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### ARTICLE

# Effect of Pretreatment Process on the Metal-Porcelain Bonding Mechanism and Properties of CoCr Alloy Dental Crown and Bridge Manufactured by SLM

Wang Di, Wu Shibiao, Fu Fan, Ye Guangzhao, Mai Shuzhen,

Qian Zeyu, Song

### Changhui, Yang Yongqiang

South China University of Technology, Guangzhou 510640, China

**Abstract:** This paper aims to investigate the influential mechanism of grinding and sand blasting pretreatment of CoCr alloy manufactured by SLM (selective laser melting) process on the metal-porcelain bonding of the restorations, and then provides references for SLM manufacturing CoCr alloy dental crown and bridge restorations with good mechanical properties. Three groups of CoCr alloy substrates were manufactured by SLM process with optimal parameters. The samples were subjected to different grinding and sand blasting pretreatment, and the porcelain sintering was carried out with the same parameters. The samples of different groups were analyzed by three-point bending test, roughness test, SEM and EDS. Results show that the metal-porcelain bonding strength of samples in group a with grinding and sand blasting is  $36.79\pm0.49$  MPa, which is obviously higher than those of group b with no grinding but sand blasting and group c with only grinding. The average metal-porcelain bonding effect of group a and c is better than that of group b, and the element diffusion on metal-porcelain interface is observed in all three groups. The results also show that there are significant differences among the three groups on residues and element distribution of metal-porcelain peeling surface. The final conclusion is that the surface roughness obtained by the pretreatment of sand blasting and grinding can ensure the strong chemical combination of metal and porcelain and considerable mechanical properties combination, which can improve the performance of metal-porcelain restorations manufactured by SLM.

**Key words:** selective laser melting (SLM); pretreatment; dental restorations; metal-porcelain bonding mechanism; failure mode; CoCr alloy

Traditional dental restorations are usually made by casting and CNC processing which have the disadvantages of long manufacturing time and low precision<sup>[1]</sup>. In recent years, SLM metal additive manufacturing technology has developed rapidly, owning the merits of a wide range of materials, high forming precision, good mechanical performance and many other advantages, and also has been successfully applied to the manufacturing of complex medical devices and parts, such as personalized bird-beak repair, pelvis fracture surgical repair guide, personalized knee implants and skull repair implants<sup>[2,3]</sup>.

The metal-porcelain bonding strength is one of the important indicators to test whether the porcelain-fused-to-metal (PFM) dental restoration is suitable. The PFM restoration with poor metal-porcelain bonding strength is easy to crack in the occlusal surface and even leads to the peeling of porcelain layer caused by the interface bonding failure<sup>[4,5]</sup>. The metal-porcelain bonding strength is essential for the performance of PFM dental restorations. Some scholars have

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Corresponding author: Yang Yongqiang, Ph. D., Professor, School of Mechanical and Automotive Engineering, South China University of Technology, Guangzhou 510640, P. R. China, Tel: 0086-20-87111036, E-mail: meyqyang@scut.edu.cn

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studied the metal-porcelain bonding properties of dental restorations made by traditional methods. The research on the metal-porcelain bonding properties of CoCr alloy dental restorations made by SLM process is rare, and the related metal-porcelain bonding mechanism is even less<sup>[6,7]</sup>. Al Jabbari, Xiang et al. had compared the bonding strength, failure mode and microhardness of the alloy ceramic restorations made by casting, machining and SLM. And the results showed that the metal and porcelain bonding performance of the CoCr alloy prepared by SLM was better<sup>[8,9]</sup>. Zhang et al. investigated the effect of PFM temperature on the metal-porcelain bonding properties of SLM molded CoCr alloy restorations, the results showed that the metal-porcelain bonding strength is higher than that of ISO 9693: 1999<sup>[10]</sup>; Ren et al. analyzed the influence of sintering times on the metal-porcelain bonding properties of SLM and cast-molded CoCr alloy<sup>[11]</sup>; Huang et al. tested the co-ordination degree of SLM, cast-molded CoCr alloy and cast Au-Pt alloy PFM crowns with human body, and it was found that the degree of compatibility of SLM molded CoCr alloy PFM crowns was similar to that of the latter two<sup>[12]</sup>.

