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Effect of Heat Treatment Duration and Cooling Conditions on Flexural Strength and Surface Roughness of Cobalt-Chromium Alloys produced by Selective-Laser-Melting

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Abstract

Post-heat treatment may enhance mechanical properties of selective laser melting (SLM) Cobalt chromium (Co-Cr) alloys, however it is unknown what duration the heat treatment needs to last and how the rate of cooling impacts the alloy's flexural strength and surface roughness. In this study, SLM Co-Cr alloy specimens were heated at 1150 C for 1 or 6 hours and subsequently cooled by air cooling (AC), furnace cooling (FC), or water quenching (WC). Flexural strength and surface roughness were then tested. Aim of this study: Investigate the effect of heat treatment and different cooling conditions on the flexural strength and surface roughness of SLM Co-Cr alloy specimens. The heat treatment will be conducted at 1150 C for two different durations (1-hour and 6-hours) and under three cooling conditions (AC, FC, and WC). Materials and methods: A total of forty-two rectangular specimens, measuring (34×13×1.5 mm) were manufactured through SLM and divided into seven groups, six specimens for each group as follows: The first group denoted as control with no heat treatment and no cooling applied, second and third groups heated for 1 or 6 hours then cooled in the air (AH-1 and AH-6), fourth and fifth groups heated for 1 or 6 hours and left inside the furnace until they cooled (FH-1 and FH-6), sixth and seventh groups heated for 1 or 6 hours and cooled by water quenching (WH-1 and WH-6). Flexural strength and surface roughness tests were performed on the specimens. A flexural strength value of (2147.33 MPa) was indicated that the control group exhibited the highest flexural strength, whereas the FH-6 group exhibited the lowest flexural strength value (1282.66 MPa). The control group showed the worst surface roughness (1.63 µm) while all other groups demonstrated no significant differences in surface roughness ranging from (0.32 to 0.49 µm) with a slight increase in the 6-hours heated groups. Conclusions: Slow cooling rate inside the furnace affects flexural strength negatively. Therefore, a high cooling rate is recommended to get a better flexural strength. On the other hand, surface roughness results suggests that 1-hour is better than 6-hours in terms of heat treatment duration and time saving.

Introduction:

The Removable Partial Denture (RPD) is an essential part of prosthodontic sciences, a field of dentistry that is concerned with the restoration and preservation of a patient's oral function, aesthetic appeal, comfort, and health by means of the artificial replacement of absent teeth and craniofacial tissues [1]. Cobalt chromium (Co-Cr) alloys are commonly used in the production of biomedical devices, such as dental devices, due to their exceptional mechanical properties, favorable biocompatibility, minimal wear rate, and higher resistance to corrosion [2]. Selective laser melting (SLM) is a form of additive manufacturing (AM) that has emerged as an alternative to the traditional lost-wax method for fabricating Co-Cr dental devices. Findings from scientific research indicate that the mechanical properties and metal release of Cr-Co components produced

through SLM technology surpass those of components produced through conventional casting methods [3,4]. These properties belong to the higher cooling rates employed in the SLM technique, which yield microstructures with finer grains compared to those generated by conventional casting methodologies [5]. As a consequence of the unique thermal cycle of rapid melting and cooling during the manufacturing process, SLM results in the creation of residual stresses within the components [6,7]. Post-heat treatments are necessary to minimize residual stresses and enhance the mechanical characteristics of the fabricated components [8].

Materials And Methods:

The specimens were fabricated using Co-Cr powder that is commercially available (Riton Model:C01, Foshan Rxton Technology Co., China) Table (1). A 3D design software 3ds Max ® (AutoDesk, USA) was used to design the 3D models. SLM machine (Riton D-150, Foshan Rxton Technology Co., China) Fig. (2) was used for the manufacturing process of the specimens by using standard settings for the mentioned powder at a laser power of 165 W, laser wavelength of 1.064 µm, laser speed of 1050 (mm/sec), as well as a layer thickness of 20 to 50 µm. After the SLM was completed, the specimens underwent heat treatment using a (riton rt 1300 Foshan Rxton Technology Co., China) furnace to begin the heat treatment process. The machine was operating in an atmosphere with Nitrogen gas. The building direction of 0-degree angle to the plate was used.

