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Original Research Article

Comparison of fatigue behaviour and fracture toughness of cobalt chromium alloy fabricated by casting and selective laser melting methods

Chembeti Harika[1,*], K Mahendranadh Reddy[1], Venkat Adithya[1]

[1] *Dept. of Prosthodontics and Implantology, Sri Sai College of Dental Surgery, Vikarabad, Telangana, India*

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ABSTRACT

Background: Co-Cr alloys are most commonly used base metal alloy in dentistry for the fabrication of many types of metallic substructures. They gained popularity due to their good mechanical properties and low cost. There are different techniques for fabrication of Co-Cr alloys which includes casting and DMLS technique. However, change in the fabrication techniques showed variation in mechanical properties.
Materials and Methods: Forty Co-Cr samples each were made from two different techniques, Casting and DMLS. Fracture toughness was done with charpy's impact test machine and fatigue behaviour test was done with nano universal testing machine. The samples were later observed under stereo electron microscope to assess the kind of failure.
Results: Unpaired t test revealed that there was statistically significant difference between two different groups. The mean difference of fracture toughness in Casting group was higher when compared with DMLS group. The number of fatigue cycles were higher in DMLS group than the casting group.
Conclusion: Within the limitations of this in vitro study, casting group showed higher fracture toughness when compared to DMLS group, whereas fatigue limit was higher in DMLS group than casting group.

1. Introduction

Dental alloys represent a very dynamic field of dentistry. Changes that occur in this area, in fact, reflect the developments of basic scientific technologies. Mechanical and biological properties of the same alloy are largely dependent on the technological processes of forming the alloy into dental restorations.[1] The process of alloy melting and casting for dental purposes has been known for centuries and melting and casting conditions have been constantly improved – from primitive alloy melting by applying naked flame and open-air casting, to melting by applying induced current in vacuum or neutral gases. Nevertheless, even the perfect casts have certain flaws.[2]

A completely new approach to forming dental restorations appeared with the third, and soon after, with the fourth industrial revolution. The third industrial revolution, also known in the field of dentistry as Digital Dentistry or Dentistry 3.0, introduced numerous new procedures based on digital technologies (3D imaging, intraoral scans, computer-aided design and computer-aided manufacturing – CAD-CAM, cone-beam computer tomography – CBCT, computer-aided implantology).[3] The transition from the third to the fourth industrial revolution, i.e. to Dentistry 4.0, was barely noticeable. Dentistry 4.0 is not a completely new technology. In this case new system solutions were created on the platform (infrastructure) originating from the previous digital revolution. This revolution introduced greater automation in dental laboratory procedures, i.e. diagnostic and therapeutic procedures in dental offices. The selective laser melting (SLM) and compacting (sintering)

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* *Corresponding author*.
E-mail address: harika.chembeti@outlook.com (C. Harika).

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of metal powder particles is a step forward in the modern dental practice. This technology plunged us into the fourth industrial revolution, i.e. Dentistry 4.0.[4] The process of making dental restorations by sintering dental alloys basically includes three steps: digital impression, designing virtual restoration, and 3D printing.[5] Digital impression, suitable for further computer processing, can be obtained by direct 3D.[6]

Clinical success of the any prosthesis is based on the mechanical properties of the material used. Due to difference in the fabrication techniques the properties of the alloy are influenced. In the mechanical properties, fracture toughness and fatigue behaviour are tested in this study.

The purpose of the study is to evaluate the fatigue behaviour and fracture toughness of Co-Cr alloy fabricated by conventional casting and DMLS method. The null hypothesis of the current study is that there is significant difference in fracture toughness and fatigue behaviour between the specimens fabricated by casting method and DMLS method.

