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Transverse Strength and Microstructure of Cobalt-Chromium Alloy Produced by Selective Laser Melting and Casting Techniques (An *in vitro* Study)

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ABSTRACT

Aim: Investigate the differences between the transverse strength and microstructures of specimens produced by two techniques, the conventional casting technique and selective laser melting technique.

Materials and methods: Co-Cr alloy specimens are manufactured by two different techniques; conventional casting of resin pattern, selective laser melting techniques. Each technique was used to manufacture twelve specimens, making a total of twenty four. Each group was used for testing transverse strength. Two specimens of each group of transverse are used for a scanning electron microscope to show the fractured and non-fractured side microstructure. Differences in transverse strength were statistically analyzed by using the independent sample T-test.

Results: Transverse strength of the selective laser melting specimens had a significantly higher value compared to casting specimens. Scanning electronic microscope revealed casting group displayed inhomogeneous grain size (20-100) micrometer while laser group shows homogeneous grain size (2-20) micrometer.

Conclusion: As laser has a better transverse strength than casting, scanning electron microscope displays fine crystalline structure while casting has big crystalline features.

Key words Cobalt chromium alloy, Transverse strength, SEM, Laser melting specimens, Scanning electronic

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INTRODUCTION

Cobalt-Chromium (Co-Cr) are base metal dental alloys. Co-Cr alloys are largely employed in the production of double crown, precision attachment, conventional clasp retained and combined, removable partial dentures [1,2]. Because of their high strength, high corrosion and wear resistance, excellent biocompatibility and relatively low cost [3,4]. The weight alloy for removable partial dentures should be no less than 20% and the overall weight of chromium, cobalt and nickel should be no less than 85%, according to ANSI/ADA specification no. 14. The alloys meet the standards for toxicity, hypersensitivity and corrosion in a satisfactory manner. The density of these alloys ranges from 7.6 to 8.3 g/cm^3 [5]. Alloys for metal ceramic prostheses (PFM) should have the potential to bond to dental porcelain which requires adherent oxides on the surface and thermal contraction coefficients that are comparable to those of dental porcelain [6].

Conventional casting considered as the old method of manipulation of Co-Cr. Wax and resin used in the production procedure, resins are more durable and have less flow than pattern waxes and burnout without residue. Melting Co-Cr alloys requires ideal heating sources, their fusion temperatures range from 1150°C to 1500°C. The induction system allows for superior temperature control, resulting in a homogeneous mass of all alloy components, lowering the risk of oxidation and contamination of the molten alloy [7-10].

3D printing is a type of additive manufacturing method that involves depositing successive layers of material to build a 3D item. This technology is now being widely used with metal powders to fabricate dental restorations and prostheses [11,12].

Selected Laser Sintering (SLS) technology was developed and patented by Joe Beaman and Carl Deckard in 1989 [13]. Manufacturing processes are getting more automated and digital dental technology is becoming more ubiquitous. This technique is a direct import from 3D printing and rapid prototyping technologies that are often utilized in [14,15].

The laser beam is focused on the powder and the laser's energy is sufficient to melt it. By adjusting the wavelength, laser source and power, the melting process may be fine-tuned. Due to the heat gradients created during manufacturing, substantial internal stresses are seen in materials. Additional heat treatment is required to alleviate these strains [16-18].

SLM alloys' unique microstructure determines their superior mechanical qualities. Metal framework mechanical qualities, particularly flexural strength, play an important role in the long term clinical effectiveness of metal ceramic restorations [19,20].

Fine cellular dendrites, uniform microstructure due to the high solidification rate during the SLM process and dendritic elongated grains made comprised the microstructure of the SLM processed metal [21]. The surface microstructure of the alloy produced by casting is heterogeneous in turn. It determines the alloy's properties in biological situations where it comes into direct touch with bodily fluids [22].

The aim of this study is to show how Co-Cr produced by two different techniques responds to transverse strength and after flexural strength testing and fractured of specimen how microstructure look like.

The null hypothesis suggested that would be no difference in transverse strength and microstructure of selective laser melting and conventional casting techniques.

MATERIALS AND METHODS

Preparation of resin samples

Co-Cr alloy specimens were manufactured by two different techniques; conventional casting of resin pattern, selective laser melting techniques. Total specimens twenty four of two techniques, twelve specimens of each group were used for testing transverse strength. Accordance to ISO 22674:2016 measurement of the specimen of transverse test 34 mm length, 13 mm width and 1.5 mm thickness.

Laser specimens was fabricated by selective laser melting machine (D-150, Riton, China) and the Co-Cr powder alloy (Cocrmow, Mti, China), while casting specimens produced by electric induction casting machine (Dentamatic 3000, Tokmet, Bulgaria). The ingot used was (Cocrwmo, Schefter, Germany). Both group were sand blasted with 250 mm Al_2O_3 powder (strahltechnik, Renfert, Germany) and then finished with finishing of specimens, with tapered carbide bur. Smoothening is done by polishing wheels.

