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Bond and fracture strength of metal-ceramic restorations formed by selective laser sintering

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PURPOSE. The purpose of this study was to compare the fracture strength of the metal and the bond strength in metal-ceramic restorations produced by selective laser sintering (SLS) and by conventional casting (CAST). **MATERIALS AND METHODS.** Non-precious alloy (StarLoy C, DeguDent, Hanau, Germany) was used in CAST group and metal powder (SP2, EOS GmbH, Munich, Germany) in SLS group. Metal specimens in the form of sheets (25.0 × 3.0 × 0.5 mm) were produced in accordance with ISO 9693:1999 standards (n=30). To measure the bond strength, ceramic was fired on a metal specimen and then three-point bending test was performed. In addition, the metal fracture strength was measured by continuing the application of the load. The values were statistically analyzed by performing independent *t*-tests (α=.05). **RESULTS.** The mean bond strength of the SLS group (50.60 MPa) was higher than that of the CAST group (46.29 MPa), but there was no statistically significant difference. The metal fracture strength of the SLS group (1087.2 MPa) was lower than that of the CAST group (2399.1 MPa), and this difference was statistically significant. **CONCLUSION.** In conclusion the balling phenomenon and the gap formation of the SLS process may increase the metal-ceramic bond strength. ***[J Adv Prosthodont 2014;6:266-71]***

KEY WORDS: Selective laser sintering; Metal-ceramic restoration; Bond strength; Fracture strength; Balling phenomenon

INTRODUCTION

Recently, rapid prototyping (RP) using 3-D images has been developed and its use has spread widely in the field of dental technology. RP is a common name for a variety of technologies[1] for directly producing 3-D shaped products from computer aided design (CAD) files or digitally scanned data. Depending on the manner of production, RP technologies can be divided into stereolithography (SLA),[2] laminated object manufacturing (LOM),[3] selective laser sintering (SLS),[4,5] and other technologies. In particular, SLS involves melting and laminating a metal powder with a laser on the basis of modeled CAD data.[6] This technology has the advantage of shortening the production process and reducing the number of working hours, because the metal restoration can be produced in a digital environment without going through the complex process of wax carving, investment, and casting.[7,8] Another advantage of this method is that less material is wasted than in the cutting method using a milling device, because lamination is carried out only on the necessary part. SLS technology is used widely in other industries,[9] but it has only recently been introduced into the dental field for production of dental restorations.

Therefore, despite these advantages, there is a lack of data establishing the adequacy of these technologies for clinical applications, because very few studies assessing the quality of restorations produced by SLS have been carried out. The criteria for the clinical application of a new technology for producing restorations include biocompatibility of the materials,[10] aesthetics,[11] fracture resistance,[12] corrosion resistance,[13] and economic feasibility.[14] In the case of metal-ceramic restorations specifically, the strength of the

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Received 25 October, 2013 / Last Revision 13 June, 2014 / Accepted 16 June, 2014

This study was supported by a grant of the Korea University.

 pISSN 2005-7806, eISSN 2005-7814

bond with the ceramic[15,16] can also be an important criterion.

To meet these requirements, one-sided metal substructures can be produced using a centrifugal casting machine after the metal ingot is completely melted, as in conventional casting.[17] However, SLS locally sinters powder particles in specific areas based on the amount of energy per unit area supplied by the irradiating laser. If the powder is only partially sintered by the laser, spaces will be present between particles because of the balling phenomenon[18,19] and will affect both the strength of the metal itself and, especially, the strength of the bond between the metal and the ceramic. The balling phenomenon, which occurs typically in SLS, results in the combination of only a part of the center of a spherical particle with the nearby material.[20,21] This phenomenon is affected by both the laser power and the scan speed.[22-24] Therefore, the scan speed, particle size, specifications of the laser, and so on in the SLS process may affect the results. However, in some studies,[25,26] the exact mechanism and specifications of the equipment used are not reported. Moreover, while differences in the bond strengths between the metal and ceramic have been reported, the strength values of the metal substructure and their dependence on the SLS conditions have not been reported.

Even though the bond strength between the metal and the ceramic is a very important factor determining whether the metal-ceramic restoration can be successfully produced by SLS, there have been very few reports on this until recently. Therefore, in this study, we have attempted to explore the clinical applicability of the SLS method by comparing the bond strength between the metal and ceramic and the fracture strength of the metal itself in metal-ceramic restorations produced using the conventional casting (CAST) method and SLS. The null hypothesis in this study is that the bond strength between the metal and the ceramic does not vary with the production method.