2. Materials and Methods

1. Sprue wax – 4mm and 2mm diameter
2. Investment material [Brevest investment material]
3. Vaccum mixer [Tornado]
4. Casting ring liner
5. Cobalt chromium alloy [scheftner dental alloys]
6. Crucible
7. 3D printing machine [Phrozen sonic mini 4k]
8. Burn out furnace [Unident]
9. Centrifugal casting machine [Unicon]
10. Recyclable sand blaster
11. Carborundum discs
12. Metal finishing points
13. Laser sintering machine [concept laser]
14. Universal testing machine [ITW Biss]
15. Charpy’s test machine [IMPACT]
16. Stereo microscope [4x magnification NANBEI]

2.1. *Methodology*

A total of 40 specimens were fabricated for this study. All the 40 specimens were randomly divided into two groups, Group 1 – specimens for fatigue behaviour testing and Group 2 – specimens for fracture toughness testing. Each group was subdivided into two subgroups (i) specimens fabricated by casting method or casting group (ii) specimens fabricated by direct laser metal sintering method or DMLS group, with ten samples in each subgroup (n = 10).

2.2. *Preparation of specimens for fatigue behaviour test*

2.2.1. *Casting method*

An STL file for dumbbell shaped specimen was designed by using a software – siemens nx12 with the dimensions of 54.8mm of total length in which length of jig holding area was 14mm with 6mm diameter on both sides and length of testing area in the centre was 26.8mm with 4.37mm diameter. Ten wax patterns were printed for the samples by using Phrozen sonic mini 3D printer. The STL file was fed into machine and wax material was placed on the platform. The printed wax patterns were obtained and were checked for the dimensions with digital vernier callipers. The 3D printed wax patterns were sprued. One sprue of diameter 4mm was attached to the pattern at the centre of the pattern at 45^0 angle and attached to crucible former. The length of the sprue was maintained for 5mm and patterns were arranged such that, they were 6mm below the top of the casting ring. The sprue was held with forceps and inserted into the hole in the crucible former.

Two auxillary sprues of 2mm gauge shaped into oval form and were attached on two corners of the pattern. The junction between sprue and crucible was smoothed. The casting rings were lined with moistened liner. The wax patterns were sprayed with surface tension reducing agent. Phosphate bonded investment material (brevest investment material) was mixed by taking 480g of powder with 91.5ml of liquid and 4.5ml of water in a mixing bowl and hand spatulation was done for 30 seconds followed by mechanical spatulation for 60 seconds by vaccum mixer according to manufacturers instructions. The investment material was brushed on to the pattern gently from a single point. Then slowly the rings were filled with investment material up to the rim and they were allowed to set for 20minutes. The moulds were placed in a preheated burnout furnace at 350^0 C maintained for 60 minutes and gradually increased to 700^0c to 1030^0c and maintained for 30 minutes.

Casting was done in a centrifugal casting machine and Co-Cr alloy pellets were melted at a temperature of 1300^0c. After the alloy filled the moulds, they were removed from the casting machine and allowed to cool gradually until they reached to room temperature. A knife was used to trim the investment at the bottom end of the ring and the ring liner was exposed. Then investment was pushed out of the ring. It was then broken apart under running water. The remaining investment was carefully removed with a small blunt instrument and any traces were dissolved in hydrofluoric acid. The sprues were cut with carborundum disc and finishing was done with metal finishing points followed by polishing which was done by sandpaper discs. As samples were ready, their dimensions were measured with digital vernier calliper.

2.2.2. By Selective laser melting method

An STL file for dumbbell shaped specimen was designed by using a software – siemens nx12 with the dimensions of 54.8mm of total length in which length of jig holding area was 14mm with 6mm diameter on both sides and length of testing area in the centre was 26.8mm with 4.37mm diameter. Ten samples of DMLS were designed in the system (COMPACT LASER). The device (SLM 125) was equipped with laser of 400 W power, scanning speed of 10m/second, beam diameter of 70-100 μm and variable Layer Thickness: 20 μm – 75 μm. The Co-Cr alloy fine metal powder was dispensed on the platform and spreaded evenly, the laser beam on top sintered the metal locally and the powder got fused, forming a thin layer. A series of layers were formed which eventually fabricated dumbbell shaped specimens. Prepared samples dimensions were measured with a digital vernier calliper.

