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RESEARCH AND EDUCATION

Effect of two heat treatments on mechanical properties of selective-laser-melted Co-Cr metal-ceramic alloys for application in thin removable partial dentures

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ABSTRACT

Statement of problem. Heat treatment has been used to reduce the residual stress of alloys fabricated by selective laser melting (SLM) to avoid deformation. Co-Cr metal-ceramic alloys are used to fabricate metal-ceramic restorations and removable partial dentures (RPDs) on the same substrate by SLM. A heat treatment that enables the fabrication of metal-ceramic restorations and RPDs with excellent mechanical properties should be evaluated.

Purpose. The purpose of this in vitro study was to determine the effects of 2 heat treatments on the mechanical properties of SLM Co-Cr metal-ceramic alloys intended for the fabrication of thin RPDs.

Material and methods. Tensile bars were manufactured using cast metal-ceramics (C-MC group), RPD alloys (C-RPD group), and SLM Co-Cr metal-ceramic alloys. The SLM specimens were subjected to 2 different heat treatments, L1 at 880°C and L2 at 1100°C, and were further divided into subgroups (L1-MC, L1-RPD, L2-MC, and L2-RPD). Thirty-six tensile specimens were prepared in C-RPD, L1-RPD, and L2-RPD (simulated partial denture alloys for clinical use) and in C-MC, L1-MC, and L2-MC (simulated metal-ceramic alloys); 18 metal-ceramic bond strength specimens were prepared in C-MC, L1-MC, and L2-MC groups (n=6). The tensile test and 3-point bend test were conducted using a universal testing machine. The fracture surfaces of the L2-RPD tensile bar were examined using a scanning electron microscope. The Student *t* test (α=.05) was used for statistical analysis.

Results. No significant differences were observed between the bond strengths of L1-MC and C-MC (P=.74) or between those of L2-MC and C-MC (P=.124). The 0.2% yield strength ($\sigma_{0.2}$) and elongation of all SLM specimens exceeded the minimum requirements required for the fabrication of thin RPDs as prescribed in ISO 22674:2016. The $\sigma_{0.2}$ value of L1-MC and L2-MC was significantly higher than that of C-MC. Significant differences in $\sigma_{0.2}$ values were found among the 3 RPD groups, L1-RPD>L2-RPD>C-RPD. For the elongation, significant differences were found among the 3 groups, L2-RPD>C-RPD>L1-RPD. The fracture surface of L2-RPD showed clear submicroscale dimples with fusion defects.

Conclusions. When Co-Cr metal copings and RPD frameworks were fabricated on the same substrate simultaneously using SLM, heat treatment at 1100°C was found more suitable than at 880°C to release residual stress, considering the toughness required for dental prostheses. (J Prosthet Dent 2018;119:1028.e1-e6)

Co-Cr alloys have been used instead of Ni-Cr alloys for metal-ceramic restorations and removable partial dentures (RPDs) because of the prevalence of nickel allergy.[1-3] These prostheses are conventionally fabricated using lost-wax casting.[1-3] In the casting process, the elemental compositions of Co-Cr metal-ceramic alloys and partial denture alloys are different. Information about Co-Cr alloys for metal-ceramic restorations and RPDs as provided by the manufacturers is listed in Table 1. Co-Cr alloys for metal-ceramic restorations contain more tungsten and trace elements such as Nb and V, which can improve high-temperature strength and decrease the linear coefficient of thermal expansion.[4-6] Co-Cr RPD alloys have higher Cr, Fe, Mn, and C content, which enhances their strength, hardness, and castability but

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Clinical Implications

A Co-Cr alloy can be used to fabricate dental metal copings and RPD frameworks simultaneously on the same substrate using SLM. The prostheses should be heat treated at 1100°C rather than at 880°C to relieve stress.

decreases their toughness.[7] The information in Table 1 shows that for the fabrication of thin RPDs, the 0.2% yield strength ($\sigma_{0.2}$) of the alloys must be higher than 500 MPa and the elongation (E) must be higher than 2%[8]; thus, the $\sigma_{0.2}$ value of cast Co-Cr metal-ceramic alloys is insufficient for fabricating thin RPDs.