MATERIALS AND METHODS

A non-precious alloy (StarLoy® C, DeguDent GmbH, Hanau, Germany) was used to produce the metal specimens used for CAST group, and Co-Cr metal powder (SP2; EOS GmbH, Munich, Germany) was used to produce the SLS group specimens. The composition of each material is presented in Table 1.

The coefficients of thermal expansion (CTEs) of the materials used in this study are shown in Table 2. Since a compressive stress occurs in the ceramic portion while the metal and ceramic cool after firing, the CTE of the metal must be slightly higher.[27] Therefore, in this study, a ceramic powder (Vita VM13, Vita Zahnfabrik, Bad Säckingen, Germany) that fulfills these conditions was chosen.

To measure the bond strength of the metal and the ceramic, 15 metal specimens were produced for each group. The specimens were produced as sheets (25.0 × 3.0 × 0.5 mm) in accordance with the ISO 9693:1999 standards. To fabricate the SLS group specimens, designs were produced in CAD and converted to STL files. The files were transmitted to the SLS equipment (EOSINT M280, EOS GmbH, Munich, Germany), after which the Co-Cr powder was sintered by the laser. The specifications of this SLS method are based on the standard method recommended by the manufacturer. The scan speed was set to 7 m/s, the lamination thickness was set at 100 µm, and the Yb-fiber laser power was 200 W. The production speed was 20 m^3/s, and the particle size of the metal powder was 20 µm.

However, to produce the metal specimens using the usual casting method (i.e., to produce the CAST group specimens), the mold of one of the SLS group specimens was replicated with silicone (Deguform® Plus, DeguDent Dentsply, Hanau, Germany). The wax pattern of this mold was produced by filling the mold with melted wax. The wax pattern was then invested with a phosphate-bonded investment for nonprecious metals, while the Co-Cr specimens were produced by centrifugation. The investment material and excess oxide layer attached to the surfaces of the specimens were sandblasted with 50 µm aluminum oxide (Al_2O_3) particles (Cobra, Renfert GmbH, Hilzingen, Germany) at a pressure of 3-4 bars. Sandblasting was also carried out for the SLS group specimens.

Table 2. CTEs of the materials used in the present study

Physical characteristics	CTE (m/m°C) × 10^{-6}
SP2	14.0-14.5
StarLoy® C	14.0-14.3
VITA VM13	13.1-13.6 (opaque) 13.6-14.0 (base dentin)

Table 1. Material composition in wt%

	Co	Cr	W	Nb	V	Mo	Si	Fe	Mn
SLS (SP2)	61.8-65.8	23.7-25.7	4.9-5.9	-	-	4.6 5.6	0.8 1.2	max 0.5	max 0.1
CAST (StarLoy® C)	54.9	24.5	10.0	2.0	2.0	1.0	1.0	0.1	-

SLS group: selective laser sintering. CAST group: conventional casting.

In order to observe the microstructure of the metal surface, one specimen was randomly selected from each group and observed under a scanning electron microscope (SEM). An accelerating voltage of 10.0 kV was employed. The observation area was limited to the portion that was to be fired to form the ceramic bond.

Ceramic samples of 8 mm in width, 3 mm in length, and 1 mm in height were fired on metal specimens of both the CAST and SLS group specimens according to the ISO 9693:1999 standards. In accordance with the instructions of the manufacturer, a thin opaque lining was applied before the ceramic was fired, and then the body ceramic was layered and fired.

To measure the metal-ceramic bond strength in each group, three-point bending tests were carried out with a universal testing machine (OTU-05D, Oriental TM Corp., Gyeonggi-do, Korea) in accordance with ISO Standard 9693.[28] After the specimen was placed on a 20 mm wide support, a load was applied at a crosshead speed of 1.5 mm/min using a sphere of 1 mm in diameter. During the test, the fired ceramic portion was oriented toward the bottom. To evaluate the bond strength, the load at which the metal and ceramic failed was measured. Then, to measure the fracture strength of the metal itself, the load was continually applied even after the failure of the ceramic. To compare and analyze the measured values, a statistical program (SPSS 12.0, SPSS Inc., Chicago, IL, USA) was used and the results of each group were tested using independent *t*-tests at the significance level α=.05.

RESULTS

The properties of the ceramic-contacting surfaces of the metal specimens observed before firing were as follows. Diagonal lamination was observed on the surfaces of metal specimens produced by SLS (Fig. 1A and Fig. 1B). The thickness of each layer was about 100 µm, and the spacing between the layers was about 10-20 µm (black arrow)(Fig. 1C). However, the laminated structure was absent in the metal specimens in the CAST group (Fig. 1D and Fig. 1E). Furthermore, when the image of the surface of the SLS group specimen was magnified, the balling phenomenon was observed, resulting in spaces between two particles or locations where two particles touch each other (Fig. 2A and Fig. 2B).

