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# **RESEARCH ARTICLE - MEDICAL TECHNIQUES**

# Bond Strength of Porcelain Fused to Casting and Selective Laser Base Metal Alloy with a Bonding Agent

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Article Info.	Abstract				
	Purpose: The goal of this study was to assess how different bonding agents affected the shear bond strength of a				
Article history:	and a 3D printer. Approach: A total of 40 disks of the cobalt-chromium alloy were made by using casting (Co–Cr) as the				
Received	control group (n=20 specimens) and additive manufacture by selective laser melting. SLM from Co-Cr (n=20				
10 May 2022	specimens) the disks were airborne particles abraded with110 µm aluminum oxide particles, then two main groups separated into subgroups based on the type of bonding agent used (with and without addition), shear bond strength was				
Accepted	used, before the SBS test the metal and ceramic interfaces were examined using test scanning electron microscopy				
06 July 2022	(SEM). The elemental makeup of the bonding agents was determined using an energy-dispersive X-ray spectroscopy (EDS) test. The results of the SBS tests as well as the failure modes were noted. Data were analyzed using the Least				
Publishing	Significant Difference (LSD) test ( $a = 0.05$ ).				
30 September 2022	Result: Bond strength was much stronger in Co-Cr specimens with bonding agents than in Co-Cr specimens without has disc exact. The much stronger of silver (Si) and exact the interface of silver (Si) and exact the				
	the production method and the presence of a bonding agent.				
	Conclusion: By use of a bonding agent on various Co-Cr metals improves bond strength with ceramics.				
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	Publisher - Middle Technical University				

Keywords: Co-Cr alloy; Dental ceramic; Bonding agent; Shear bond strength; Lost wax technique.

# 1. Introduction

The lost-wax process is a metal casting method in which molten metal is poured into a device designed from a wax model. Advances in dental technology have resulted in other methods of fabrication for alloys to the classic lost wax treatment over the last several decades[1], faults made by technical employees in the laboratory, as well as challenges with measurement and dental stone models, can prohibit this procedure from producing optimum results in the creation of metal infrastructures. As technology progresses, the use of computer-aided design/computer-aided manufacturing (CAD/CAM) technologies for the rapid construction of fixed restorations are becoming more common [2]. For reasons, such as excessive bur abrasion in milling technology, the system's failure to provide the desired time savings, the excessive amount of residual material, and the difficulty of producing more than one complex restoration at the same time, the application of "Rapid Prototype Manufacturing Technologies" in dental prosthetics treatment has recently been emphasized to physically generate three-dimensional models created in the CAD unit, it employs procedures such as selective laser sintering (SLS) device, direct metal laser sintering (DMLS) technique, and machine selective laser melting (SLM) [3].

Additive technique the other approach is laser sintering, which is based on laser melting that is selective laser melting (SLM). Because of their low cost and labor efficiency, both procedures are promising for future dental applications. SLM restoration, for example, the traditional lost wax repair is more expensive and time-consuming, while maintaining the same high quality of the final result [4]. The purpose of this study was to test the bond strength of dental ceramic to Co-Cr alloys made by casting and a 3Dprinter before adding further bonding ages to the specimens. We also intended to employ adhesion agents available on the market and SLM 3D printer machine Co-Cr dental alloy for casting, and they were able to improve the bonding strength between the ceramic and the alloy.

Nomenclature			
CAD/CAM	computer-aided design/computer-aided manufacturing	SBS	shear bond strength
SLM	selective laser melting	3D printer	three-dimensional printer
SEM	scanning electron microscopy	PFM	porcelain fused to metal
EDS	Energy dispersive X-ray spectroscopy		

## 2. Materials and Method

#### 2.1. Specimens' grouping

In this study, a total of 40 disc samples (10 mm diameter x 4 mm thickness with ring base (12 mm diameter x 1 mm thickness) [5]were fabricated of Co-Cr alloy for casting (Adentatec, Germany) and for SLM (Mediloy<sup>®</sup> S-Co BEGO (Germany) then divided into two groups (20 discs for each group) according to their fabrication technique and addition of vita bonding agent as follows:

Group A and B fabricated by conventional casting included in:

Group A (n = 10): There was no use of bonding material.

group B (n =10): the agent (Vita NP Bond paste, Bad Sackingen, Germany-based Vita Zahnfabrik) was an indication

Group C and D fabricated by a 3D Printer laser machine (SLM) included in:

group C (n =10): the agent was not applied.

group D (n =10): the agent (Vita NP Bond Paste (Bad Sackingen, Germany-based Vita Zahnfabrik) was an indication.