Recently, selective laser melting (SLM), a powder-based additive manufacturing technique that emerged in the late 1980s and 1990s, has been adopted for the production of dental restorations.[9,10] SLM uses a laser beam to selectively melt successive layers of powder according to computer-aided design (CAD) data. The layers are rapidly cooled to directly obtain a net-like product. Advantages of the technology include economical use of materials and manpower, bulk production, and flexible design.[10-12] Furthermore, SLM has been used to fabricate metal copings and RPD frameworks.[12,13] The composition of the alloy powders strongly affects the bond strength of dental porcelain, whereas the layer thickness in SLM does not influence the bond strength.[14] In addition, the metal-ceramic bond strength, marginal accuracy, and corrosion resistance of restorations made with SLM are comparable with those of cast restorations.[13,15-17] However, Co-Cr alloys with different compositions cannot be used on the same substrate simultaneously without modifying the fabrication technique. Figure 1 shows metal copings and RPD frameworks fabricated simultaneously on the same substrate using a Co-Cr alloy. To comply with the requirements of ISO 9693, the metal-ceramic bond strength (τ_b) must be greater than 25 MPa[18]; thus, Co-Cr metal-ceramic alloys are selected for manufacturing metal copings and RPD frameworks by SLM. However, the mechanical properties of the metal-ceramic alloys obtained by SLM should be investigated because of their importance for RPD frameworkss.[7,18]

In addition, components manufactured by SLM undergo deformation because of residual stress.[19,20] During SLM, the last fused top layer shrinks upon cooling with a magnitude dependent on the underlying (already solidified) material, and between layers, large residual stresses accumulate, resulting in the distortion of the product.[19,20] Although the support structure, as seen in Figure 1, can limit this distortion, heat treatment has also been used to reduce and avoid the deformation as far as possible,[19-21] especially for fixed partial dentures with a long span and RPDs. Heat treatment can eliminate the crystal defects of metals, such as dislocations, to stabilize the microstructure with the reduction of residual stress.[21] The mechanical properties of alloys are also affected by the heat treatment.[21]

ISO 22674:2016[8] requires that preoxidation and 4 veneer firings be performed before determining the tensile properties of metal-ceramic alloys. The mechanical properties of Co-Cr alloys fabricated by SLM have been examined preliminarily[22]; however, a more rigorous investigation from the viewpoint of their dental application is indicated.

Therefore, the purpose of this in vitro study was to evaluate the effects of 2 heat treatments on the mechanical properties of selective-laser-melted Co-Cr metal-ceramic alloys intended for the fabrication of thin RPDs, with a cast Co-Cr metal-ceramic alloy and RPD alloy used as controls. The null hypotheses were that the mechanical properties of Co-Cr alloys would not depend on either heat treatment or manufacturing method.

MATERIAL AND METHODS

Solibond C plus–cast Co-Cr alloys for metal-ceramic restorations (Table 1) were used for the C-MC specimens, and alloy powders (particle size, 10 to 63 μm) with the same compositions were used for the SLM specimens. Stellite C3–cast Co-Cr RPD alloy (Table 1) was used for the C-RPD specimens. Metal strips (25×3×0.5 mm) for the metal-ceramic bond strength test[18] and dumbbell-type tensile bars for the mechanical property test[8] were fabricated using an SLM machine (M100; EOS) and a vacuum casting machine (Argoncaster; Shofu, Inc).

Custom-made polymethylmethacrylate (PMMA) tensile bars and strips were used as patterns for the cast specimens (Fig. 2). Before the additive manufacturing step, the specimens were designed by a 3-dimensional automated technique from CAD design (Cambridge; 3Shape A/S). SLM layers were printed by adding new layers in the long axis of the test bars and strips; the laser power was 170 W, layer thickness was 30 μm, scan speed was 7 m/s, laser spot diameter was 40 μm, and the scanning strategy of the laser involved a rotation of 67 degrees between layers.