The means and standard deviations of the bond strengths exhibited by the two groups of specimens are listed in Table 3. The mean (SD) bond strength of the SLS group specimens was 50.60 (6.27) MPa, and the mean (SD) bond strength of the CAST group specimens was 46.29 (5.96) MPa. These values indicate the absence of any statistically significant difference between the two groups. The mean (SD) fracture strength of the SLS group specimens was 1087.2 (112.8) MPa, and upon fracturing, the metal specimens separated into two pieces. However, when a load of 2399.1 (48.7) MPa was applied, the CAST group specimens were merely bent without fracture. The independent *t*-test result showed statistically significant difference between the two groups (Table 4).

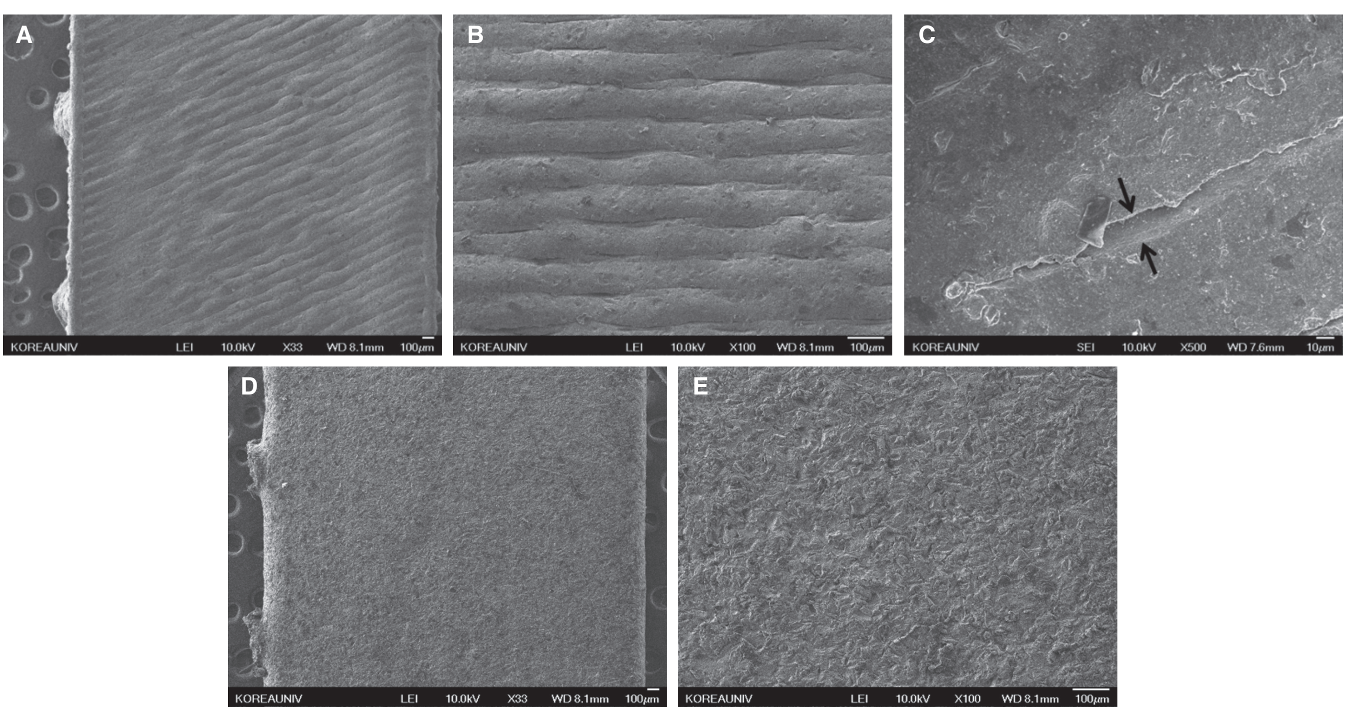

Fig. 1. SEM images of SLS specimens magnified at ×33 (A), ×100 (B), and ×500 (C) and of CAST specimens at ×33 (D) and ×100 (E). The black arrow was the spacing between the layers (about 10-20 µm).

