

Laser Powder Bed Fusion of Co-Cr: A Material Comparison

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Abstract. Laser Powder Bed Fusion (LPBF) of cobalt-chrome (Co-Cr) alloys has been recognized for producing parts exhibiting the strength and being devoid of defects typically associated with cast Co-Cr alloys. Therefore, such parts are considered safe and comparable to cast Co-Cr alloys for intraoral use. Since additive manufacturing offers the advantage of creating complex geometries, the use of this thermo-physical process in a controlled manner allows for the homogenous production of the complex geometries typically required for dental products. This paper compares the mechanical properties claimed to be achievable through LPBF by the powder supplier against real world mechanical properties as used in dental applications. This comparison will serve as a baseline from which the mechanical properties achieved can be further enhanced to suit other possible applications of Co-Cr.

1 Introduction

Metallic dental devices produced using LPBF of Co-Cr alloys show adequate yield strength, tensile strength and elongation as compared to cast Co-Cr alloys in a study which was conducted by Viderščak, Dalibor. et al [1]. It was reported by Viderščak, Dalibor. et al [1] that the flaws typically found in cast Co-Cr alloys alloy were reduced in the samples that were processed using LPBF due to the homogeneity of the LPBF process. The mechanical properties of the Co-Cr samples processed using LPBF were found to be suitable for dental use according to the ISO 22674:2016 standard [2] and the tensile test data showed that the results achieved for a LPBF Co-Cr alloy are superior to that of a cast Co-Cr alloy Segbaya [3]. A study conducted by Denti [4] showed that the cast alloy exhibited superficial macro-porosity, which was attributed to uneven solidification of the molten metal.

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Furthermore, Denti [4] went on to investigate the effect of a stress relieving heat treatment on both the LPBF and the cast specimens, where it was found that the applied heat treatment increased the tensile strength of the LPBF specimens. However, it was noted that the LPBF specimens showed a drastic decrease in percentage elongation. The results for specimens as found by Denti [4], were produced using LPBF, and were found to be consistent with those exhibited in an earlier study conducted by Segbaya [3]. Kruth [5] found that over and above the attractive mechanical properties exhibited by Co-Cr alloys produced via LPBF, an attractive time saving was found across the entire value chain.

The aim of this study was to compare the material properties of a Co-Cr alloy as supplied by Electro Optical Systems GmbH (EOS) against the actual material properties obtained after manufacturing the same Co-Cr alloy at the Centre for Rapid Prototyping and Manufacturing (CRPM), in Bloemfontein, South Africa. This will serve a baseline for future work, where the development of heat treatments specific to particular applications of Co-Cr will be investigated.

2 Methodology and Results

2.1 Methodology

For this comparative study on Co-Cr used for dental applications, test specimens were prepared and heat treated to a heat treatment process as prescribed by EOS. Fifteen tensile specimens were manufactured in an EOSINT M280 Direct Metal Laser Sintering (DMLS) system at the CRPM, using default processing parameters as prescribed by EOS. To fully characterise the material, 5 specimens were analyzed in the as-built state, 5 specimens were submitted to the stress relieving heat treatment and 5 had both the stress relieving and solution annealing heat treatments, as prescribed by the material supplier, EOS in the material heat treatment guide [6]. Fig. 1 shows the stress relieving heat treatment protocol prescribed by EOS, while Fig. 2 shows the prescribed solution annealing protocol as prescribed by EOS in the material heat treatment guide [6]. The stress relieving protocol was carried out using a Nabertherm LH120/12 furnace, while the solution annealing was carried out using a Zubler Vario Press 300 EZR dental furnace. The tensile specimens were machined to ASTM E8 specifications after the respective heat treatments.