The stress-relief processes for the SLM Co-Cr alloys involved multiple heating steps. For heating process 1, the specimens were placed in a furnace and heated to 500°C in 60 minutes and held at 500°C for 45 minutes. The furnace was then heated to 880°C in 60 minutes, and the specimens were held at 880°C for 60 minutes. After that, the furnace was cooled, and the furnace door was opened when the temperature was 600°C. Finally,

Table 1. Dental cast Co-Cr alloy for metal-ceramic restorations and RPD (manufacturer's information)

Cast Co-Cr Alloy	$\sigma_{0.2}$/MPa	E/%	Type and Application	Composition (wt%)
ET-02; ET alloy	≥420	12	IV MC	Co63.0, Cr24, W8.3, Mo3.0
Keragen; Eisenbacher	≥360	10	IV MC	Co61.6, Cr27.8, W8.5, Mn0.3, Fe 0.2, Si1.6, C<0.1
Solibond C plus; YETI	≥495	10	IV MC	Co63.0, Cr24.0, W8.1, Mo2.9, Si1.1, Nb0.9
Argeloy NP Supreme; Argen	475	8	IV MC	Co61, Cr27, W5.0, Mo6.0, Si1.0, Mn<1.0, Fe<1.0, C<1.0
Wirobond C; Bego	480	6	IV MC	Co61, Cr26, W5.0, Mo6.0, Si1.0, Fe0.5, Ce0.5, C<0.02
Vitallium 2000; Dentsply Sirona	600	6	V RPD	Co63.1, Cr28.5, Mo6.0, Mn, Si, C, N<1.0
ROBUR 400; Eisenbacher	705	2.6	V RPD	Co62.5, Cr28.5, Mo6.1, Fe0.7, W0.6, Mn0.55, C 0.5
Wironit extra-hard; Bego	625	4.1	V RPD	Co63.0, Cr30.0, Mo5.0, Si1.1, C<1.0
Argeloy N.P. Partial; Argen	552	6.5	V RPD	Co64.0, Cr28.0, Mo6.0, Mn<1.0, Fe<1.0, W<1.0, C<1.0
Remanium GM 380; Dentaurum	640	6	V RPD	Co64.6, Cr29.0, Mo4.5, Si, Mn, N, C<1.0
Vera PDS Hard; Aalbadent	675	6	V RPD	Co63.5, Cr27.0, Mo5.5, Fe2.0, Ni<0.99
Stellite C3; Stellite	710	≥3	V RPD	Co61.0-65.0, Cr28.0-32.0, Mo4.0-6.0

$\sigma_{0.2}$, 0.2% yield strength; E, elongation; MC, metal-ceramic; RPD, removable partial denture.

Figure 1. SLM metal copings, removable partial denture framework, and support structure. SLM, selective laser melting.

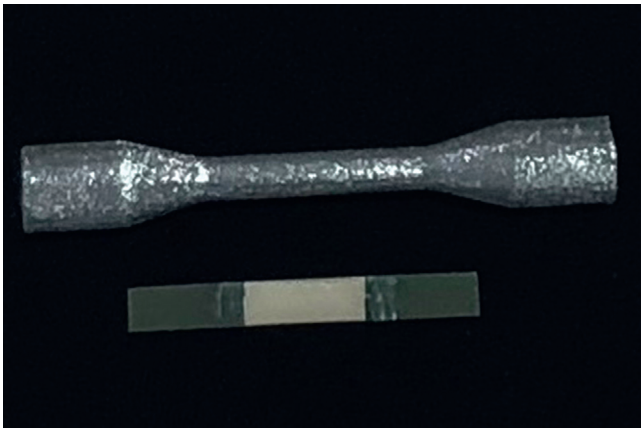

Figure 2. Representative experimental cast specimens.

specimens were removed from the furnace when the temperature was 300°C. For heating process 2, the specimens were placed in a furnace and heated to 1100°C at 8°C/min and held at 1100°C for 30 minutes. The furnace door was opened after the furnace had cooled to 600°C, and the specimens were removed from the furnace when the temperature was 300°C.

All metal strips were polished using 400-grit Al_2O_3 abrasive paper, washed in deionized water for 5 minutes, air dried, and preoxidized (for 5 minutes at 950°C). Then, they were airborne-particle abraded using 110-µm Al_2O_3 before the porcelain application. A porcelain veneer (VMK 95; VITA Zahnfabrik), with a thickness of 1.1 mm and consisting of a layer of opaque porcelain and body porcelain, was applied in the central area (3×8 mm) of each metal strip and fired in a furnace (Multimat NTXpress; Dentsply Sirona); the temperature profiles were based on commercial porcelain-firing programs and are shown in Table 2. The grouping, numbers, and thermal histories of the specimens are summarized in Table 3.