Then, porcelain build-up for all discs (10 mm diameter x 2 mm thickness) was done using feldspathic porcelain [5], Fig. 1.



Fig 1. Samples from different metal substrate techniques

#### 2.2. Fabrication of metal substrates

Group A and B: The lost wax casting technique was used to create the 20 discs, as well as the wax pattern preparation. All wax discs were fabricated using CAD/CAM technology to ensure uniform diameter and thickness. The wax discs were designed similar to the CAD/ CAM fabricated Co-Cr discs using a computer-aided design program (Exocad Dental CAD, Darmstadt, Germany, v.2016), A five-axis milling machine was used to mill wax patterns from a casting wax blank for CAD/CAM (Imes-Icore, 5 axis, COR TEC 350 iPRO, Germany). In a centrifugal casting apparatus, the Co–Cr specimens were created, using a phosphate-bonded investment; (Bego- Bellavest SH; Germany). The castings were removed and 110µm aluminum oxide powder was used to abrade the airborne particles for 15 seconds. (Dentify GmbH, Scheffelstr.22) below 3 bar pressure, and at 90 angle 2 cm distance from the nozzle of sand blast machine [6].

Group C and D: 20 discs were prepared with the commercial SLM method Laser (EP-M150METAL DENTAL 3D PRINTER) was used to manufacture metal cylinders, and from Co–Cr the design file was transferred to the SLM part (E Plus 3D PRINTER control software), outfitted with 100W Yb-fiber laser. The specimens' distance measurement axes were positioned perpendicular to the building platform. The Co–Cr powders used have particle sizes of 10-45 m, respectively. Use the SLM device with the proper laser. (For example; Nd: YAG Laser (wavelength approx. 1060–1100 nm) and enough laser power density (200 W) or power density at the surface (25 kW/mm<sup>2</sup>) according to the manufacturing instruction to melt the granules selectively using a protective gas (example; Nitrogen). BEGO has the parameter settings for the EOS M270 SLM device, including the production parameters, and can install them on the client's equipment. To minimize residual stresses caused by the laser's localized heat input and to modify the microstructure for improved mechanical performance.

#### 2.3. Application of a bonding agent

The surfaces were airborne-particle abraded 110  $\mu$ m before applying a bonding agent [7], then steam cleaned for 15 seconds, and all specimens were for time 8 minutes it was ultrasonic bath rinsed with water, using an ultrasonic cleaning machine, leaving building surfaces intact, the metal substrates for all the specimens group placed immediately in a preheated furnace (oxidation firing) at 500 °C the heat was increased to 980 °C; holding time 1 minute. The entire procedure took done in the open air. A bonding agent (Vita NP Bond Paste) is a product manufactured was added to the metal surface (thickness 0.1 mm, as recommended by the technique) [7], using a tweezer and disposable brush taken from the container and placed in the 3-part vertical show in Fig. 2, to the surface to determine the same thickness on all the specimens and firing according to the Table 1.



Fig 2. A and B (Vita NP Bond paste was applied to the surface of the alloy), C and D (Vita NP Bond was firing)

# 2.4. Porcelain veneering of the Co-Cr discs

All specimens were fired six times (with and without a bonding agent) according to the porcelain manufacturer's guidelines. The identical furnace (Vita ceramic Vacuum 6000 M oven) was used for all specimens. The firing times were selected according to the specifications provided by the manufacturer, Table 1 explains the times firing and materials used in this investigation.

