in the ceramic specimen. The bone and ceramic specimens were bonded by fiber laser irradiation with 300 or 400 W laser power and 1.0 s exposure time. Figure 4 shows the cross-section of the laser-irradiated point as observed microscopically. The laser irradiation melted both the ceramic and bone specimens, resulting in the generation of both a void in the bone beneath the hole and a bonding substance around the hole in the ceramic. The melted region where the bonding

substance was generated was about 4 mm in diameter. The bonding substance spread into the surface of the void in the bone, creating the bond.

Figure 5 shows the SEM images of the bonding area in the cross section shown in Fig. 4. In Fig. 5 (b), the upper region indicates the ceramic specimen and the lower region indicates the bone specimen. To enlarge the boundary region between the ceramic and the bone specimens, as shown in Figs. 5 (c) and (d), the bonding substance protruded deeply into the bone specimen.

Figure 6 shows the weight fractions, or percent weights, of constituent elements in the bone specimen, ceramic specimen, and bonding substance at the three positions depicted in Fig. 5. The bonding substance consisted of elements from the bone, such as oxygen, calcium, phosphorus, and carbon, and elements in the ceramic specimen, including magnesium, aluminum, and silicon.

Figure 7 shows the typical resistance force behavior of the bonding during the fracture test, as measured using the load cell. At the maximum resistance force, the ceramic specimen was removed from the bone specimen through fracture of the bonding substance, and this force value was defined as the shear strength of the bonding.

Figure 8 shows the shear strength of the bonding produced using 300 and 400 W laser powers, where the hole in the ceramic specimen was 2.0 mm in diameter. Seven specimens were tested under each set of conditions. The shear strength at 300 W laser power was 3.3 ± 1.0 N, and that at 400 W was 5.3 ± 2.3 N. The shear strength produced using 400 W laser power was 1.6 times that generated by 300 W laser power, although the standard deviation also increased. Additional experiments were also conducted at 400 W laser power and 1.0 s exposure time.

Figure 9 shows the difference in shear strength among the three hole sizes evaluated. The shear strength generated using a 1.0 mm diameter hole was 3.8 ± 1.3 N, which was lower than the strengths generated using 2.0 mm and 3.0 mm diameter holes. The standard deviation using 3.0 mm diameter hole was larger than that using 2.0 mm diameter hole.

4. Discussion

In this study, a small hole was bored at the center of the ceramic specimen to allow the laser beam to pass directly through to the bone specimen and the ceramic

specimen placed onto a bone specimen could be bonded to the bone specimen using a fiber laser irradiation instantaneously. The bonding strength in the current study became more than six times higher than the strength in the previous method [3].

The ceramic specimens used in this study consisted of HAp and MgO-Al_2O_3-SiO_2 glass. Because HAp has a high thermal expansion coefficient, laser irradiation may easily generate cracks and break the HAp specimen [3]. To prevent cracking and breakage, glass, which has a lower thermal expansion coefficient than HAp, was added to the ceramic specimen. The glass component in the ceramic contributed to excellent bonding between the bone and ceramic specimens. These specimens were melted and bonded by fiber laser irradiation and bonding substance was generated around the hole in the ceramic, developing greater resistance to shear forces. Additional study should be conducted to assess the biocompatibility of HAp-glass ceramics and the bonding substance generated during laser irradiation, for example, cell cytotoxicity assessments and animal experiments.

The generated bonding substance protruded deeply into the bone specimen, as shown in Fig. 5. This structure may enhance the shear strength of bonding between the

two specimens. The structure of the bonding substance in this case was different from that generated in the previous study. In the previous study, the bonding substance was a microporous foam-like material, like a rivet, generated within the melted area and the bonding strength was quite low [3]. In that case, the thin bone specimen placed onto the ceramic specimen was irradiated using a CO_2 laser beam and gas produced by disintegrated collagen components in the bone may have been blown against the melted MgO-Al_2O_3-SiO_2-glass in the ceramic, accounting for the porosity of the bonding substance. In the present study, although the microporous structure was partially identified, the bonding substance spread into the surface of the void in the bone. The morphological differences of the bonding substances may depend on the irradiated material - bone or ceramic - potentially resulting in increased shear strength of the bonding substance. Furthermore, the difference in wavelength between fiber and CO_2 lasers may also affect the properties of the bonding substance and the subsequent bonding strength. For hard tissue ablation, Er:YAG and CO_2 lasers have frequently been used [1, 2, 6]. However, since the present study is focused on the melting and bonding of bone and ceramics, as opposed to cutting, these wavelengths may adversely affect the

bonding strength.