Table 2. Porcelain-firing programs used (manufacturer's recommendations)

Firing Programs	Sintering Temperature (°C)	Heating Rate (°C/min)	Hold Time (min)
Wash Opaque 1	950	75	1
Opaque 2	930	72	1
Dentin	930	55	1
Glaze	930	82	1

The 3-point bend test to examine the bond strength of the specimens was conducted according to ISO 9693.[18] The tensile test was performed in air at 25°C using a universal testing machine (3367; Instron) at a displacement rate of 2 mm/min, and the fracture surfaces of the L2-RPD specimens were examined using a scanning electron microscope (SEM; EVO 18; Carl Zeiss Jena).

Data were analyzed with statistical software (IBM SPSS Statistics, v24.0; IBM Corp), and the Student *t* test (α=.05) was used to verify the significance of values.

Table 3. Specimen numbers and thermal histories in each group

Group	Specimens (n) Tensile Property	τ_b	Stress Relief	Preoxidation	Porcelain Programs
C-MC	6	6	/	√	√
L1-MC	6	6	Process 1	√	√
L2-MC	6	6	Process 2	√	√
C-RPD	6	0	/	/	/
L1-RPD	6	0	Process 1	/	/
L2-RPD	6	0	Process 2	/	/

MC, metal-ceramic; RPD, removable partial denture. τ_b, metal-ceramic bond strength; Preoxidation, 5 min at 950°C; Porcelain programs, as shown in Table 2; √, undergone corresponding treatment; /, without corresponding treatment.

Table 4. Mechanical properties

Groups	$\sigma_{0.2}$/MPa	E/%
C-MC	517 ±19.0	7.9 ±3.53
L1-MC	1058 ±18.8	4.7 ±1.17
L2-MC	827 ±18.6	5.7 ±1.13
C-RPD	726 ±65.1	6.5 ±1.45
L1-RPD	1217 ±21.0	3.4 ±1.00
L2-RPD	878 ±54.5	8.6 ±0.92
L1-MC and C-MC	P<.001	P=.068
L2-MC and C-MC	P<.001	P=.186
L1-MC and L2-MC	P<.001	P=.176
L1-RPD and C-RPD	P<.001	P=.016
L2-RPD and C-RPD	P<.001	P=.002
L1-RPD and L2-RPD	P<.001	P<.001

E, elongation; MC, metal-ceramic; RPD, removable partial denture. σ0.2, 0.2% yield strength.

RESULTS

All τ_b values for the C-MC, L1-MC, and L2-MC groups exceeded the requirement τ_b>25 MPa, as defined by ISO 9693 for compatibility testing of metal-ceramic systems in dentistry.[18] No significant difference was found in the bond strength between L1-MC (37.7 ±2.7 MPa) and C-MC (37.3 ±1.58 MPa) (P=.74) or between L2-MC (40.63 ±4.59 MPa) and C-MC (P=.124). The mechanical properties of the specimens are shown in Table 4. The $\sigma_{0.2}$ value of L1-MC and L2-MC was significantly higher than that of C-MC, and the $\sigma_{0.2}$ value of L1-MC was higher (P<.05) than that of L2-MC. The $\sigma_{0.2}$ values for the RPD groups indicated a progression in which L1-RPD>L2-RPD>C-RPD, whereas the elongation progression was L2-RPD>C-RPD>L1-RPD. Compared with C-RPD, statistical analysis demonstrated that L1-RPD and L2-RPD had higher (P<.05) $\sigma_{0.2}$ values, whereas L1-RPD had a significantly lower elongation and L2-RPD had a significantly higher elongation. Furthermore, the elongation of L2-RPD was higher (P<.05) than that of L1-RPD, with lower yield strength (P<.05). The fracture surfaces of L2-RPD tensile bars are shown in Figure 3. The fracture mainly originated from the zones in which the powders were not fused or were incompletely melted (Fig. 3A,B). Figure 3C showed clear submicroscale dimples with fusion defects.