Firing process	Preheating Temperature (°C)	Drying time (min)	Raise of Temperature (°C/min)	Vacuum	Final Temperature	Holding time (min)	Total time (min)
Oxidation	500	0	100	yes	980	5	9:48
Bonding agent	600	6	60	yes	960	1	13
Opaquer1	500	2	79	yes	950	1	8:38
Opaquer2	500	2	79	yes	950	1	8:38
Dentin 1	500	6	55	yes	935	1	15:54
Dentin 2	500	6	55	yes	920	1	15:38

Table 1 Specification of groups and materials used in this study

# 2.5. Scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDS)

For the SEM analysis, one metal sample (10 mm diameter x 4 mm thickness) was prepared for each group. Many photos were obtained with the help of SEM at various magnification strengths. The project has received superb visual representations of the metal surface structure and features from the SEM photos Fig.4, The interfacial surface of each sample was sputtered coated with gold and the bond surface of each sample was ready to be inspected under a scanning device electron microscope. The specimens were then fixed on an aluminum holder and images were taken at magnifications of 1000x [1]. Using energy dispersive X-ray spectroscopy (EDS), the elemental composition of the same specimens was qualitatively evaluated, and the weight % of each tracked element was calculated, Fig. 3 and Table 2.

# 2.6. Shear bond strength testing and mode of failure

A universal Instron testing machine was used to assess shear bond strength (SBS) for all groups (laryee WDW-50, China). The specimen mounting, as well as the one-of-a-kind testing device, which consists of the lower part stationary and upper part sliding stainless steel pieces, are shown in Fig .4, 0.5 mm/min cross-head speed, the upper part of the device slides down the grooves of the stationary part, loading the PFM interface until fracture done [5]. The values of the SBS were kept track of. To determine the type of failure, a digital microscope (Dinolite, Taiwan) was used to examine the manner of failure at a magnification power of 100x to identify the failure modes. They were grouped into the following categories: (1) adhesive type failures, which occur between the dental alloy and the metal oxide porcelain ; (2) cohesive type failures, which occur totally with the dental ceramic; and (3) mixed failures, are a mix of adhesion and cohesion.[3], mode of failure is shown in Fig. 6, Table 5.

### 2.7. Statistical analysis

Statistical methods were used to examine and evaluate the results including descriptive statistics (mean value, standard deviation, standard error, maximum and lowest values of the SBS test, bar chart for mean values of SBS) and Statistical analysis by Least Significant Difference (LSD) test



Fig 3. Chisel part of Instron universal testing machine loading the PFM interface until fracture

### 3. Results

#### 3.1. Scanning electron microscopy (SEM)

Firstly, in Fig. 4 of the metal-ceramic interface investigation as a result of SEM analysis, cross sections of the metal substrate dental ceramic specimens are presented before the SBS test. In specimens generated by a lost wax technique or 3d printer that did not apply a chemical agent, there was a considerable oxide film covering between the ceramic layer (1) in Fig. 4A and the metal part (2) in Fig. 4A; however, groups made by a 3D printer that received either ceramic bonding agent layer (2) in Fig. 4D had a connection in a quiet area between the metal and porcelain elements.



Fig 4. SEM images for metal-ceramic interface (A): casting without bonding an agent (1 ceramic layer, 2 metal surfaces), (B): casting with an agent (1 metal surface, 2 bonding agents, 3 ceramic layers), (C): SLM without an agent (1 metal, 2 ceramic), and (D) SLM with an agent (1 metal, 2 agents, 3 ceramic).

#### 3.2. Energy dispersive X-ray spectroscopy (EDS) Analysis

The effect of the bonding agent on shear bond strength will be related to two elemental compositions Si and Ti weight % [7], Table 2 shows the EDS results in weight % for the bonding agents used in this study. The bonding agent had much more titanium (Ti) and Si than specimens without a bonding agent, whereas the bonding agent had more Si and Ti in group B than the bonding agent in group D.

Elemental	А	A B		D
Groups -	Weight %	Weight %	Weight %	Weight %
C	8.2	8.7	9.0	19.9
0	33.9	37.8	32.8	23.5
Na	4.0	8.2	6.3	3.0
Mg	0.3	0.4	0.3	0.2