The bonding substance consisted of elements from both the bone and the ceramic specimens, indicating that the laser irradiation melted both specimens and generated a bonding substance consisting of both materials. As shown in Fig. 6, the weight fraction of calcium in the bonding substance was greater than that in the bone specimen. This may have been caused by a reduction of carbon towing to sputtering during irradiation. The reduction and aspiration of sputtering materials should be examined in additional studies.

Bonding could not be conducted at laser powers less than 200 W or greater than 500 W at a 1 s exposure time. It appeared that there was an optimal incident energy for melting and strong bonding of both materials. In this study, to reduce the laser power for a clinical device, the exposure duration was set as 1 s. Based on these conditions and the results shown in Fig. 8, the optimal incident energy was determined to be 400 J, but this may depend on the melting temperatures and thermal expansion coefficients of both the bone and the ceramic specimens. A lower energy is insufficient to melt the specimens and generate the bonding substance, and a higher energy increases risk of

cracks in the ceramics and strong sputtering. This study identified the optimal irradiation conditions to bond these materials using a fiber laser.

In addition, the influence of the hole size in the ceramic specimen was investigated. The bonding strength generated using a 1 mm diameter hole was lower than that produced using holes of either a 2 or 3 mm diameter. Furthermore, there was no difference in bonding strength between specimens with holes of 2 and 3 mm diameters. The bonding strength was dependent on the incident energy applied directly to the bone specimen through the hole. An appropriate hole size will distribute energy optimally between the bone and the ceramic specimens to enable effective melting and bonding between the materials.

This study investigated the irradiation conditions for fiber laser bonding of HAp-glass ceramics to bovine cortical bone with maximal shear strength. This laser bonding technique will be useful for creation of a temporary joint between bone tissues and implant materials. However, in the present experiments, a heat-damaged region was observed in the cross section (see Fig. 4). Several previous studies have discussed thermal effects of the laser cutting process on bone regeneration and healing [7-11].

Eriksson and Albrektsson pointed out that the inherent regenerative capacity of bone was almost completely extinguished by thermal injury resulting from tissue exposure to a temperature of 50 °C for 1 min [11]. The present study did not measure the temperature of bone specimens during the laser bonding process or thermal effects on bone healing, although the exposure time of laser irradiation was quite short. Eyrich and Majaron et al. sprayed water on the bone during the laser cutting process to reduce thermal damage [8, 10]. Additional study should be conducted to identify potential methods for reduction of heat damage in bone tissue during laser bonding.

5. Conclusions

This study confirmed that fiber laser irradiation at low power, of up to 400 W, could bond HAp-glass ceramics to cortical bone specimens in a short time in vitro. A bonding substance was generated around the small hole in the ceramic; it protruded deeply into the bone specimen, developing greater resistance to shear forces. The constituent elements in the bonding substance were similar to those in the bone and the ceramic specimens. The shear strength of bonding depended primarily on the irradiation

power, i.e., incident energy, and the hole size in the ceramic specimen.

Acknowledgments

This study was supported by the Adaptable & Seamless Technology Transfer Program through Target-driven R&D (A-STEP) of the Japan Science and Technology Agency (JST), under Grant No. AS2311222F. The laser experiments were performed at the Industrial Research Institute of the Hokkaido Research Organization (HRO) and were supported by Mr. Yohei Sakuraba (HRO) and Mr. Satoshi Matsushiro (Hokkaido University).