The heat treatment was conducted as follows: (1) increasing the temperature of the furnace to 760 C, (2) keeping this temperature for 10 minutes, (3) increasing the temperature to 1150 C, (4) keeping the 1150 C temperature for a duration of one hour or six hours., then (5) switching off the furnace. Heat treatment was performed on a rate of 10 C/min [5].

The first two groups of specimens were taken out from the furnace and cooled to room temperature in the open air after heat treatment, these groups were named AH-1 and AH-6. The second two groups were kept in the furnace until they reached room temperature, the names for these groups were FH-1 and FH-6, respectively. The final groups were taken out from the furnace after the 1 hour and 6 hours timer expired and cooled by quenching them in the water. These groups named WH-1 and WH-6, correspondingly. The control group was the only group that did not receive any heat treatment or cooling.

After separation from the plate, supporting structures were completely removed using a metal engine and tapered carbide burs. In order to preserve the dimensions of the specimens, the elimination of the green oxide layer was carried out with the use of stone burs (NHT02, Denmart, China) in accordance with the instructions provided by the manufacturer. Specimens were smoothed by using silicon wheels (silicon wheels, San I grinding wheel, Taiwan), and finally polished by using waterproof emery paper (ranging from 240 grit to 1000 grit) until they became reflective [5,11].

1. Flexural Strength Test

Forty-Two rectangular shaped specimens, six for each group with the dimensions of (34×13×1.5 mm) Fig (1) according to ISO 22674:2016 (Dentistry-Metallic Materials for Fixed and Removable Restorations and Appliances), were used to perform the flexural strength test [9,21]. The specimens were placed in universal testing machine (Gunt wp 300, Germany). Force was applied perpendicular to the longitudinal center-line. The crosshead speed was set to 1.5 mm/min until the specimen fractured. The distance between the supports was 20 mm, and the bending piston's diameter was 2 mm. Transverse strength is measured in MPa, by applying this formula

$$\textbf{Stress} = \frac{\textbf{3 x load x length}}{\textbf{2 x width x (thickness)}^2}$$

2. Surface Roughness Test

Surface roughness (Ra) test was performed on the rectangular shaped specimens (34×13×1.5 mm), (n = 3 for each condition), using a profilometer (Talysurf 10, Taylor Hobson, Metrology Division, Leicester, U.K). with the following parameters: 8 mm traverse length, standard critical wavelength 0.25 mm, velocity 0.1 mm/s. The measurements were done by using a 2-mm-diameter probe perpendicular to the polishing direction with a cutoff length of 0.4 mm. Each measurement was repeated three times on different locations of the specimen, and finally the mean value was calculated.

Results:

The statistical analysis was done using the SPSS program (version 29) (IBM Corp., NY, USA), employing descriptive statistics, normality tests, analysis of variance test ANOVA, in addition to multiple range test of Duncan to demonstrate the difference between groups at $P \leq 0.05$ significance level.

1. Flexural Strength Test

Specimen readings were calculated by measuring the maximum force applied prior to specimen deformation or fracture. The force was then converted into flexural strength using the following equation.

Flexural Strength αf = 3PL / 2wt2

Flexural strength was measured in mega Pascal (MPa). The data groups were subjected to descriptive statistics. Table (2) shows the mean, standard deviations, minimum and maximum values.

A normality test was performed on the obtained values Table (3). Shapiro-Wilk test revealed that the values were distributed normally ($P > 0.05$). One-way ANOVA test for the values of flexural strength for all groups viewed in Table (4). The analysis statistically demonstrates a highly significant difference ($P < 0.001$) between all the groups of different cooling conditions and the control group. Duncan's multiple range test Fig (3) showed that all groups significantly differ from control group and from each other with the highest value of tensile strength found in the control group. The test also showed that there was significant decrease in tensile strength in the furnace cooled (FH-6) group.