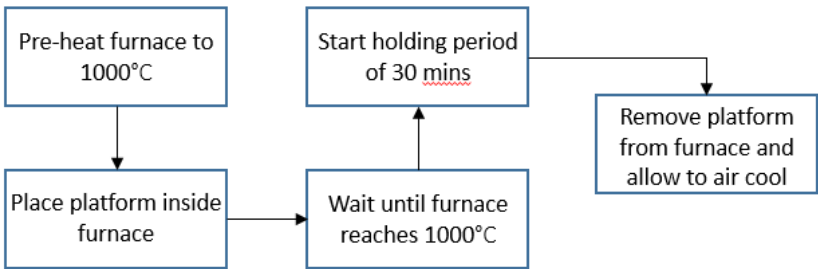


Fig. 1. Stress relieving heat treatment protocol

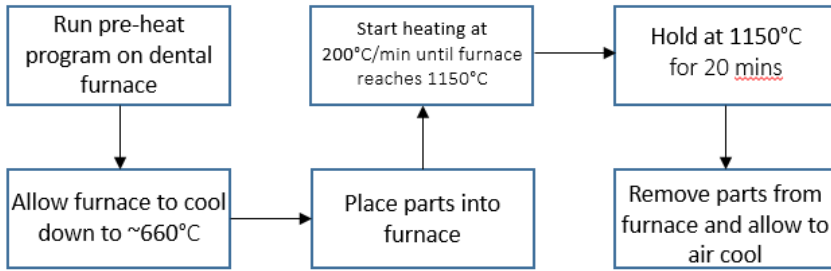


Fig. 2. Solution annealing protocol

To determine the characteristics of the material, optical microscopy was conducted on two different cross sections of the tensile specimens after the tensile testing was conducted. In order to verify the material strength specifications as listed by the material supplier, EOS, mechanical tensile tests were conducted on all 15 specimens. The cross-sections analysed were in the XY plane and perpendicular to the Z-axis of the EOSINT M280 machine. The specimens were mounted, polished and etched with a 32% hydrochloric acid solution, where after the optical microscopy was performed using an Axioskop A1 optical microscope. Micro-hardness tests were conducted on the specimens using a weight of 300g. Twelve hardness readings were taken for each specimen along two directions, i.e. in the XY plane and perpendicular to the Z-axis, respectively.

2.2 Results

The processing route of a material has a decisive effect on its microstructure, which has a direct effect on the mechanical properties of the given material, as described by Eckert, *et al.* [7]. Krakhmalev [8] found that for the LPBF process the temperature gradient due to the spatial distribution of the laser beam does not permit equilibrium to be maintained at the solid-liquid interface, resulting in the as-built Co-Cr microstructure being different from the conventionally processed Co-Cr microstructure. Fig. 3 shows an image of the as-built Co-Cr microstructure of the cross-section taken in the XY plane. In these micrographs the scan direction is clearly visible and is indicative of a typical DMLS process whereby the scan strategy is rotated between layers by 47 degrees as per the recommended processing parameters from the material supplier, EOS.

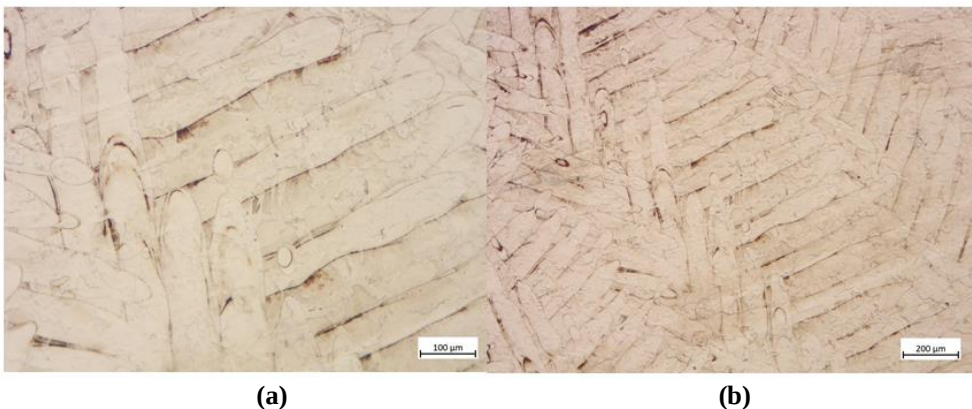


Fig. 3. Optical micrographs of cross-sections in the XY plane of an as-built Co-Cr specimen at a) 10x magnification and b) 20x magnification

Fig. 4 shows an image of the microstructure of the cross-section taken perpendicular to the X-axis (in the XY plane) of the as-built specimen. A clearly defined microstructure is presented, which shows layered tracks that are typical of the DMLS process. From the observed microstructure it is evident that the metal powder fused thoroughly and an almost parabolic edge characterized the solidification pattern of each track of the molten powder. This confirmed that each layer solidified on the underlying layer, with the laser tracks overlapping each other, thereby limiting any porosity. However, the high-temperature gradient during the LPBF process due to the localized thermal input from the Gaussian laser beam, induced thermal stress in the as-built parts, as found in a study by Mugwagwa *et.al.* [9]. As a result, subsequent heat treatment was required to relieve the material of the residual stress and to improve the mechanical properties of the specimen.