References

[1] Stübinger S. Advances in bone surgery: the er:YAG laser in oral surgery and implant dentistry. Clinic. Cosm. Invest. Dent. 2010; 2: 47–62.

[2] Deppe H, Horch HH. Laser applications in oral surgery and implant dentistry. Lasers Med. Sci. 2007; 22: 217-221.

[3] Ogita T, Kanaoka M, Todoh M, Tadano S. In Vitro Laser Bonding of Bovine

Cortical Bone Specimen and TCP-Glass Ceramics. J. Biomech. Sci. Eng. 2012; 7: 248-258.

[4] Chai YC, Carlier A, Bolander J, Roberts SJ, Geris L, Schrooten J, Van Oosterwyck H, Luyten FP. Current views on calcium phosphate osteogenicity and the translation into effective bone regeneration strategies. Acta Biomater. 2012; In press.

[5] Oyane A, Wang X, Sogo Y, Ito A, Tsurushima H. Calcium phosphate composite layers for surface-mediated gene transfer. Acta Biomater. 2012; 8: 2034–2046.

[6] Stanislawki M, Meister J, Mitra T, Ivanenko MM, Zanger K, Hering P. Hard tissue ablation with a free running Er:YAG and a Q-switched CO_2 laser : a comparative study. Appl. Phys. B. 2000; 72: 1–6.

[7] Martins GL, Puricelli E, Baraldi CE, Ponzoni D. Bone Healing After Bur and Er:YAG Laser Ostectomies. J. Oral Maxillofac. Surg. 2011; 69: 1214-1220.

[8] Eyrich GKH. Laser-osteotomy induced changes in bone. Med. Laser Appl. 2005; 20: 25–36.

[9] Wallace RJ, Whitters CJ, McGeough JA, Muir A. Experimental evaluation of laser cutting of bone. J. Mater. Proc. Tech. 2004; 149: 557–560.

[10] Majaron B, Šušterčič D, Lukač M, Skalerič U, Funduk N. Heat diffusion and debris screening in Er:YAG laser ablation of hard biological tissues. Appl. Phys. B. 1998; 66: 479-487.

[11] Eriksson RA, Albrektsson T. The effect of heat on bone regeneration: an experimental study in the rabbit using the bone growth chamber. J. Oral Maxillofac. Surg. 1984; 42: 705-711.

Figure and table legends

Figure 1 The laser irradiation system. Fiber laser beam (1070-1080 nm wavelength) generated by an ytterbium laser system (YLS-2000: IPG Photonics, USA) was focused with a lens (focal distance was 150 mm). The surface of a ceramic specimen placed onto a cortical bone specimen was irradiated with a laser beam at the center of the hole. The irradiation distance, which was the distance between the focal point and the ceramic specimen surface, was 34 mm. The diameter of the irradiated area on the ceramic specimen depends on the irradiation distance; this diameter was 5 mm.

Figure 2 Experimental system for obtaining the fracture shear force of the bonding plane between ceramic and cortical bone specimens. The specimen was fixed with a vise and was subjected to shear force from a steel plate using a material testing machine (Model 3365: Instron, USA).

Figure 3 Top view of bonding area as observed using an optical microscope (irradiated

laser power: 400 W, hole size: 2 mm, shear strength: 8.8 N).

Figure 4 Cross-sectional image of the bonding area in the specimen as observed using an optical microscope (irradiated laser power: 400 W, hole size: 2 mm).

Figure 5 SEM images of cross section of the bonding area shown in Fig. 4: (a) optical microscopic image; (b) SEM image at the edge of the bonding area, showing the bonding substance spreading into the void in the surface of the bone; (c) enlarged SEM image of the boundary area between the cortical bone specimen and the bonding substance; and (d) an additional enlarged SEM image of the boundary area.

Figure 6 Percent weights of constituent elements of the bone specimen, ceramic specimen, and bonding substance.

Figure 7 Increasing resistance force of the specimen bonding area during the fracture test as indicated in Fig. 3. The maximum resistance force indicates the shear strength of

the specimen, which was 8.8 N.