2. Surface Roughness Test

Readings were obtained by the use of the Profilometer for each specimen. The surface roughness of rectangular shaped specimens was tested (n = 3 for each condition). Three readings were taken from each specimen, and the mean value of them was recorded. Surface roughness (Ra) for each condition is shown in Table (5). The measurement unit of Ra is (micron µm). A normality test was performed on the values Table (6). The results of the Shapiro-Wilk tests indicated that the data adhered to a normal distribution, with a ($p < 0.05$). One-way ANOVA test for surface roughness values for all groups viewed in Table (7). The analysis statistically demonstrated a highly significant difference ($P < 0.001$) between the control group and all other groups of different cooling conditions. According to Duncan's multiple range analysis Fig (4), all groups differ significantly from the control group, which has the highest value of surface roughness. The test also showed that there was a decrease in Ra values in all groups compared to the control group. And the 6-hours groups showed increase in Ra compared to the 1-hour groups.

Discussion:

1. Flexural strength test

The results of the present study revealed that the flexural strength was the highest in the control group with no heat treatment or cooling applied to the specimens. Analysis of variance test (One-way ANOVA) coupled with Duncan's multiple range analysis demonstrated significant differences between all the groups and the control group, and the higher values were found to be in the one hour heat treated groups rather than the six hours, except for the water quenched group. SLM Co-Cr alloys were improved by post-production heat treatment by releasing residual stress, which led to a more homogeneous microstructure and improved mechanical properties such as flexural strength (10). Mergulhão et al., (12) studied the characteristics of Biometallic Co-Cr-Mo alloy to Additive Manufacturing using selective laser melting technology and reported that the Transverse strength (TRS) test results (2501.2 ± 9.7) MPa was satisfactory and met the ISO 22674:2016 standards. According to Alexandrino et al., (13), the build angle had an impact on the flexural strength and microhardness of the Co-Cr alloy produced by additive manufacturing, but not on the surface free energy or surface roughness. Padrós et al., (14) reported that chemical segregation, residual stresses, and porosity (both internal and external) were all observed in the microstructures of casting and SLM, all of which have an impact on the flexure load to fracture. Kocak et al., (15) came to the conclusion that metal frame weight can be decreased by changing the inner architecture, but this has no effect on flexural strength.

2. surface roughness

Surface roughness of Co-Cr varies depending on manufacturing processes, alloy composition, finishing and polishing technique, bur types, and polishing direction. Surface roughness has been extensively studied in biofilm production. According to in vivo studies, smooth surfaces attract less biofilm than rough surfaces (16). One-way ANOVA test and Duncan's multiple range analysis showed that all 6-hours heat treated groups had more surface roughness than the 1-hour groups. The Heat-treated Co-Cr-Mo clasp specimens for 6 hours at 0^{o} building direction showed the worst surface roughness among all other conditions due to the thick oxide layer formed after heat treatment (11). Vittayakorn et al., (17), Due to stress relief and thermal grain development, heat treated samples showed less porosity and surface roughness. Wai cho et al., (5) stated that there was no significant difference in surface roughness between Air cooled and furnace cooled specimens that were heat treated for 1 hour. Rahim et al., (18) concluded that the surface roughness quality of the Co-Cr denture framework produced by SLM is comparable to that produced by the conventional lost-wax casting method.

Shen et al., (19), stated that the roughness might speed up the deterioration of the opposing tooth enamel. Surface roughness causes mechanical interlocking to form. If the outer surface of the casting has excessive roughness or faults, further finishing and polishing are necessary. A poor technique can significantly increase surface roughness and produce surface defects. Hong et al., (9) stated that the difference between the three groups he studied (SLM, Casting and Milling) was not significant because there was no high polishing applied to the specimens in order not to generate excessive heat to the surface or change the surface properties. Mengucci et al., (20) noted that the average roughness of all analyzed samples shows an unexpected constant value, independently on the postproduction treatment.