The final dimensions of all specimens were verified by using digital calipers (DCP-300 n, PCE, UK).

Evaluation of transverse strength and microstructure

The specimens were placed in a three point device (microcomputer testing machine, Jianqiao, China). Force applied perpendicular to the longitudinal centerline, resulting in a chisel like form between the centers of support. A compression load cell with a maximum capacity of 5 kV was used to measure the length.

Transverse strength: Using a universal testing machine with a load at the center and a 20 mm spread between the supporting points (L) and a 1.5 mm/min crosshead speed. The loading plunger was in the center, with the test samples held at either end of the two supports. The diameters of the supports and plunger were both 2 mm. Transverse strength measured in N/mm^2, by applying this formula transverse strength can be calculated $R=3WL/2bd^3$ [23,24].

Scanning Electronic Microscope (SEM): Two specimens were selected from each technique for scanning, one done by unbroken surface scanning and the other one by unbroken surface. SEM device used (inspect F 50, FEI, USA) different image has been taking magnification ranged from 40-4000 x, HV 10.00 KV, pressure 1.15 e-4 pa inspected field measurement by micrometer according to manufacturer instructions [25,26].

Statistical analysis

Transverse specimens readings calculated by measuring the highest force applied before deformation or fracture of specimen, to transform this force into transverse strength the following equation is used $R=3WL/2bd^3$ (N=12) for each production technique. Data were analyzed statistically by using (SPSS, version 26). The collected values were subjected to normality testing. The Shapiro-Wilk and Kolmogorov-Smirnov tests found that the values followed a distribution that is normal.

RESULTS

Table 1 show descriptive statistics were applied to the two groups of data. The means, standard deviations and standard errors, in addition the minimum and maximum values.

Table 1: Means, SD and standard errors, minimum and maximum values of transverse strength of two group.

Group	N	Minimum	Maximum	Mean	Std. error	Std. deviation
Casting	12	980	1157	1075.833	18.35543	63.58507
Laser	12	1395	1770	1587.5	37.80422	130.9577
Total	24					

When the independent sample T test is applied to laser and casting values, it reveals that the variances are equal because sig>0.05 in levene's test for equality of variances between two groups. While the equality of means T test reveals, between casting and laser, there is a significant variation in transverse strength. Sig<0.05 suggests that there is a transverse strength difference between casting and laser. According to mean value laser has more transverse strength than casting.

Scanning electronic microscope: Figures 1 and 2 show SEM images of fragmented Co-Cr specimens, while Figure 3 displays the Co-Cr specimens' original intact smooth surfaces. The dendritic solidification microstructure in the casting group was in homogeneous, with dendritic zones and inters dendritic areas (Figure 2A). Casting specimens had a grain size of 20 µm–100 µm. SLM groups with grain sizes of 2 µm–20 µm showed a substantially finer and non-equilibrium structure.

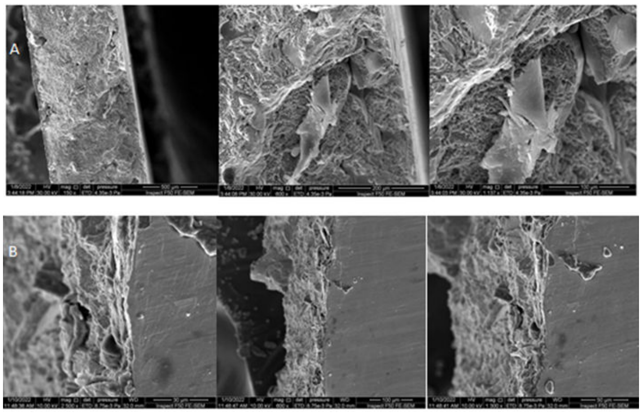

Figure 1: Scanning Electronic Microscope (SEM) pictures of broken surfaces created by; (A) Casting on Co-Cr alloy specimens (150-1100 mag); fracture area indicates irregular dendritic structure; (B) SLM, (600-2500 mag); fractured area indicate cleavage steps.