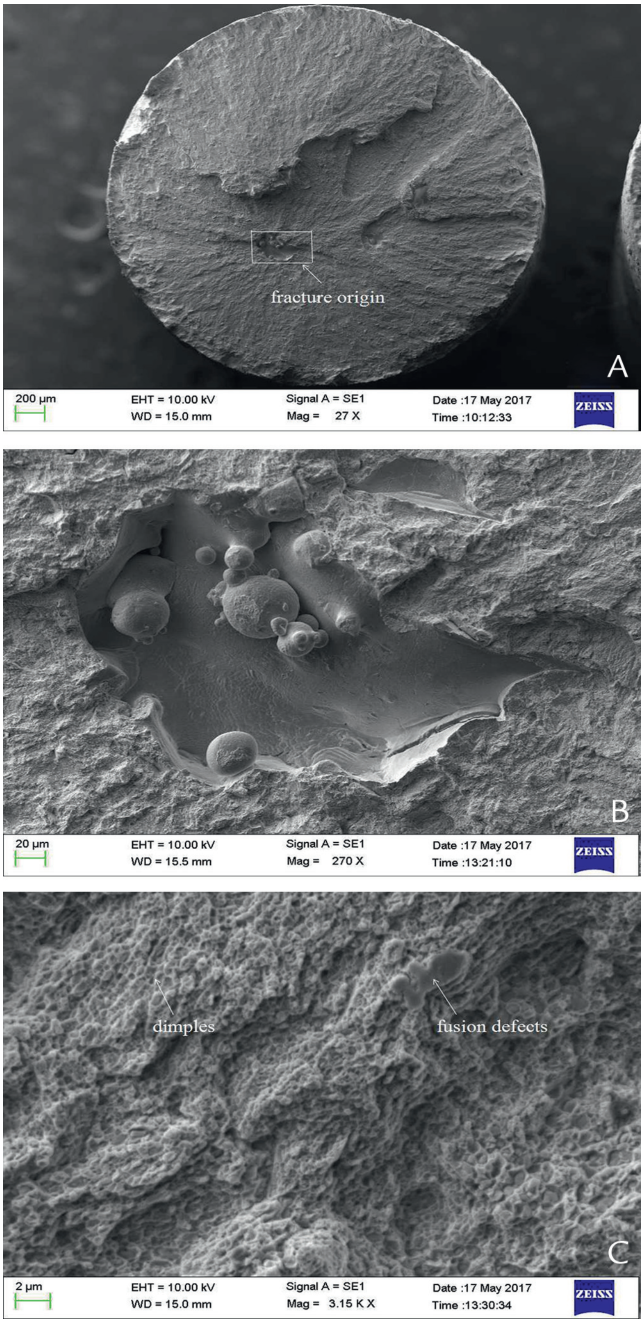

Figure 3. Scanning electron micrographs of L2-RPD fracture surfaces after tensile test. A, Original magnification ×27. B, Original magnification ×270. C, Original magnification ×3150.

DISCUSSION

The effects of 2 heat treatments on the mechanical properties of dental Co-Cr alloys fabricated by SLM were investigated in this study, and the results led to the rejection of the null hypotheses.

The strength and toughness of Co-Cr alloys depend on the microstructure that consists of a face-centered cubic (fcc) crystal lattice that is stable at high temperatures and on a hexagonal close-packed (hcp) structure that is stable at low temperatures.[23-25] The fcc structure generally exhibits ductility as it facilitates dislocation motion. In contrast, the hcp structure leads to more brittleness because it limits dislocation motion.[23] In the isothermal transformation of a Co-29Cr-6Mo alloy, without the addition of nitrogen or carbon, the fcc to hcp transformation occurs at 900°C, and the transformation does not occur at 1000°C,[23] implying that the transformation could occur below 900°C and would not occur above 1000°C. The phase diagram for the Co-Cr alloys with different compositions shows that the temperature for an fcc to hcp transformation is nearly 900°C and that the small difference in temperature is probably due to a slightly different elemental composition.[6,26,27] Based on these results, 2 heat treatment processes were chosen with maximum temperatures of 880°C and 1100°C for relieving residual stress in this study.