Conclusion:

Flexural strength and surface roughness of Co-Cr alloy specimens fabricated using SLM and heated to 1150 C for one and six hours under three cooling conditions (Water quenching, Air, or furnace) were investigated. Within the limitations of this study, we reached the following conclusions. Increasing the cooling rate enhances the flexural strength compared to the slow cooling rate inside the furnace. Increasing the duration of heat treatment for 6 hours increases the surface roughness of the alloy, with no significant differences among the groups of different cooling conditions. Surface Roughness for all groups is much lower than control group. Heat treatment for one hour at 1150 C then water quenching of the alloy delivers the most convenient results in terms of time saving, flexural strength and surface roughness.

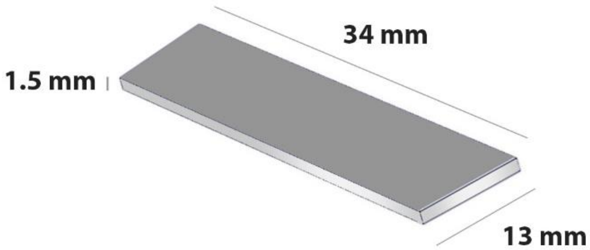
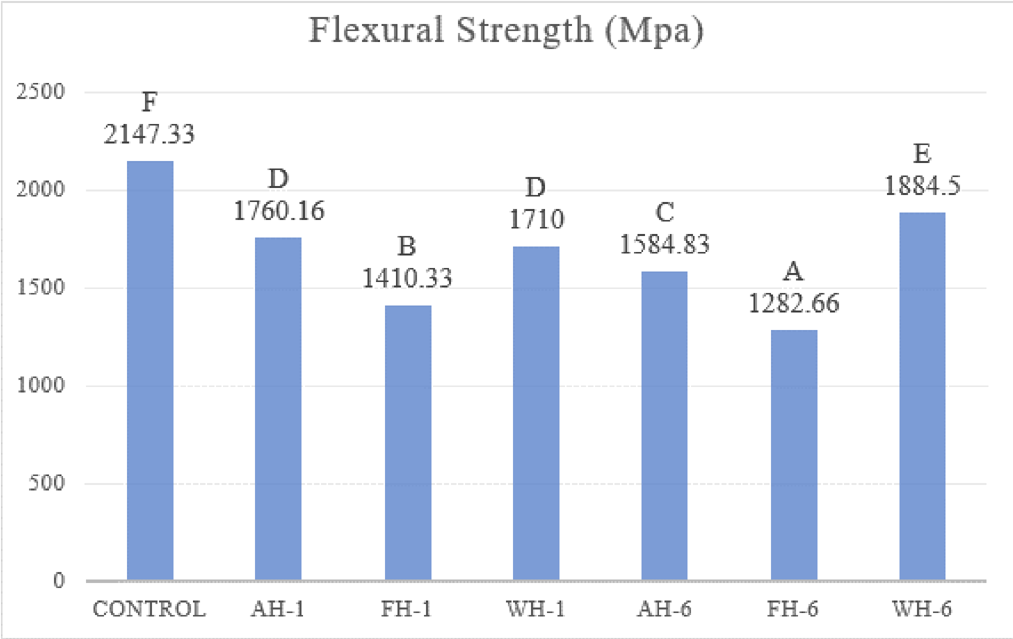
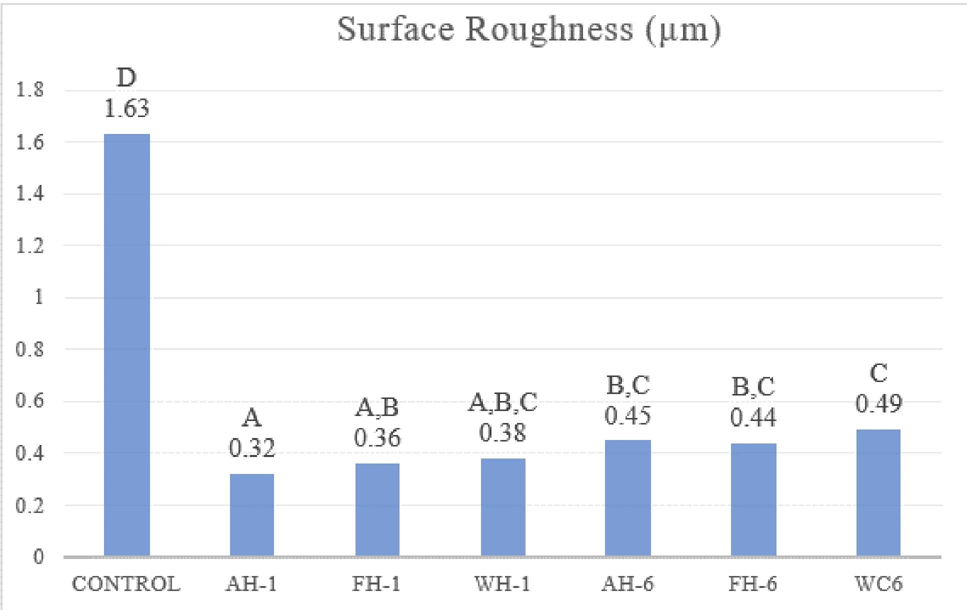