This paper studies the influence of polishing and sand blasting, two kinds of common pretreatment processes, on the metal-porcelain bonding strength of CoCr alloy formed by SLM. The influence mechanism of these two pretreatment processes on the metal-porcelain bonding properties of CoCr alloy dental crown and bridge was further studied, which could provide references for SLM technology to manufacture CoCr alloy dental restorations better.

#### **1** Experiment

The SLM manufacturing equipment applied in the manufacturing of CoCr alloy metal substrate for PFM sintering process was developed by South China University of Technology and named Dimetal-100. The PFM sintering process of the metal samples was carried out by Ivoclar Vivadent's Programat P310 porcelain furnace.

The powder used for the SLM manufacturing of metal substrate samples is the CoCr-1 alloy powder of MTI China, and its main components are shown in Table 1. The powder was prepared by a gas atomization method and its SEM image is shown in Fig.1. The porcelain powder used in the metal substrate sample PFM process is VITA series porcelain powder. The CoCr alloy substrate sample is manufactured by optimized SLM process parameters, including laser power 160 W, scanning speed 400 mm/s, scanning distance 0.08 mm, processing layer thickness 0.03 mm, spot diameter 0.08 mm, oxygen content of less than 0.1% during the manufacturing process.

Table 1 Chemical composition of CoCr-1 alloy powder (wt%)

Со	Cr	Mo	W	Si	С	0
Bal.	25.7	5.9	5.6	<1.5	< 0.03	< 0.05

Fig.1 SEM image of CoCr-1 alloy powder

Nine metal substrates were manufactured and divided into three groups: group a, b and c. The metal samples were designed according to ISO9693: 1999 standard, of which the size is shown in Fig.2. Among them, a group samples' porcelain surface was ground, followed by sand blasting for 30 s; b group samples' porcelain surface was not ground to retain the surface characteristics of SLM, directly sand blasting for 30 s; c group samples' porcelain surface was ground.

The surface roughness  $R_a$  values of the above three groups of metal substrates were measured using a Talysurf CLI 1000 surface profiler. After three sets of substrate samples were ultrasonically cleaned, the surface PFM sintering was carried on according to the PFM sintering procedure shown in Table 2 to complete the production of metallic PFM samples.

The three-group bending test was going to be carried out using the Shimadzu EZGRAPH series electronic component material testing machine to obtain the time-stress curve of each sample to determine the breaking force  $F_{\text{fail}}$ . The average thickness of each group of metal substrates and porcelain layers was measured by a vernier caliper and according to the determination coefficient k given by the ISO9693: 1999



Fig.2 Size of metal-porcelain samples

Table 2 PFM sintering procedure of CoCr alloy

	Temperature/	Heating	Preheat	Sintering	Vacuum
	°C	rate/°C·min <sup>-1</sup>	time/min	time/min	degree/%
1st opaque porcelain	940	55	3	5.5	98
2nd opaque porcelain	920	55	3	5.5	98
Body and transparent porcelain	920	60	3	1.5	98

standard, as shown in Fig.3 to determine metal thickness and Young's modulus of the metal material as a function of the coefficient k; according to the formula:

$$\tau_{\rm b} = k F_{\rm fail} \tag{1}$$

to calculate the metal-porcelain bonding strength  $\tau_b$  of each sample, where  $F_{\text{fail}}$  is the breaking force and the coefficient *k* is a function of metal thickness and Young's modulus of the metallic material. The microscopic morphology of the metal-porcelain interface was observed and examined by EDS line scanning spectroscopy using the scanning tunneling microscope Nova NanoSEM 430. At the same time, the microstructure of the metal-porcelain surface peeling was observed and examined by Nova NanoSEM 430 surface scanning energy spectrum analysis.