2.3. Preparation of samples for fracture toughness test

2.3.1. By casting method

An STL file (Figure 1) for rectangular shaped wax pattern with a V notch in the middle of sample of standard dimensions (55 mm x 10 mm x 2.5 mm) was designed in a software – simens nx. Ten wax patterns were printed for the samples by using Phrozen sonic mini 3D printer. Ten wax patterns were printed for the samples by using Phrozen sonic mini 3D printer. The STL file was fed into machine and wax material was placed on the platform. The printed wax patterns were obtained and were checked for the dimensions with digital vernier callipers. The 3D printed wax patterns were sprued. One Sprue of diameter 4mm was attached to the pattern at the centre of the pattern at 45^0 angle and attached to crucible former.

The length of the sprue was maintained for 5mm and patterns were arranged such that, they were 6mm below the top of the casting ring. The sprue was held with forceps and inserted into the hole in the crucible former. Two auxillary sprues of 2mm gauge shaped into oval form and were attached on the two corners of the pattern. The junction between sprue and crucible was smoothed. The casting rings were lined with moistened liner. The wax patterns were sprayed with surface tension reducing agent. Phosphate bonded investment material (brevest investment material) was mixed by taking 480g of powder with 91.5ml of liquid and 4.5ml of water in a mixing bowl and hand spatulation was done for 30 seconds followed by mechanical spatulation for 60 seconds by vaccum mixer according to manufacturers instructions.

The investment material was brushed on to the pattern gently from a single point. Then slowly the rings were filled with investment material up to the rim and they were allowed to set for 20minutes. The moulds were placed in a preheated burnout furnace at 350^0 C maintained for 60minutes and gradually increased to 700^0c to 1030^0c and maintained for 30 minutes. Casting was done in a centrifugal casting machine and Co-Cr alloy pellets were melted at a temperature of 1300^0c. After the alloy filled the moulds, they were removed from the casting machine and allowed to cool gradually until they reached to room temperature. A knife was used to trim the investment at the bottom end of the ring and the ring liner was exposed. Then investment was pushed out of the ring. It was then broken apart under running water. The remaining investment was carefully removed with a small blunt instrument and any traces were dissolved in hydrofluoric acid. The sprues were cut with carborundum disc and finishing was done with metal finishing points followed by polishing which was done with sandpaper discs. As samples were ready, their dimensions were measured with digital vernier calliper.

2.3.2. By selective laser melting method

An STL file for rectangular shaped specimens with v notch was prepared by using a software – siemens nx12 with dimensions of (55mm x 10mm x 2.5mm). For the fabrication of ten samples, the data was transferred into the machine. Ten samples of DMLS were designed in the system (COMPACT LASER). The device (SLM 125) was equipped with laser of 400 W power, scanning speed of 10m/second, beam diameter of 70-100 μm and variable Layer Thickness: 20 μm – 75 μm. The Co-Cr alloy fine metal powder was dispensed on the platform and spread evenly, the laser beam on top sintered the metal locally and the powder got fused, forming a thin layer. A series of layers were formed which eventually fabricated rectangle shaped specimens (Figure 1). Prepared samples dimensions were measured with a digital vernier calliper. Now that all the specimens were fabricated in standardized dimensions in two methods, they were tested for fatigue behaviour and fracture toughness.

2.4. Fracture toughness test

The Charpy impact test measured the energy absorbed by a standard notched specimen while breaking under an impact load. The specimens with notch in the middle in standard dimensions were placed on the platform. Pendulum was raised and released with the specimen in place. Pendulum hits the specimen and raised to a height after breaking the specimen. The height of the pendulum raised was noted.

The results obtained were statistically analysed.

2.5. Fatigue behaviour test

The fatigue behaviour was measured using Universal Testing Machine. The specimens were placed such that they were held by the upper and lower jigs on either sides. The specimens subjected to load and the load was applied in cycles.

1. Testing parameters: Mean: 0.410KN
   Amplitude: 2
   Frequency: 4.5 HZ
2. The force at which the metal broke was noted.
3. The obtained values were calculated and statistically analysed.

The broken surfaces of the dumbbell shaped specimens were observed under stereo electron microscope.