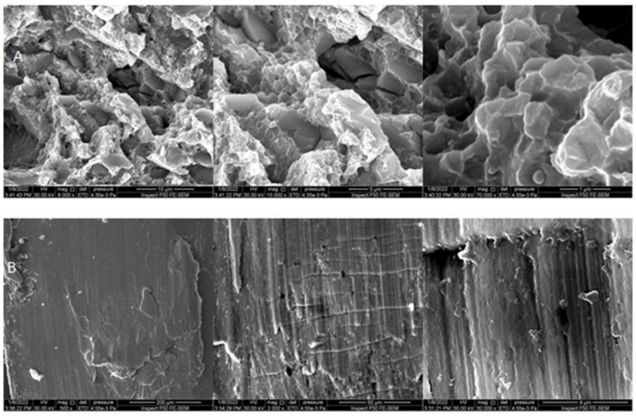

Figure 2: SEM pictures of Co-Cr alloy specimens with broken surfaces created by; (A) Casting (8000-70000); large crystalline structure indicate dendritic structures); (B) SLM (500-30000); area indicate cleavage steps.

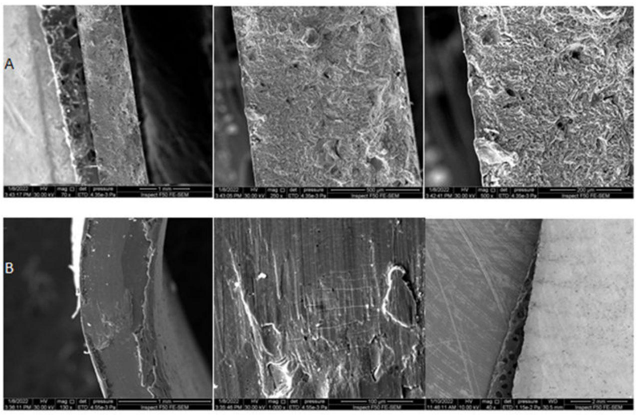

Figure 3: SEM pictures of Co-Cr alloy specimens created by; (A) Casting with their original unbroken surfaces (70-500); the dark colored portions represent dendritic crystal formations that are irregular; (B) SLM (40-1000) there is microscopic crystalline formations that seem like white grains.

DISCUSSION

In the oral environment, the material is subjected to mechanical loading. When teeth or structures that replace teeth come into contact, such as during chewing, swallowing or bruxism, mechanical loading occurs, so it needs specific mechanical properties [27].

Depending on the primary purpose of the prosthesis, the choice of Co-Cr alloy is made by the dentist in collaboration with a dental technician. From this standpoint, several properties should be available in the alloy to be used. But most importantly, in this study, we focus on transverse strength and microstructure. Because they are most relevant to our demand in this study.

The mechanical characteristics of Co-Cr dental alloys were examined and compared using two different manufacturing methods (casting and selective laser melting techniques). The results of the study clearly demonstrated that the mechanical characteristics of alloys are highly dependent on production procedures as well as the chemical compositions of the alloys utilized. As a result, the null hypothesis that the groups made using different production procedures would have equal mechanical properties was rejected.

The specimens that will be used in transverse strength testing. The casting group had the largest elastic modulus, which means that a certain level of stress is necessary to distort a material. The laser group had a greater yield and flexural strength. Lower yield strength indicates lower levels of plastic deformation stress, while lower flexural strength indicates lower levels of flexion fracturing stress [28].

The mechanical characteristics of SLM and casting Co-Cr alloys were compared and casting group specimens were found to be inferior to SLM group specimens [29].

The casting group was tougher and absorbed more overall energy before breaking. The SLM group was the most vulnerable because it shattered at a low strain level [30].

SLM framework specimens were brittle and harder than castings, according to Co-Cr alloy bridges, but the SLM group was comparatively brittle due to breaking under less strain. The qualities of the final product can be affected by parameters such as, layer thickness, building direction and scan speed in the SLM manufacturing method [31].

SLM Co-Cr alloys benefit from post production heat treatment for releasing residual stress, resulting in a more homogeneous microstructure and improved mechanical properties [32]. The production process has an impact on the mechanical properties of the Co-Cr alloy.

The flexural strength and micro hardness of the Co–Cr alloy generated by AM were influenced by the build angle; however, it had no effect on surface free energy or surface roughness [33].

Metal framework mechanical properties, particularly flexural strength, are critical to metal ceramic restorations' long term clinical success.

The laser sintered metal frames had a flexural strength is higher than cast metal structures. The test group sintered with the shortest layer thickness had the highest flexural strength values, so the framework length increased, the flexural strength decreased [34]. Microstructure has a substantial impact on the mechanical performance of dental Co-Cr alloys. Cast specimens all exhibit a fibrous topography, which is caused by a higher plastic deformation till fracture. The size and location of segregations have a significant impact on the mechanical characteristics that affect fracture mechanisms [35].

The mechanical properties of the SLM alloy were improved. For metal ceramic restorations, Co-Cr dental alloy manufactured with SLM offers a viable alternative to conventional cast alloy [36]. SLM Co-Cr was less porous and had better mechanical properties than cast Co-Cr [37]. For constructing RPD frameworks, SLM followed by heat treatment is an effective method [38]. SLM technology can be used to make dental bridges and crowns, according to ISO 22674:2016 and ISO 9693-1:2012. For fixed dental restorations, SLM may be preferable to standard fabrication procedures [39].