#### 2 Results and Discussion

#### 2.1 Three-point bending strength test

Fig.4 shows the three-point bending test samples of metallic PFM substrate manufactured by SLM with different surface treatments. Macroscopic observation shows that the porcelain layers of 3 groups of metal substrate specimens are uniform and the PFM effect is good.

Table 3 shows the thickness of metal substrate and porcelain layer measured by the vernier caliper. Then, the thickness of each metal substrate sample and Young's modulus (200 GPa) of the CoCr alloy are used to determine the coefficient k according to the curves shown in Fig.3. Table 3



Fig.3 Diagram of the determination coefficient  $k^{[19]}$ 



Fig.4 Three-point bending test specimens

# Table 3Thickness measurement and coefficient kof each sample

Sample	Metal substrate thickness/mm	Metal substrate & porcelain thickness/mm	Coefficient, <i>k</i> /mm <sup>-2</sup>	Surface roughness, <i>R</i> <sub>a</sub> /µm
a-1	$0.5200 {\pm} 0.007$	1.673±0.015	3.50	3.58
a-2	$0.5300 \pm 0.010$	$1.660 \pm 0.017$	3.56	2.91
b-1	$0.5320 \pm 0.013$	$1.613 \pm 0.005$	3.32	9.53
b-2	$0.5240 {\pm} 0.008$	$1.540 \pm 0.010$	3.46	10.07
c-1	$0.5360 {\pm} 0.005$	$1.600 \pm 0.010$	3.24	2.47
c-2	$0.5340{\pm}0.005$	$1.563 \pm 0.015$	3.28	2.70

also lists the surface roughness  $R_a$  of each group of metal substrate samples, and we found that different pretreatment process leads to different surface roughness values, and surface roughness of the grinding pretreated metal substrate is significantly lower than that of the only sand blasting pretreated sample. Among them, the surface roughness of the metal substrate pretreated with grinding and sand blasting is similar to that of the metal substrate only grinded, and the former is slightly higher. The effect of different pretreatment processes on metal-porcelain bonding strength is partly reflected by the effect of surface roughness on metal-porcelain bonding strength.

Fig.5 shows the three-point bending stress curves of each group of CoCr alloy. The highest point of the curves is the breaking force when the porcelain layer is peeled off at one end of the sample. The metal-porcelain bonding strength of each group was calculated according to formula (1), as shown in Table 4.



Fig.5 Time-stress curves of CoCr alloy with different surface roughness: (a) group a, (b) group b, and (c) group c

Table 4 Vietai-borcelain bonding strength results of samples (vi)	Table 4	Metal-porcelain b	onding strength	results of sam	ples (MP
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Sample	1	2	Mean value
Group a	36.44	37.13	36.79±0.49
Group b	32.53	32.74	32.64±0.15
Group c	35.99	34.85	35.42±0.81

The results show that the average metal-porcelain bonding strength of group a of CoCr alloy samples subjected to conventional grinding and sand blasting pretreatment is  $36.79\pm0.49$  MPa, and the average metal-porcelain bonding strength of group b of specimens subjected to sand blasting pretreatment is  $32.64\pm0.15$  MPa, while the average metal-porcelain bonding strength of the samples subjected to conventional grinding pretreatment is  $35.42\pm0.81$  MPa. The average metal-porcelain bonding strength of all three groups is higher than that of the ISO9693: 1999 standard value of 25 MPa, which indicates that the metal-porcelain bonding strength of the SLM molded alloy CoCr restorations meets the standard requirements<sup>[13]</sup>.