C-MC, L1-MC, and L2-MC were fabricated according to the procedure specified for metal-ceramic alloys intended for clinical use. First, the metal-ceramic bond strength and mechanical properties of SLM and cast Co-Cr alloys were estimated to verify their suitability for metal-ceramic restorations. The results showed that these alloys complied with the minimum requirements for τ_b>25 MPa, which was consistent with previous results.[17,18] In addition, the $\sigma_{0.2}$ value for L1-MC and L2-MC was significantly higher (P<.05) than that for C-MC, which was more conducive to reducing the deformation caused by occlusal forces on the prostheses and decreasing the stress at the interface between the porcelain layer and the metal.[7] Considering that L1-MC had a higher yield strength than L2-MC, the heat treatment at 880°C may be more appropriate for reducing residual stresses in metal-ceramic restorations.

The specimens in the C-RPD, L1-RPD, and L2-RPD groups were mechanically tested. The $\sigma_{0.2}$ values of these specimens exceeded 500 MPa, and elongation exceeded 2%, satisfying the minimum requirements of ISO 22674 for metallic materials for fixed and removable restorations and appliances in dentistry.[8] Nevertheless, fracture toughness is especially important for RPDs.[7] Clasps with low elongation can fracture more easily than those with high elongation and high strength.[7] Although L1-RPD exhibited a higher strength than C-RPD, its significantly lower elongation suggested its poor toughness for RPD applications. In contrast with C-RPD, L2-RPD not only had a higher strength but also had a higher fracture elongation, which indicates that it would be better suited for thin RPD applications. The fcc to hcp transformation at 880°C may explain the differences in the elongation and strength between L2-RPD and L1-RPD.

The fracture surface of L2-RPD showed submicroscale cellular dimples, possibly originating from the fracture of submicroscale cellular dendrites, which is consistent with previous observations.[22] Cast Co-Cr alloys have low strength and ductility because of their coarse microstructure and solidification defects.[26-28] In the Co-28Cr-9W-1Si alloy with 0.03 wt% carbon, the grain size of the cast specimen was 81.6 ±8.3 μm, and the grain size was 45.6 ±3.6 μm for 0.06 wt% carbon.[6] Compared with the casting process, the "discrete-stacked" forming method typical of SLM constrained the grain growth.[22] Considering the diameter of the laser spot (40 μm), the layer thickness of the powder (30 μm), and the rapid cooling of SLM, it was very difficult to achieve grain sizes greater than 40 μm for the SLM specimens, which have potentially resulted in higher strength. However, the precise grain characteristics of SLM specimens with different thermal histories should be examined further. Numerous small dimples, shown in Figure 3C, also indicated the ductile fracture modes of the L2-RPD specimen. Another major defect in SLM-fabricated specimens was the incomplete or nonexistent fusion of the metal powders, as seen in Figure 3B.

If Co-Cr metal-ceramic alloys, after relieving residual stresses, were also suitable for fabricating RPD frameworks by SLM, the metal copings and RPD frameworks could be printed simultaneously from the same substrate using the same process parameters. This would optimize production throughput and reduce manpower, resources, time, and cost. Under these experimental conditions, the heating process at 1100°C was more suitable for fabricating RPDs because of the improved toughness compared with specimens heated at 880°C. Therefore, when fabricating metal copings and RPD frameworks on the same substrate by SLM, heat treatment at 1100°C is better suited for releasing residual stresses.

In this study, the effects of different heat treatments on the microstructure and grain characteristics of Co-Cr alloys fabricated by SLM were not investigated directly, which is an objective for future studies. The residual stress and distortion of SLM dental restorations, especially RPD frameworks, should be measured in the future.

CONCLUSIONS

Within the limitations of this in vitro study, the following conclusions were drawn:

1. SLM-fabricated Co-Cr alloys heat treated at 880°C and 1100°C to reduce residual stress exhibited mechanical properties that exceeded the minimum requirements according to ISO 22674:2016.[8]
2. These alloys could be used for metal-ceramic restorations and thin RPDs when fabricated using SLM.

3. However, when Co-Cr metal copings and RPD frameworks were fabricated on the same substrate simultaneously, the heat treatment at 1100°C was more suitable for relieving the residual stress, considering the toughness of dental prostheses.

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