Flexural strength of different settings additively manufactured commercial Co-Cr dental alloys shows different mean values. Sandblasting with Al_2O_3 and welding decrease flexural strength of Co-Cr alloy [40]. The flexural strength of casted samples is clinically acceptable [41]. Changing the interior architecture of metal frames reduces their weight but has no effect on their flexural strength or flexural modulus [42]. Porosity (inner and surface), residual stresses and chemical segregation were all found in the microstructures of casting and laser, all of which play a role in flexure load to fracture [43].

The results for flexural strength measurement in this research proved to be significantly superior in specimens produced by selective laser melting to those produced by casting techniques.

This is because flexural strength increases when the microstructure is uniform, fine grained and less porous. As a result, the SLM group's microstructure may justify its enhanced flexural strength. Microstructural heterogeneity and the existence of large holes in the cast group can operate as stress concentrators, causing failures; hence its reduced flexural strength may be justified [44].

The SEM study of the specimens reveals a generally evenly annealed microstructure with the typical shape of additive manufacturing technologies. Local porosity yellow circles have also been discovered in the specimens. There were no obvious variations in any of the selected locations. With grain sizes ranging from 50 to 100 μm and homogenous surfaces on the casting group nano scale crystalline forms on the SLM group's unbroken surface were discovered, whereas the fractured surface had a layered structure. It can be seen that the fracture of the casting group's broken surface happened simultaneously with the dendritic structure. The broken surface of the SLM group showed stair like cleavage steps, which are common in brittle materials.

After heat treatment, in the as built samples' x-y and x-z planes, many fine cellular and columnar dendritic features were less evident. In contrast, as cast samples had substantially bigger precipitates [45].

Due to dendritic segregation, the dendritic and inter dendritic zones have different solute distributions, which could affect mechanical properties.

The SLM groups, on the other hand, demonstrated a homogeneous and dense structure without pores, as well as complete local melting and quick solidification, defects and porosities are reduced. This could account for the SLM specimens' higher mechanical characteristics when compared to the casting group. Furthermore, the smallest grain size was found in the SLM samples, followed by the casting samples. When a fast cooling method like SLM is used instead of a long cooling process like casting, smaller grains are obtained [46].

Only the finest powder with an exceptionally narrow grain size distribution was chosen for the SLM powder [47]. When compared to the casting method, the Co-Cr alloy produced by SLM had significantly finer grains and better mechanical properties. Varied grain sizes can contribute to different mechanical characteristics in other alloys [48]. Grain refining can improve the alloy's strength, ductility and toughness, which could explain why SLM outperforms casting in terms of mechanical performance. Furthermore, rapid solidification of SLM specimens may improve mechanical performance by increasing the solute element solution limit and reducing dendritic segregation. Rapid solidification might strengthen the solution by preserving a high level of the supersaturated solid solution ingredient would precipitate later, enhancing the second phase strengthening effect.

Because of the entire local melting and rapid cooling of the SLM printing process, a dense structure with very homogenous grains and few porosities and defects may be produced, which explains the homogeneity.

The first microstructure of the selective laser melted Co-Cr alloy has a "fish scale" structure, which is a typical melt track created during the layer by layer laser melting process. SEM micrographs taken at low magnification show distinct melt track structures as well. The SLM Co-Cr alloy has a homogenous structure along the melt track, with elongated columnar grains and cellular like sub-grains [49].

The microstructure of the Co-Cr alloy is determined by the production process. In our research, SLM produces a more homogeneous surface with tiny grain size, whereas casting produces an irregular distribution of large scale size dendrites on unbroken surface while, in fractured surface in SLM stair like cleavage steps layer by layer, casting shows irregular dendritic structure.

Due of the quick cooling process based on laser melting, SLM has a homogeneous matrix. While, casting show presence of carbon and oxygen in carbides and oxides precipitates can affect their mechanical qualities. In the Co-Cr alloy microstructure, black patches represent oxides and dark white patches indicate carbides [50].

The solidification mechanism is determined by the cooling rate. While slow cooling during casting results in dendritic solidification, excessive cooling rates in SLM result in cellular solidification. Although some materials go through cellular eutectic solidification, the eutectics solidify as an intercellular network [51].

CONCLUSION

Within the limitation of this *in vitro* study, the following was concluded:

- Mechanical properties of SLM show a better result as compared to casting. SLM shows better transverse strength than casting.
- The microstructure of show homogenous surface with fine grain in SLM surface, while large irregular crystalline in casting.

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