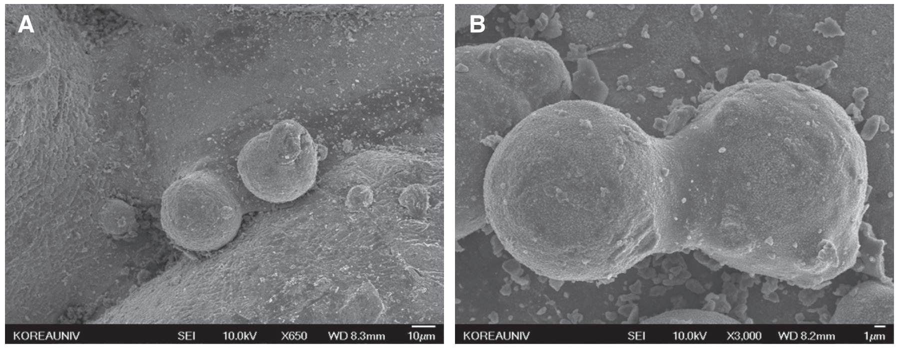

Fig. 2. SEM images of an SLS specimen magnified at ×650 (A) and ×3,000 (B).

Table 3. Independent t-test results for the bond strength values (MPa)(n=15)

Group	Alloys	Values of mean bond strength (SD)	*P* value
SLS	SP2	50.60 (6.27)	>.05
CAST	StarLoy® C	46.29 (5.96)	

Table 4. Independent t-test results for the fracture strength values (MPa)(n=15)

Group	Alloys	values of mean fracture strength (SD)	*P* value
SLS	SP2	1087.2 (112.8)	.000
CAST	StarLoy® C	2399.1 (48.7)	

DISCUSSION

For a valid comparison of the bond strengths between the metal and the ceramic obtained by using two different production methods, it is desirable that each ingredient used in the experiments should be identical,[29] including the oxide film formation behavior,[30] CTEs,[31] etc. To minimize the effect of different composition and to find pure effect of manufacturing methods, materials with the most similar composition were chosen among materials provided (Table 1). In order to reduce the errors caused by the differences in the oxide film, the sandblasting times were set to be identical. To reduce the differences in the CTE, metals and ceramics with similar CTEs were used (Table 2).

The bond strengths showed no statistically significant difference, but it is estimated that the SLS group showed higher bond strengths than the CAST group because the gap caused by the additive manufacturing method widened the surface area. As shown in Fig. 1, the SLS specimens exhibited 100-μm-thick layers aligned in the laser irradiation direction. The maximum gap between the layers was around 20 μm, and the bond strength was likely to be increased because of the penetration of the ceramic powder through the gaps. Furthermore, according to Gu and Shen,[32] the balling phenomenon occurs during layer-by-layer lamination during laser sintering. The balls formed at this time can be expected to increase the bond strength with the ceramic by increasing the surface area and by causing undercut. The studies performed by Korkmaz and Asar[33] indicated that the bond strength between a Co-Cr alloy and a ceramic was 41.46-85.16 MPa, while Nieva *et al.*[34] reported values of 57.11-63.81 MPa. These values are similar to or slightly higher than that reported in this study. Moreover, the results reported in this study are significantly higher than the minimum bond strength (25 MPa) between the metal and the ceramic specified in ISO 9693. Hence, it may be possible to use SLS to produce metal-ceramic restorations.

However, as seen in the results section, when a load was applied to the SLS group specimens even after the failure of the metal-ceramic bond, the metal portion fractured at 1087.2 MPa. It has been previously reported[35] that the maximum occlusal load that can be applied to the posterior part is 1,031 MPa, which determines the limit at which clinical applications are possible. Furthermore, in the study undertaken by Fischer *et al.*,[36] the experimental results for the Co-Cr alloy frame produced using the SLS method indicated a pattern similar to that observed in the present study, which further supports the clinical applicability of this method.

Based on the present study, SLS method showed similar bond strength to the conventional method. However, from a clinical point of view, this would have various advantages such as reduced working time, minimal material loss and minimal laboratory error.

In summary, according to the results of the present study, there was no significant difference between the metal-ceramic bond strengths obtained using SLS and casting. However, because of the layering produced by SLS, the fracture strength of the metal by itself appeared to be lower than that in the specimens produced by casting. Therefore, to increase the fracture strength of the metal itself while maintaining the metal-ceramic bond strength, additional studies are required to improve the materials and equipment used in SLS.

CONCLUSION

In conclusion, the balling phenomenon and the gap formation of the SLS process may increase the metal-ceramic bond strength.

ACKNOWLEDGEMENTS

The authors would like to thank E-Master Dental Hub Laboratory for processing the Co-Cr specimens with the EOS equipment.

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