Al $5.7$ $3.2$ $1.2$ $2.9$ Si $9.8$ $12.6$ $9.5$ $10.3$ K $3.6$ $3.2$ $1.1$ $1.5$ Ca $0.7$ $0.9$ $1.0$ $0.4$ Ti $0$ $14.9$ $0$ $6.6$ Cr $7.9$ $6.2$ $11.1$ $9.9$ Fe $0.3$ $0.3$ $0.4$ $0.4$ Co $7.4$ $3.2$ $26.3$ $21.6$					
Si9.812.69.510.3K $3.6$ $3.2$ $1.1$ $1.5$ Ca $0.7$ $0.9$ $1.0$ $0.4$ Ti0 $14.9$ 0 $6.6$ Cr $7.9$ $6.2$ $11.1$ $9.9$ Fe $0.3$ $0.3$ $0.4$ $0.4$ Co $7.4$ $3.2$ $26.3$ $21.6$	Al	5.7	3.2	1.2	2.9
K $3.6$ $3.2$ $1.1$ $1.5$ Ca $0.7$ $0.9$ $1.0$ $0.4$ Ti $0$ $14.9$ $0$ $6.6$ Cr $7.9$ $6.2$ $11.1$ $9.9$ Fe $0.3$ $0.3$ $0.4$ $0.4$ Co $7.4$ $3.2$ $26.3$ $21.6$	Si	9.8	12.6	9.5	10.3
Ca $0.7$ $0.9$ $1.0$ $0.4$ Ti $0$ $14.9$ $0$ $6.6$ Cr $7.9$ $6.2$ $11.1$ $9.9$ Fe $0.3$ $0.3$ $0.4$ $0.4$ Co $7.4$ $3.2$ $26.3$ $21.6$	Κ	3.6	3.2	1.1	1.5
Ti 0 14.9 0 6.6   Cr 7.9 6.2 11.1 9.9   Fe 0.3 0.3 0.4 0.4   Co 7.4 3.2 26.3 21.6	Ca	0.7	0.9	1.0	0.4
Cr7.96.211.19.9Fe0.30.30.40.4Co7.43.226.321.6	Ti		14.9		6.6
Fe0.30.30.40.4Co7.43.226.321.6	Cr	7.9	6.2	11.1	9.9
Co 7.4 3.2 26.3 21.6	Fe	0.3	0.3	0.4	0.4
	Co	7.4	3.2	26.3	21.6

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#### 3.3. Measurement for shear bond strength

The testing of SBS of PFM was compared in this study. Samples are made with different Co-Cr alloy manufacturing processes, including 3D printer and traditional casting techniques. In Fig .5 graph with bars showing the mean distribution and standard deviations of (SBS) of the tested groups. For each metal substrate ceramic combination, the average SBS values were obtained, comparing the four samples of dental porcelain bond strength (n=40), in Table 3, the calculated percentage bond strength for each porcelain applied with on specimens, showed the mean distribution and standard deviations of (SBS) of the four tested groups and minimum, maximum value. Group B recorded the highest average (SBS) mean values (10.607 MPa) followed by group A (8.288 MPa) while the lowest average (SBS) mean values were recorded intermediate in group D (9.386 MPa). According to Table 4, when compared between different groups. Show in statistically a major difference was shown between groups A and B with a p-value (0.05) and no statistically significant difference between traditional A and laser group C without an additional bonding agent, and a statistically significant difference was shown between groups fabricated by 3D Printer laser machine C and D from the other hand there was a statistically significant difference found between groups fabricated by 3D Printer laser machine C and D.

Table 3 Descriptive statistic of the shear bond strength for two different process techniques of studied groups

Groups	Ν	Mean	Std. Deviation	Min.	Max.
Cast (Without Agent)	10	8.288	.47403	9.100	7.700
Cast (With Agent)	10	10.607	1.15702	12.269	8.973
Laser (Without Agent)	10	8.133	.94338	9.800	7.127
Laser (With Agent)	10	9.386	1.09157	10.818	7.700



Fig 5. Bar chart showing the mean distribution of shear bond strength (SBS) of the studied groups

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(D) Capung		Mean	Std.	P- Value	Sig.	95% Conf	95% Confidence Interval	
(I) Groups	(J) Groups	(I-J)	Error			Lower Bound	Upper Bound	
	Cast (With Agent)	-2.3190*	.44072	.000	S	-3.2026	-1.4354	
Cast (Without Agent)	Laser (Without Agent)	.1550**	.44072	.726	NS	7286	1.0386	
	Laser (With Agent)	$-1.0980^{*}$	.44072	.016	S	-1.9816	2144	
Cast (With Agent)	Laser (Without Agent)	$2.4740^{*}$	.44072	.000	S	1.5904	3.3576	
	Laser (With Agent)	$1.2210^{*}$	.44072	.008	S	.3374	2.1046	
Laser (With Agent) Laser (With Agent)		-1.2530*	.44072	.006	S	-2.1366	3694	

Table 4 Statistical analysis by Least Significant Difference (LSD) test

\* The mean difference is significant at the 0.05 level.