#### 2.2 Analysis of metal-porcelain bonding interface

Fig.6a, 6c and 6e show the SEM images of the metal-porcelain bonding interface of the 3 sets of specimens, and there is a significant difference in the bonding interface among the three sets of specimens. In Fig.6a, the surface of CoCr alloy substrate of the sample is ground and sand blasted. It can be found that the SLM characteristic surface of the substrate is relatively smooth, but a certain amount of small bulge structure exists. A small amount of small voids are scattered along the substrate surface in the metal-porcelain bonding region, and a small amount of bubbles exist in the opaque porcelain layer. In Fig.6c, the SLM characteristic

surface of the CoCr alloy substrate is only sand blasted. It can be found that the surface of the CoCr alloy substrate is very flat and smooth. The typical periodic fluctuation of the SLM manufacturing parts has not been observed<sup>[14,15]</sup>. The metal-porcelain bonding zone is evenly distributed along the substrate surface with smaller voids. A large number of small bubbles are scattered inside the opaque porcelain layer. The surface of the CoCr alloy substrate of Fig.6e is ground only. It can be found that the surface of the substrate is uneven and there are a large number of protrusions and pits.

According to the principle of SLM manufacturing, there will be bumps and periodic surface undulation due to the welding seam overlap. At the same time, the unavoidable splashing behavior in the SLM manufacturing process can also lead to the influence on the surface and internal properties of the parts, which are embodied in the local splash granules on the surface, and a decrease in surface quality, internal voids and inclusions<sup>[16]</sup>. During the sintering process, the viscous opaque porcelain will be affected by the fluctuated surface of substrate, the splashing of particles and the adhesion of powder, so that the local area in the opaque porcelain will form irregular gaps covering the substrate. The surface of the CoCr alloy substrate (group b), which is pretreated only by sand blasting, will form some new tiny pits due to the immobilization of the powder on the surface and the removal of splashed particles. However, the influence on the periodic fluctuation on the surface of the substrate is small. The occurrence of new depressions increases the roughness of the substrate surface and its contact area with the viscous opaque porcelain, and increases the blocking effect of the substrate surface on the cladding process of porcelain layer. Therefore,



Fig.6 SEM surface morphologies (a, c, e) and EDS spectra (b, d, f) of metal-porcelain bonding interface: (a, b) group a, (c, d) group b, and (e, f) group c

the gap on the metal-porcelain interface and the number of bubbles increases. The periodic fluctuation of the substrate surface is seriously weakened after the grinding pretreatment (group c). Smooth surface is very conducive to the viscous opaque porcelain's uniform cladding on the surface of the substrate, and greatly reduces the irregular gap defects along the metal-porcelain interface due to a block of the cladding process which leads to the minimum number of voids and bubbles in the corresponding metal-porcelain interface; The CoCr alloy substrate (group a) pretreated with grinding and sand blasting has a relatively smooth and flat surface. Because of the additional effect of sand blasting, the surface of the substrate uniformly spreads a certain number of small protrusions structure which increases the surface roughness and contact area with the viscous opaque porcelain, resulting in metal-porcelain interface intersperses with a small amount of little gaps.

Meanwhile, the EDS results of Fig.6b, 6d and 6f show that the metal elements Co, Cr and porcelain elements Si, O are diffused in the vicinity of the bonding interface to form a df fusion layer with a width of about 2  $\mu$ m, mainly rich in O, Cr compounds. The difference of oxygen concentration between the metal substrate and the oxide porcelain layer leads to the migration of oxygen ions in the sintering process to produce redox reaction and form a strongly adherent diffusion layer to generate a chemical bond between the CoCr metal and porcelain.