Stereoelectron microscope was used for the observation of the samples by light reflected from the surface of the sample rather than transmitted through it. In this instrument two separate optical paths with two objectives and eyepieces were used to provide slightly different viewing angles to the right and left eyes. This arrangement produced a three-dimensional visualization of the sample. Here 10 x magnification was used to observe the samples and images were taken.

3. Results

The Fracture toughness values obtained were tabulated and subjected to statistical analysis. The values of casting and DMLS group were compared.

Table 1: Values obtained in the fracture toughness test for casting group and DMLS group

Sample	Casting Group	DMLS Group
Sample 1	28 joules	24 joules
Sample 2	24 joules	24 joules
Sample 3	28 joules	24 joules
Sample 4	28 joules	24 joules
Sample 5	28 joules	2O joules
Sample 6	32 joules	24 joules
Sample 7	28 joules	24 joules
Sample 8	24 joules	24 joules
Sample 9	28 joules	24 joules
Sample 10	28 joules	24 joules

The raw data in the above table shows the values of the fracture toughness of casting group and DMLS group. The highest value in casting group was 32 joules and least was 24 joules. The highest value in DMLS group was 24 joules and 20 joules.

Table 2: Fracture toughness of Co-Cr alloys fabricated by casting and DMLS (mean, standard deviation SD)

Group	Fracture toughness	
	Mean	SD
Casting	27.60	2.57
DMLS	23.60	1.26
p-value	<0.001	

Mean and standard deviation of fracture toughness of two groups were mentioned in the above table. The mean of casting samples was 27.60 and the standard deviation was 2.57. The mean of DMLS group samples was 23.60 and the standard deviation was 1.26. Unpaired t test was performed to compare the mean difference amongst two groups.

INFERENCE: unpaired t test was used to analyse the data of fracture toughness test .The mean value and standard deviation (SD) of casting group were 27.60 and 2.57 respectively. The mean value and standard deviation (SD) of DMLS group were 27.60 and 2.57 respectively. The test indicated there was a significant difference between both the groups with p value of <0.001.

The Fatigue behaviour values obtained were tabulated and subjected to statistical analysis. The values of casting and DMLS group were compared.

The raw data in the above table shows the values of the fatigue behaviour of casting group and DMLS group. The highest load value in casting group was 0.43 KN and least was 0.033 KN. The highest load value in DMLS group was 0.045 KN and 0.033 KN. The highest number of cycles in casting group was 76302 and least number of cycles was 54956. The highest number of cycles in DMLS group was 99980 and least number of cycles was 79926.

Mean and standard deviation of fatigue behaviour of two groups were mentioned in the above table. The mean load of casting samples was o.37 and the standard deviation was 0.005. The mean load of DMLS group samples was 0.38 and the standard deviation was 0.003. The mean of number of cycles of casting samples was 67801.30 and the standard deviation was 6628.12. The mean of number of cycles of DMLS group samples was 97001.50 and the standard deviation was 6072.49.Unpaired t test was performed to compare the mean difference amongst two groups.

4. Discussion

The results obtained are in accordance with relevant data found in the literature referring to the chemical composition of EOS Co-Cr SP2 alloy determined based on EDS analysis, but also based on X-ray diffractometry analysis (XRD) performed by other authors.[7] Chemical compositions of alloys differ slightly depending on the manufacturer and the surface that the analysis was performed on. The microstructure of the sintered Co-Cr alloy is lamellar in nature, with two dominant phases: ε-Co and/or ε-Cr (fcc – face-centered cubic) and γ-Co (hcp – hexagonal close-packed). This structure was determined based on XRD analysis.[8] The microstructure of two types of samples is observed: a sintered sample and a sintered and thermally treated sample. The same structure with slightly lower intensity of peaks is determined with the thermally treated sample.[9]

Microstructure of the sintered Co-Cr alloy does not indicate intermetallic phases, contrary to the cast Co-Cr alloy. Upon casting, Co-Cr alloys create an intermetallic phase (Cr7 C3 and Cr23C6).[10] In theory, the structures obtained by applying SLM technology are not porous.