\*\*The mean value is greater than >0.05 level.

# 3.4. The mode of failure

Failure mode was classified as cohesive (fracture inside ceramic layers), adhesive (fracture interface between ceramic and metal), or mixed (cohesive and adhesive failure) by analyzing the ceramic remnants. In the present study, the mode of failure analysis showed only cohesive and mixed failures, while adhesive failure was not found in all groups. The fracture mode distribution of de-bonded samples after SBS is shown in Fig. 6. In the casting group without an addition agent, the fracture mode was 40 % for cohesive fracture and 60 % for mixed fracture. In the bonding agent casting group, the facture mode was 20 % for cohesive fracture and 80 % for mixed fracture. In the laser group without an addition agent, the fracture and 50 % cohesive fracture. While in the bonding agent laser group 40 % cohesive fracture and 60 % mixed fracture, failure is shown in Table 5.



Fig 6 Shows the Co-Cr surfaces of specimens after de-bonding from the ceramic after the shear bond strength test

Groups	Cohesive failure		Mixed failure		Adhesive failure	
	No	%	No	%	No	%
А	4	40	6	60	0	0
В	2	20	8	80	0	0
С	5	50	5	50	0	0
D	4	40	6	60	0	0

#### 4. Discussion

The strength of the metal-ceramic interfacial contact is influenced by chemical bonding, micromechanical interlocking, coefficients of thermal expansion (CTEs), and the metal substrate of the production methodology. The primary focus of this study was chemical affinity at the point of contact, and different manufacturing techniques for the fabrication of dental alloy great care was taken to remove the influence of other variables as much as possible [8].

A comparison was made in this study, the SBS of PFM samples made with various cobalt-chromium dental alloy manufacturing processes, including lost wax and additive technique. The techniques of manufacturing were no statistically significantly affecting the shear bond strength of both casting and SLM groups, it was graphically represented in Fig (5). this study agreement with (Serra et al., 2014) [6], there was no difference in bond strength according to the fabrication method. When the SLM process was applied, many studies (Akova et al., 2008; Wu et al., 2014) [9-11], looked at the metal-ceramic bond strength. Some of them(Akova et al., 2008, Jie et al., 2014) [9, 10], used the shear test to test the metal-ceramic bond strength, while others (Wu et al., 2014, Bae et al., 2014) [11, 12] used the three bending test. Regardless of the test performed, there were no significant variations in the strength of metal-ceramic bonds, as found in any of the studies.

Bonding agent materials are used to facilitate communication between these various materials. The process of dispersion bonding should be possible, eliminating the acute interface, and along the contact, a focus between it two pieces of work should eventually be established, which can be obtained if the oxide portion is less or the metal surface is oxidized [7].

This study result that all specimens with bonding agents had much higher bond strength than specimens without bonding agents. This is in agreement with many studies and dis agreement with others. Specimens with a bonding agent had substantially better bonding strength than specimens without an additional bonding agent [13]. There is a lot of debate in the literature concerning how bonding agents boost bonding strength when it comes to chemical bonding at the interface. According to the research studies with quantitative data, an adhesive agent improves the binding strength of dental Ti alloys,[14-16]. Effect of lesser significance (Depending on the special system, this can be whether it's good or bad), as casting cobalt-chromium alloy is an indication [17]. Curtis et al., applied a bonding agent to dental Ti alloy using multiple ways (spray, paste, and powder) and observed when examined specimens without a bonding agent, no change in metal-ceramic bond strength was observed [14]. Al-Hussaini et al [18], the three-point bonding strength of dental feldspathic porcelain and pure titanium cast alloy was investigated utilizing a bonding agent greatly elevated the PFM three-point binding strength, according to (Wehnert et al.,2009) [16], work on titanium porcelain bond strength.

