Mechanical and microstructural evaluation of CoCr alloys manufactured via selective laser melting (SLM)

M. V. Mergulhão[1], C. E. Podestá[1,2], M. D. M. Neves[1]

[1]Nuclear and Energy Research Institute, Materials Science and Technology Center, SP, Brazil
[2]High Bond Industry of Metal Alloys, Import and Exports LTD, Indaiatuba-SP, Brazil
e-mail: marcellovertamatti@usp.br

Abstract: Dental prosthesis components are widely produced with CoCr alloys. New near net shape techniques has been used nowadays as an alternative route. The aim of this study was evaluated the mechanical properties and the microstructural characterization of CoCr dental alloys manufactured by SLM and to investigated the correlation of chemical composition between mechanical properties of standard specimens. The tensile specimens was manufactured by SLM technique in standard dimensions by two different composition of CoCr dental alloy and were chemical analyzed using scanning electron microscope with energy-dispersed X-ray spectroscopy (SEM-EDS) and X-ray fluorescence. The mechanical properties as uniaxial tensile (yield strength, maximum tensile, rupture tensile, elongation and elastic modulus) and Vickers hardness were evaluated. The microstructure of the samples were characterized using optical microscope (OM) and SEM-EDS. The mechanical results indicate higher values for both composition of CoCr alloys fabricated via SLM technique. The micrographs revealed a characteristic morphology of layer used in the SLM technique.
Keywords: CoCr alloys, selective laser melting, biomaterial, dental prothesis.

Introduction

The development of AM technologies, according to Van Noort, (2012), provided the manufacture of customized implants. The production of metal components by means of high power laser, known as Selective Laser casting (SLM - Selective Laser Sintering), was promising since it allows the manufacture of customized components and high final physical properties compared to conventional techniques. At the end of the 80s emerged results from early research seeking a way to produce a physical model from a three-dimensional digital model. Rapid Prototyping (RP) is the term used to represent the great variety of technologies using the method to produce complex geometries from a three-dimensional (3D) model, obtained by Computer Aided Design (CAD) using: CT or MRI scan with the use of a Computer Aided Manufacturing (CAM) (GIBSON, 2005; GOUVEIA, 2009; VAN NOORT, 2012). Powder of Co-Cr alloys (*Stellite*) have their use widespread in the automotive, aviation and aerospace industry, because these alloys have excellent resistance to corrosion and wear. The high biocompatibility of these alloys has made this attractive material for the manufacture of components of medical and dental sectors (ASM INTERNACIONAL, 2000; CRAIG, 2012). In literature are reported several studies to consolidate the powder of Co-Cr alloy. The mechanical, physical and electrochemical properties of Co-Cr-Mo alloy for dental implants were evaluated for better determination of the production parameters on sintering with the improvement aimed at restoration and medical implants (FAZIRA et al., 2013;

HENRIQUES et al., 2012; ÖRTORP et al., 2011). According Jabbari *et al.,* (2014), Co-Cr alloys are used almost exclusively in the manufacture of metal structures prosthesis and recently is replacing Ni-Cr alloy, or alternatively for the production of restorations in porcelain fused to metal (PFM), due to the Co-Cr alloy is Ni-free does not have allergic responses or toxic effects related to Nickel. The aim of this study is to demonstrate the mechanical properties and microstructures of specimens manufactured by SLM technology, using different Co-Cr alloys. Yet there is an important knowledge of performance properties, dimensional, mechanical and microstructural of this process as reported recently works (MERGULHÃO et al., 2015a, 2015b; PODESTÁ et al., 2015).

Experimental Procedure

The Co-Cr alloys gas atomized was provided in accordance with SLM process granulometric range (20-50 μm). The confirmation of the chemical composition by fluorescence spectrometer X-ray energy dispersive (Shimadzu EDX-720 equipment) of powders is presented in Table 1.

Table 1 – Chemical composition (weight %) of Co-Cr-Mo powder.