Figure 8 Shear strengths at laser powers of 300 and 400 W. Each bar indicates an average for the seven specimens; the errors correspond to the standard deviations.

Figure 9 Influence of hole size in the ceramic specimen on the shear strength. Each bar indicates an average of the seven specimens; the errors correspond to the standard deviations.

Figures and tables

Figure 1

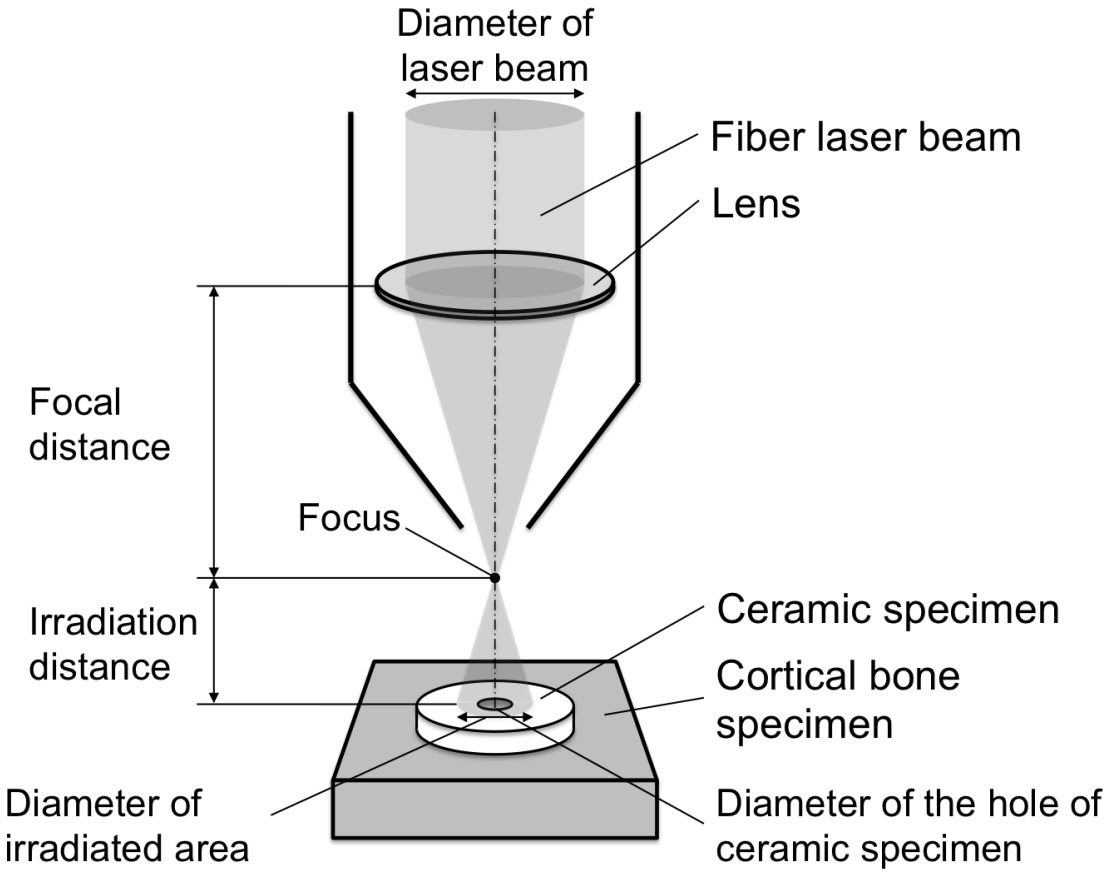


Figure 2

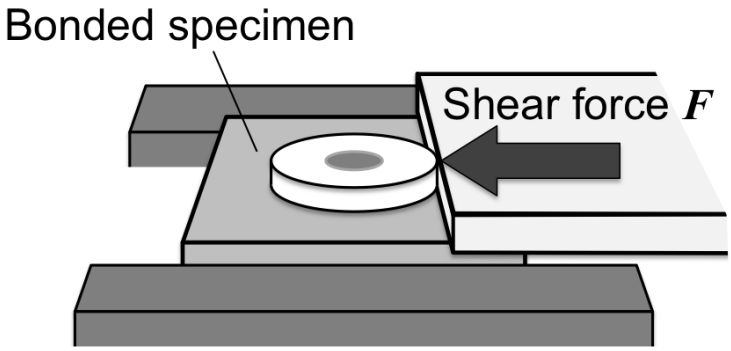


Figure 3

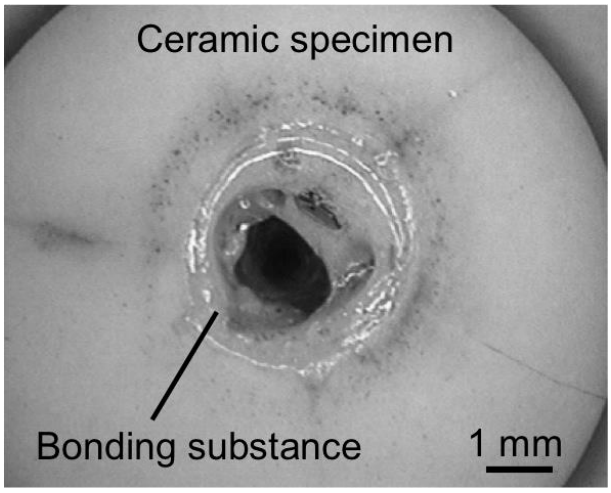


Figure 4

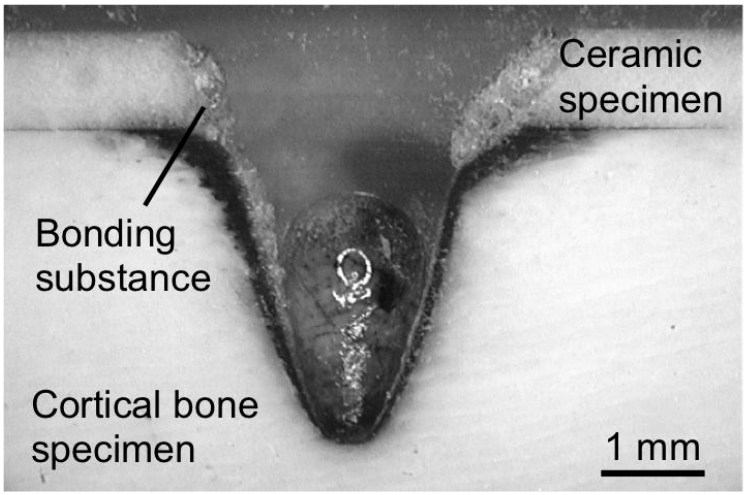

Ceramic
specimen
Bonding
substance
Cortical bone