Fig (1): Test Specimen

Fig (2): SLM Machine



(A)



(B)

Fig (3): (A) Duncan's multiple range test for flexural strength (A: Lowest value, F: Highest value), and (B) Duncan's multiple range test for surface roughness(A: Lowest value, D: Highest value)

Table (1): Co-Cr powder Composition according to manufacturer

Co	60 %
Cr	28%
W	5 %
Mo	< 1 %
Si	< 0.75 %
Fe	< 0.75 %
C	< 0.16 %
Ni	< 0.1 %

Table (2): Descriptive analysis for Flexural Strength

	N	Mean	Std. Deviation	Minimum	Maximum
Control	6	2147.33	124.78408	1982.00	2265.00
AH-1	6	1760.16	117.62554	1628.00	1969.00
FH-1	6	1410.33	71.89344	1344.00	1528.00
WH-1	6	1710.00	56.00714	1661.00	1810.00
AH-6	6	1584.83	101.34972	1450.00	1712.00
FH-6	6	1282.66	67.29537	1220.00	1407.00
WH-6	6	1884.50	89.84153	1794.00	2051.00
Total	42				

Table (3): Normality test for Flexural Strength

Group	Shapiro-Wilk		
	Statistic	df	Sig.
Control	.847	6	.149
AH-1	.924	6	.535
FH-1	.868	6	.218
WH-1	.870	6	.226
AH-6	.933	6	.600
FH-6	.855	6	.173
WH-6	.866	6	.209

Table 4: ANOVA Test for flexural strength

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	3042850.667	5	507141.778	58.596	<.001
Within Groups	302921.167	35	8654.890		
Total	3345771.833	40			

Table (5): Descriptive statistics for Surface roughness

	N	Mean	Std. Deviation	Minimum	Maximum
Control	3	1.63	.12288	1.54	1.77
AH-1	3	.32	.03606	.28	.35
FH-1	3	.36	.04933	.31	.40
WH-1	3	.38	.04726	.33	.42
AH-6	3	.45	.03512	.42	.49
FH-6	3	.44	.02082	.43	.47
WH-6	3	.49	.02082	.47	.51
Total	21				

Table (6): Normality Test for Surface Roughness

	Shapiro-Wilk		
	Statistic	df	Sig.
Control	.876	3	.312
AH-1	.942	3	.537
FH-1	.832	3	.194
WH-1	.907	3	.407
AH-6	.993	3	.843
FH-6	.923	3	.463
WH-6	.923	3	.463

Table (7): ANOVA test for surface roughness

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	3.890	2	.648	195.907	<.001
Within Groups	.046	14	.003		
Total	3.936	16			

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