Fig. 4. Optical micrograph of a cross section of an as-built specimen perpendicular to the Z-axis (in the XY plane) at 10x magnification

Fig. 5 shows the microstructure of a stress-relieved specimen in which the track boundaries shown in Fig. 4 were no longer visible and a more homogenous microstructure had formed. The microstructure exhibits well defined grain boundaries, which resulted from the slow cooling rate as compared to the rapid cooling rate experienced during the building process. Erfanian-Nazif-Toosi *et.al.* [10] found that the relatively slower cooling rate experienced during the stress relieving process allowed the Co-Cr microstructure to attain equilibrium. This resulted in the grain boundaries of the stress-relieved samples being more defined than the as-built specimens for both cross section orientations.

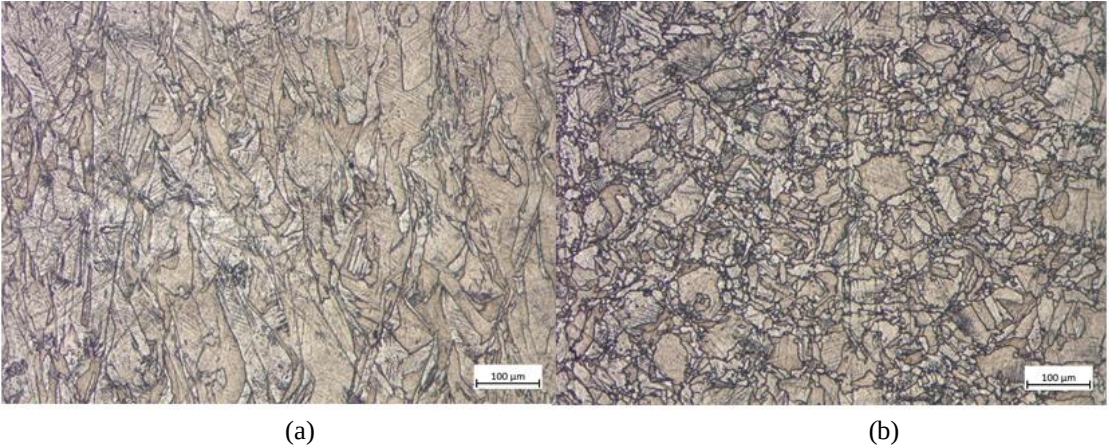


Fig. 5. Optical micrographs of cross sections of a stress-relieved Co-Cr specimen a) in the XY plane and b) perpendicular to the Z-axis, both at 10x magnification

Fig. 6 shows the microstructure of a stress relieved and solution annealed specimen. Here the track boundaries seen in Figure 4 are no longer visible and a more homogenous microstructure had formed. The microstructure also exhibits well-defined grain boundaries.

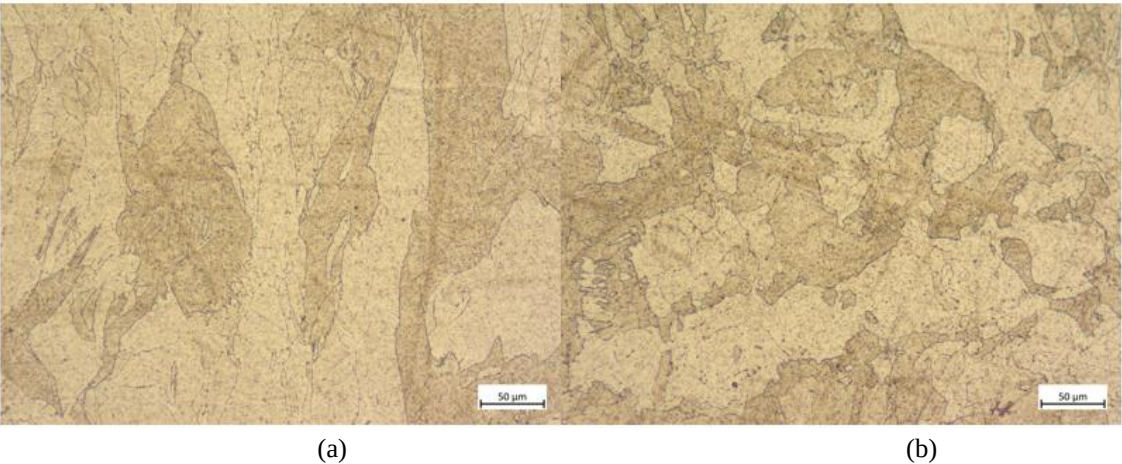


Fig. 6. Cross sections of stress-relieved and solution annealed specimens a) perpendicular to the Z-axis (in the XY plane) and b) parallel to the Z-axis at 20x magnification

Table1, captures the results from the micro-hardness tests, which were conducted on the as-built specimens, stress relieved specimens and stress relieved and annealed specimens, on cross sections perpendicular to the Z-axis.