A scanning electron microscope was used to analyze the ceramic and metal bonding surfaces before undertaking SBS testing. Fig .4 shows all groups created by the lost wax technique and 3D printer laser melting without a bonding agent, a dense oxidation layer that appeared to be a space between the substrate and feldspathic porcelain, in which there are electron bonds predominated. Bonding chemicals are intended to close (or at least lessen) this gap, according to the makers. Finally, results were obtained in a corresponding increase in binding strength, according to several research (Table 2). Even though changes in principal elements of commercially bonding agents can affect bond strength disparities. Group A alloy-porcelain contact is a clear black line in Fig. 4, indicating that interfacial adhesion is caused by roughness characteristics along the alloy-opaque layer interface. The Level of opacity is clearly visible utilizing a deeper hue and is well linked to the dentin layer (upper black line in Fig. 4). Fig. 4 is given with a lighter tint on the upside group A in Fig .4, Ti-rich layer was discovered near the alloy in Group B. This region, shown in Fig. 4, has Si, which was most likely transmitted from an opaque layer, which is likewise distinguished following deeper color, and then the dentin layer [7].

Energy dispersive X-ray spectroscopy (EDS) Analysis examination revealed that porcelain components such as aluminum (Al), silicon (Si), and titanium (Ti) were present on Co-Cr alloy surfaces. The presence of Al could be attributed to  $Al_2O_3$  air-particle abrasion residues and/or porcelain (opaque layer) attached to the alloy substrate. The Al compositions of the groups were comparable, we determined that the majority of the Al came from leftover  $Al_2O_3$  particles following air-particle abrasion. Excessive oxides that develop on the alloy surface during porcelain firing are absorbed by metal bonding agents containing Si [19, 20]. As shown in Table 2, Si was included in the bonding agents employed in our investigation to improve bond strength by controlling the thickness of the oxide layer. The presence of Si on EDS following the stress test suggested the presence of residual ceramic. The amount of Si on the surfaces of casting-fabricated alloys with bonding agent group B was the highest (Table 2), indicating that specimens in this group had the strongest bond strength in our study. In this investigation, we employed Ti-containing bonding agents (Table2) because they operate as oxygen scavengers, preventing the alloy surface from accumulating an excessive oxidation layer over time as a result of repeated fire cycles [18]. Ti is possible because of the diffusion of porcelain components into titanium oxides following the fire [21, 22] This effect could account for some of the improved metal-ceramic bond strength found on the Co-Cr metal substrate when using a bonding agent, nevertheless, a bonding agent containing Ti can moderate strong oxidation of the metal surface while also increasing the bond strength. To put it another way, the bonding agent can act as a chemical link between the metal-ceramic interface.

Mode of failure investigation, the failure mode analysis revealed that the majority of the disc samples had mixed failure, which included both adhesive and cohesive failure in the porcelain, with a porcelain fragment in contact with the metal. While some samples showed a lack of cohesiveness these outcomes were in line with expectations, those of Oliveira de Vasconcellos et al., [5, 23], looked into the influence of the cycle of heat and mass on the binding strength of porcelain fused to both Co-Cr and gold alloys, finding that both alloys had a high rate of mixed failure. Also, the mixed failure found in this investigation was the same as that found in Suliman and Styern's study [24]. Additionally, according to Papazoglou and Brantley, the mixed type of failure allows the existence of excellent metal and ceramic bonding strength. In addition agreement with Maja Antanasovaa, [25]. After the SBStest, we looked at the fracture mechanism between ceramic and metal. The most common failure mode, according to previous studies (Xiang et al., 2012) is mixed-type failure [26]. In another investigation, specimens created with the casting approach had a mixed mode of failure, whereas samples fabricated with the 3D printer SLM technique were either a mixed or adhesive mode of failure (Akova et al., 2008) [9]. In their investigation, the result of this study disagreement with Han et al. documented all adhesive type failures, despite of production process [27].

## 5. Conclusion

1- Application of bonding agent to different Co-Cr metal increases bond strength with ceramics.

2-This study shows that the SLM technology may successfully cover Co-Cr dental alloy with dental feldspathic porcelain.

3- No statistically significant difference between the traditional casting group and laser group without additional bonding agent.

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