specimen
1 mm

Figure 5

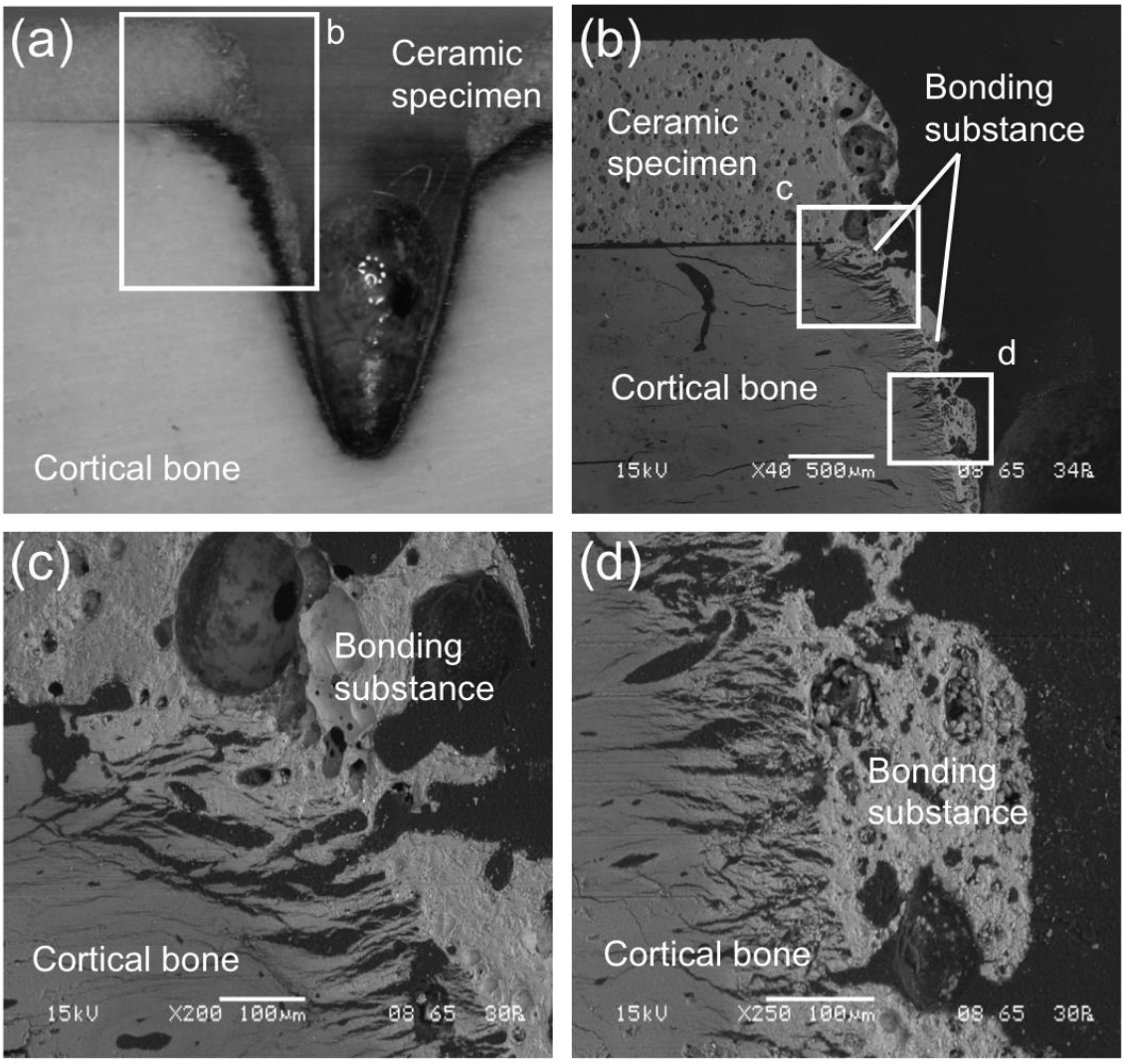

(a)
b
Ceramic specimen
Cortical bone
(b)
Ceramic specimen
Bonding substance
c
d
Cortical bone
15kV X40 500µm 08 65 34R
(c)
Bonding substance
Cortical bone
15kV X200 100µm 08 65 30R
(d)
Bonding substance
Cortical bone
15kV X250 100µm 08 65 30R