#### 2.3 Analysis of metal-porcelain peeling surface

The common failure mode of metal-porcelain in three-point bending test is divided into the following three categories: (1) adhesive mode: porcelain cracks occur in the interface between the metal and porcelain layer; (2) cohesive mode: porcelain cracks occur entirely in the porcelain layer; (3) mixed mode: including the above two kind of porcelain cracks situation<sup>[17,18]</sup>. Fig.7 shows the macroscopic morphologies of the metal-porcelain peeling surface of each group of porcelain samples. It can be seen that the surface of sample a with grinding and sand blasting and the surface of sample with no pretreatment both have porcelain layer residue on the metal-porcelain peeling surface. The porcelain layer residue of sample a is distributed uniformly, and both ends have partially



Fig.7 Macroscopic morphologies of porcelain peeling surface

broken porcelain residues. But sample b's porcelain layer residue is distributed unevenly, and the porcelain layer residue is even more in some regions; the metal-porcelain peeling surface of sample c with grinding pretreatment leaves nearly no porcelain residue. The results show that grinding and sand blasting pretreatment of the metal substrate can improve the uniformity of the metal-porcelain interface.

In order to further determine the failure mode of each sample, the distribution of elements on the metal-porcelain peeling surface of three groups was investigated by EDS area analysis.

As shown in Fig.8a and 8b, it can be seen that the microstructures of the metal-porcelain peeling surface of sample a and b are similar, and there are a large number of porcelain crystals remaining on the metal-porcelain peeling surface of the two samples. EDS results also show that a large number of Al, Si porcelain elements remain, and there are also a certain amount of Cr and Co metal elements, indicating that the fracture within porcelain layer and the fracture between metal and porcelain occur simultaneously on the peeling surface, and both fracture modes belong to the mixed type. The results show that the residual elements on the surface of sample a pretreated by sand blasting and grinding are evenly distributed, and the residual elements on the surface of sample



Fig.8 SEM surface morphologies and EDS maps of porcelain peeling surface: (a) group a, (b) group b, and (c) group c

b only by sand blasting pretreatment are segregated. This is due to the fact that the CoCr alloy substrate with only sand blasting pretreatment basically retains the original characteristic surface of the SLM process. The original characteristic surface has periodic bumps and pit structure, which leads to an obvious difference of the metal-porcelain bonding conditions in the local area of the metal substrate. From Fig.8c, it can be seen that there is almost no porcelain crystal residue leaving on the metal-porcelain peeling surface of group c, and there exist a lot of metal elements such as Cr and Co and very few porcelain elements such as Al and Si, which indicates that the fracture occurring between the metal and porcelain layer's interface bonding area belongs to the adhesion failure mode.

#### 2.4 Discussions

The metal-porcelain bonding mechanism mainly includes four parts: mechanical combination, compression combination, van der Waals force combination and chemical combination<sup>[19]</sup>, in which the mechanical combination caused by metal substrate and the opaque porcelain' interlocking chimeric during the sintering process are considered to be the most important metal-porcelain bonding. The results of EDS line scanning analysis shows that there is a chemical bond at the bonding interface between the metal substrate and the porcelain layer. The metal-porcelain bonding strength of PFM restorations depends mainly on the bonding condition between the metal substrate and the opaque porcelain<sup>[20]</sup>. During the sintering process, the viscous opaque porcelain melt into the metal substrate and was affected by the wetting ability of the opaque porcelain to the metal substrate and the surface quality of the metal substrate<sup>[21]</sup>. And the influence mechanism of pretreatment process on metal-porcelain bonding properties of CoCr alloy can be described as Fig.9.

The pretreatment of grinding removes the periodic fluctua-



Fig.9 Influence mechanism of pretreatment process on metalporcelain bonding properties of CoCr alloy

tion of the surface of the CoCr alloy and the adhesion of powder, reduces the surface roughness of the substrate and facilitates the smooth deposition of the liquid opaque porcelain on the substrate surface. The smooth cladding process reduces the probability of voids and bubbles generation, while increasing the uniformity of the metalporcelain bonding, which is conducive to a sufficient chemical combination. The new pits caused by the powder removal also increases the barrier to the cladding of the opaque porcelain to a certain extent. Therefore, the simple blasting pretreatment increases the degree of instability of the opaque porcelain cladding which directly leads to the production of voids along the surface of the substrate and a large number of bubble defects in the porcelain layer, hindering the inter-diffusion of metal and porcelain elements which is not conducive to chemical combination.