Table 3: Values obtained in the fatigue behaviour test for casting and DMLS group

Specimens	Casting group		DMLS group	
	Load	No. of cycles	load	No. of cycles
Sample 1.	O.033	74601	0.045	97563
Sample 2	0.037	64798	0.040	79926
Sample 3	0.045	69343	0.040	99902
Sample 4	0.033	70109	0.037	97930
Sample 5	0.042	54956	0.041	98567
Sample 6	0.033	76302	0.039	99740
Sample 7	0.040	70785	0.037	98952
Sample 8	0.036	58901	0.040	99721
Sample 9	0.033	70456	0.033	97834
Sample 10	0.043	67762	0.037	99980

Table 4: Fatigue behaviour of Co-Cr alloys fabricated by casting and DMLS (mean, standard deviation SD)

Group	Load		Cycles	
	Mean	SD	Mean	SD
Casting	0.37	0.005	67801.30	6628.12
DMLS	0.38	0.003	97001.50	6072.49
p - value	0.38		<0.001	

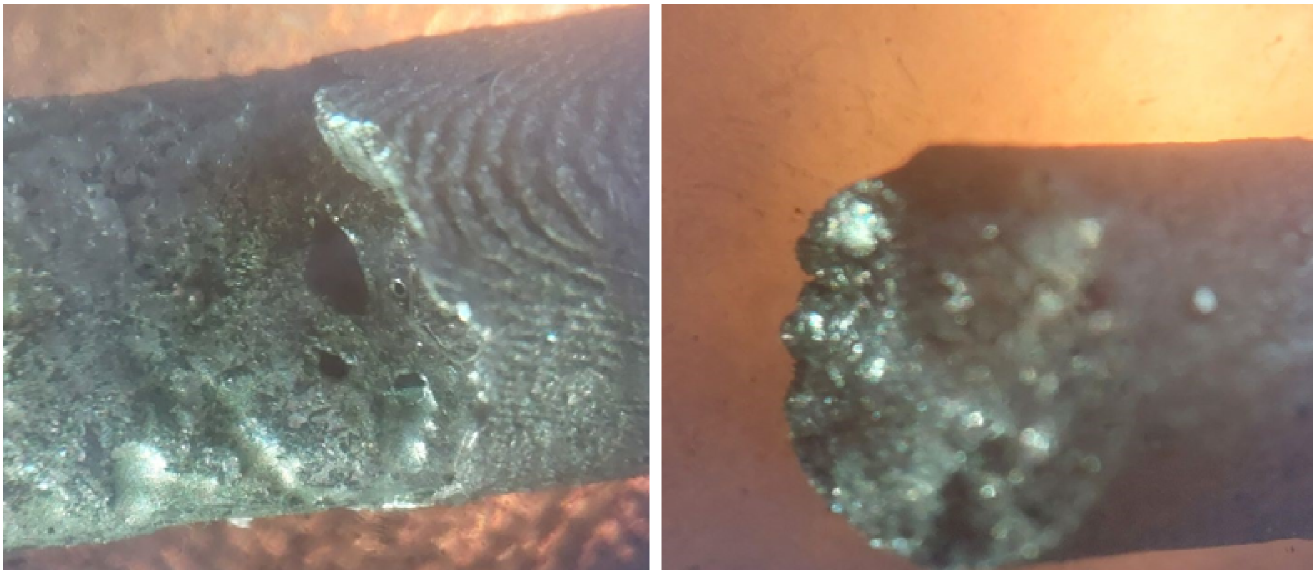

Fig. 1: The above pictures depicts the irregularities seen in the casting specimen (right side) when compared with DMLS fabricated specimen (left side)

However, this should be taken with some reserve, since the porosity of sintered structures depends on the purity of the input components (alloy powder) and sintering conditions (environment, temperature). The alloys without intermetallic phases and with minimum porosity have better mechanical properties.[11] A very precise, homogeneous alloy with good mechanical properties is obtained by laying one layer of the alloy powder over another, as confirmed by various authors (Ameer MA).[12]