Figure 6

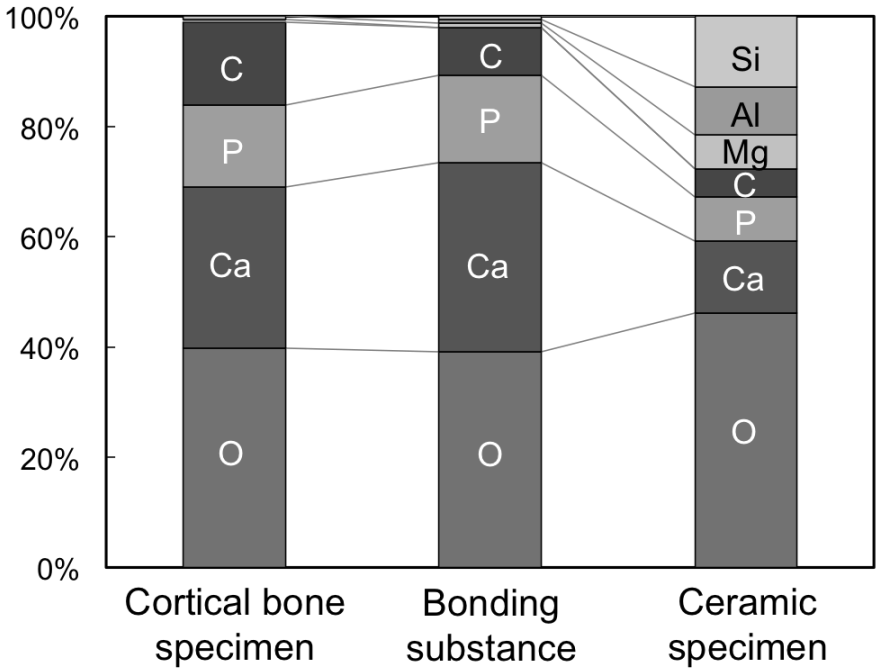

100%
80%
60%
40%
20%
0%
C
P
Ca
O
C
P
Ca
O
Si
Al
Mg
C
P
Ca
O
Cortical bone specimen
Bonding substance
Ceramic specimen

Figure 7

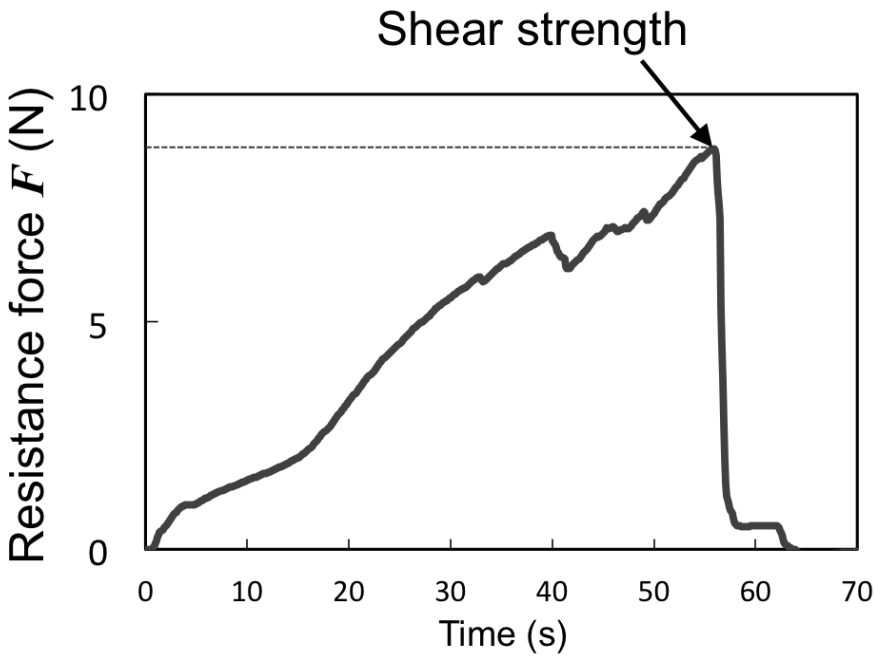

Shear strength
Resistance force F (N)
10
5
0
0
10
20
30
40
50
60
70
Time (s)