In comparison with the results of the three-point bending strength of each group samples in Table 4, it was found that the average metal-porcelain bonding strength of the samples ground in group c is much higher than that of the samples sandblasted in group b. The above results show that the chemical combination enhancement caused by sanding treatment is much stronger than that caused by sandblasting; the surface quality of the substrate pretreated by grinding and sand blasting is close to that of only grinding pretreatment. On the basis of low surface roughness obtained through grinding and additional blasting pretreatment by sanding exposed internal holes, inclusions and deep splashing will produce some extra small pits<sup>[22,23]</sup>. The small pits formed by the blasting pretreatment along the substrate surface uniformly enhance the degree of chimeric between the substrate and porcelain, and enhance the mechanical combination effect.

#### 3 Conclusions

1) SLM manufacturing CoCr alloy restorations have a good metal-porcelain bonding strength and fully meets the requirements of ISO medical standards. The restorations with surface grinding and sand blasting have the best metal-porcelain bonding properties.

2) The substrate surface with certain degree of roughness ensures sufficient metal-porcelain mechanical combination, while forms a metal-porcelain bonding area with no significant bubble defects, which facilitates the chemical reaction during the PFM process to build strong chemical combination. Grinding combined with blasting pretreatment can maintain a certain degree of mechanical combination and chemical combination between metal and porcelain, so the CoCr alloy restorations have the highest strength.

3) The metal-porcelain failure mode of PFM restorations treated by surface grinding and sand blasting and only by sandblasting are of mixed type, which has cracks occurring in the interface area of the metal and porcelain layer and inside the porcelain layer. The metal-porcelain failure mode of PFM restorations treated only by surface grinding is the adhesive type, which has cracks occurring in the interface area between the metal and porcelain layer.

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## 预处理工艺对 SLM 成型 CoCr 合金牙冠牙桥金瓷结合机制及性能的影响

王 迪,吴世彪,付 凡,叶光照,麦淑珍,钱泽宇,宋长辉,杨永强 (华南理工大学,广东广州 510640)

摘 要:为了探究打磨喷砂预处理激光选区熔化(SLM)成型 CoCr 合金基底对修复体的金瓷结合的影响机制,进而为 SLM 成型具有 良好力学性能的 CoCr 合金牙冠牙桥修复体提供参考。采用 SLM 优化工艺参数成型 3 组 CoCr 合金基底试样,分别进行不同的打磨、喷 砂预处理以相互对照,之后以相同参数进行烤瓷烧结。采用三点弯曲试验、粗糙度测试、SEM 及 EDS 等测试方法分析不同对照组试样。测试结果表明,经过打磨和喷砂预处理的 a 组试样的金瓷结合强度达到 36.79±0.49 MPa,明显高于不打磨而保留 SLM 成型特征表面的 b 组试样,以及打磨处理而不喷砂的 c 组试样,3 组试样的平均金瓷结合强度均高于 ISO9693:1999 标准规定的最小值 25 MPa。a、c 组的金瓷结合效果优于 b 组,3 组试样的金瓷结合界面均发生元素扩散,分析还发现 3 组金瓷剥落面的残留物及其剥落面元素分布有显 著差异;最终结论为:打磨、喷砂组合预处理获得的一定粗糙度的基底表面可保证金瓷间强大的化学结合以及可观的机械结合,可以显 著提高 SLM 成型金瓷修复体的性能。

关键词: 激光选区熔化; 预处理; 牙科修复体; 金瓷结合机制; 失效模式; CoCr 合金

作者简介: 王 迪, 男, 1986年生, 博士, 副教授, 华南理工大学机械与汽车工程学院, 广东 广州 510640, 电话: 020-87114484, E-mail: mewdlaser@scut.edu.cn

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