Mechanical properties of the Co-Cr alloy obtained by applying the SLM technology, which are most commonly described, are the following: properties determined based on stress–strain diagram and the metal-ceramic bond strength. The main purpose of metal sintering is to obtain the metal with the highest possible density.[13] Metal density depends on the temperature of the thermally treated metal and the amount of energy required for melting metal powder on one side and scanning, laser power, and the thickness of the powder layer and the thermally treated region on the other. Moslehifard E et al.[14] demonstrated that sintered and thermally treated Co-Cr alloys show a significantly higher tensile strength and greater modulus of elasticity than cast

Co-Cr alloys.

Residual stress appears as a result of thermally treated individual layers of the melted metal powder. Quick heating is accompanied by quick cooling, which leads to metal expansion, followed by the shrinkage of the metal. This is most striking immediately after the removal of the alloy from the machine, and it is remedied by releasing the residual stress, i.e. by thermal treatment of the alloy. For the purpose of our research, thermal treatment (releasing residual stress) is conducted in the furnace, first at the temperature of 450°C (45 minutes) and then at the temperature of 750°C (60 minutes). After the expiration of the 60-minute period, the furnace is turned off, and the furnace door is opened at the temperature of 600°C, only to turn off the stream of protective gas (argon) at the temperature of 300°C. Sintered Co-Cr alloy shows higher hardness compared to the same cast alloy. Relevant data found in the literature indicate that the hardness of sintered dental Co-Cr alloys ranges 440–475 HV10, i.e. 382 HV10, whereas the hardness of the cast Co-Cr alloy ranges 325–374 HV10.[15]

Higher hardness and more homogeneous microstructure result in increased corrosion and wear resistance.[16] Subsequent thermal treating of the sintered alloy during the process of baking ceramics (in case of metal-ceramic restorations) does not affect its corrosion resistance.[17] Relevant data found in the literature indicate that the average surface roughness (the profile roughness parameter) immediately after sintering is about 8 μm.[18] After sand-blasting Al2 O3, the roughness is reduced due to surface homogenization and uniformization. The roughness of the sintered Co-Cr alloy surfaces is several times greater than the roughness of the cast alloy surfaces. This may cause a problem when making mobile restorations (e.g. removable partial denture framework). On the other hand, a rough surface increases the wettability and reduces the contact angle, which enhances the bond between the metal and the ceramics.[19]

5. Conclusion

Cobalt Chromium alloys (Co-Cr) had gained popularity because of their good mechanical properties and biocompatibility. Co-Cr alloys are commonly used base metal alloys for metal substructures fabricated through lost wax technique. To overcome the limitations of lost wax techniques newer technologies like Direct Metal Laser Sintering (DMLS) has been developed. This difference in fabrication method may influence the mechanical properties of the material. Among mechanical properties, fatigue behaviour and fracture toughness of Co-Cr alloys fabricated by casting and DMLS has not been evaluated in the literature. This study has been aimed to compare the fatigue behaviour and fracture toughness of Co-Cr alloy fabricated by casting and DMLS method.

For fatigue behaviour test dumbbell shaped samples with the dimensions of 54.8mm of total length in which length of jig holding area was 14mm with 6mm diameter on both sides and length of testing area in the centre was 26.8mm with 4.37mm diameter were fabricated by casting method and DMLS method. They were tested by using Nano universal testing machine and the results were analysed.

For fracture toughness test rectangle shaped samples with a V notch were fabricated by casting and DMLS method. The dimensions of samples with 55 mm length x 10 mm width x 2.5 mm height were maintained. They were tested by using Charpy's.

Impact test machine. The values obtained were analysed.

Results showed that when the values were compared in the fatigue behaviour test, the values obtained by DMLS group were higher than the casting group. In fracture toughness test, the values obtained by casting group were higher than the DMLS group.

6. Conflict of Interest

None.

7. Source of Funding

None.

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Author biography

Chembeti Harika, Post Graduate Student

K Mahendranadh Reddy, Professor and HOD

Venkat Adithya, Professor

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