Figure 8

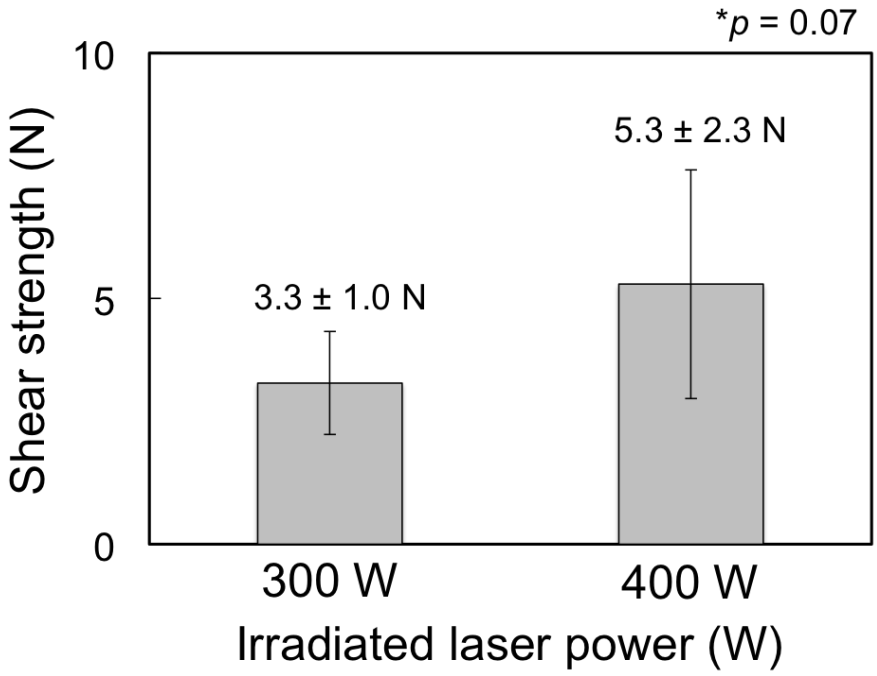
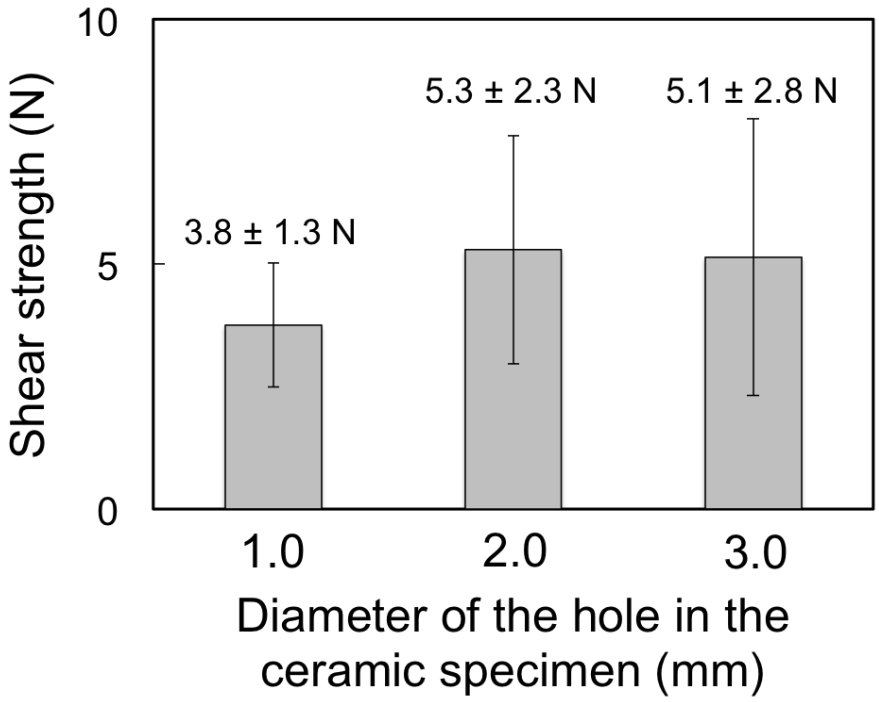

*p = 0.07
10
5
0
Shear strength (N)
3.3 ± 1.0 N
5.3 ± 2.3 N
300 W
400 W
Irradiated laser power (W)

Figure 9


10
5
0
Shear strength (N)
3.8 ± 1.3 N
5.3 ± 2.3 N
5.1 ± 2.8 N
1.0
2.0
3.0
Diameter of the hole in the ceramic specimen (mm)