Table 1. Micro hardness test results for the plane perpendicular to the Z-axis

MICRO-HARDNESS: VICKERS (HV)			
L to z Direction			
	As built	Stress Relieved	Stress Relieved and Solution Annealed
1	378	414	355
2	384	385	367
3	388	524	376
4	391	450	372
5	420	521	365
6	398	492	338
7	416	489	402
8	454	531	372
9	436	596	409
10	397	529	367
11	408	526	354
12	443	519	368
Average	409	498	371
STD Deviation	24	55	19

Table 2 captures the results from the micro-hardness test which was conducted on the as-built specimens, stress-relieved specimens and stress-relieved and annealed specimens, parallel to the Z-axis.

Table 2. Micro hardness test results for the plane parallel to the Z-axis

MICRO-HARDNESS: VICKERS (HV)			
l to z Direction			
	As-built	Stress Relieved	Stress Relieved and Solution Annealed
1	364	510	374
2	382	489	389
3	384	413	380
4	355	372	384
5	348	491	360
6	319	458	372
7	387	555	378
8	375	425	350
9	380	433	416
10	359	535	352
11	347	443	409
12	362	481	376
Average	364	467	378
STD Deviation	19	51	19

From the obtained results, it is evident that the values recorded for the stress relieving heat treated Specimens indicate that the stress relieving heat treatment had caused the material to “harden”, as an average HV value of 498HV was recorded for the XY plane and 467HV was recorded for the plane perpendicular to the Z-axis. In the as-built state, an average HV value of 409HV was recorded for the XY plane and 364HV was recorded for the plane parallel to the Z-axis. For the stress relieved and solution annealed specimens, 378HV was recorded for the plane parallel to the Z-axis. It can be deduced that the as-built specimens displayed a degree of anisotropy, since these specimens displayed different micro-hardness values when measured along axes having different directions. It was also found that the application of heat treatments decreased the anisotropy, which is consistent with a study conducted by Sigh *et.al* [11].

Table 3 summarizes the tensile test results achieved, where a 14% increase was found for the ultimate tensile strength (UTS) when comparing the as-built and the stress relieved specimens. The specimens having undergone both stress relieving and solution annealing had UTS values that were 9% higher as compared to the as-built specimens, while the % elongation decreased significantly from 7% to 4% after application of the stress relieving heat treatment. The percentage elongation increase from 4% to 6% after application of the solution annealing heat treatment indicates that some degree of ductility had been restored to the specimens after being subjected to the solution annealing process. The prescribed stress relieving plus solution annealing improves the ductility of the material which renders the material more ideal for dental applications [2].

Table 3. EOS mechanical properties vs mechanical properties determined in the current study.

	EOS Mechanical Properties		Determined Mechanical Properties	
	UTS (MPa)	% elongation	UTS (MPa)	% elongation
As-built	1050 +/- 100MPa	10 +/- 2%	1003	7
Stress Relieved	1200 +/- 100MPa	6 +/- 2%	1162	4
Stress Relieved + Solution Annealed	1100 +/- 100MPa	7 +/- 2%	1093	6

3 Conclusion

The results obtained during the material comparison study indicate that the material properties displayed by the specimens which were produced at the CRPM compared favourably with the material properties as supplied by EOS. From the experimental data achieved, it is seen that for all three conditions, i.e. as-built, stress relieved and stress relieved with solution annealing, the achieved UTS is well within the tolerance as specified by EOS. In terms of percentage elongation, the as-built specimens were slightly below the tolerance as specified, however for the intended dental application, this is negligible as the specimens would undergo stress relieving and solution annealing. Both the stress relieved, and stress relieved with solution annealed specimens exhibited a percentage elongation which was within the specified tolerance range. From the obtained results it can be concluded that the dental components produced at the CRPM conform to the specifications set out in the

ISO22674:2016 standard. Homogenous microstructures were obtained for the specimens subjected to stress relieving and annealing heat treatments, which is indicative of a homogenous solidification of the melt pool during the building process.

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