	Alloys		
Elements	ASTM F75	EOS	Remanium Star
Co	63,86 ± 0,07	62,00 ± 1,00	64,60 ± 0,20
Cr	28,96 ± 0,04	25,00 ± 1,00	26,70 ± 0,20
Mo	7,02 ± 0,01	7,00 ± 1,00	-
W	-	6,00 ± 1,00	8,00 ± 0,05
Nb	-	< 0,20	0,27 ± 0,01
V	-	< 0,20	0,19 ± 0,01
Fe	0,16 ± 0,01	0,20 ± 0,10	0,10 ± 0,01
S	-	-	0,19 ± 0,01
Si	-	-	0,18 ± 0,01

The consolidation of SLM tensile samples was carried out using a selective laser melting machine: ASTM F75 powder with a SLM®280HL machine; EOS CobaltChrome SP2® powder with a EOSINT M270 machine; Remanium® Star (by Dentaurum) powder with a Mlab cusing machine (Concept Laser®). The mechanical tests were performed using a universal testing machine (Instron 3366) under a crosshead speed of 0,2 mm/min at room temperature, following the standard recommendations (ASTM E8/E8M(2015) and ISO22674(2006)).

The microstructural characterization and fracture analysis of CoCr samples was performed after tensile test. The tensile test specimens was sectioned and was prepared as followed: mechanical grinding of abrasive SiC paper to the grain size of #1200, and final polishing OP-S 0,02μm with ChermoMet cloth. The specimens were etching in solution: 100ml HCl and 2ml H_2O_2 (1-2 minutes at room temperature). The microstructural characterization was performed using an optical microscope (Olympus - BX51M) and scanning electron microscope (SEM-EDS Philips XL30).

To evaluate the internal porosity of cast and SLM sample were measured the pycnometer density in comparison by the theoretical density. The density by pycnometer Helium, considered only the internal porosity (excluding the open porosity) was measure using the Micromeritics equipment - Model Accu PYC 1330 Pycnometer, located in the Nuclear Fuel Center/IPEN.

Results and Discussion

Mechanical data results and the verification with minimum mechanical properties required by the standards are presented in Table 2 and stress curves Figure 1. According Takaichi *et al.*, (2013), the mechanical properties of SLM samples have values similar to those obtained in tensile tests, respectively: 516 MPa for the yield strength, 912 MPa for maximum tension and 10,7% elongation to consolidated by SLM. The data obtained for the modulus of elasticity for both techniques accord with the minimum required by standard ISO22674(2006). However, the mechanical properties obtained for the consolidation of the alloy CoCr alloys by SLM technique fits the type 5 as manufactured.

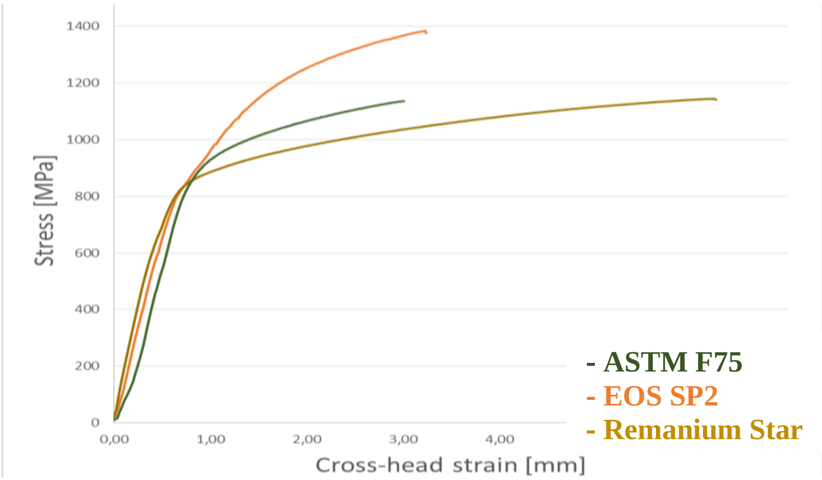

Figure 1: Tensile curves of stress-strain SLM CoCr alloys.

Table 2 – Comparison of the results (mean and standard deviation) of mechanical properties in the SLM samples of CoCr alloys compared with the mechanical properties required according to the standards ISO 22674(2006).

Reference	*Type*	*0.2% YS [MPa]*	*Elongation [%]*	*E [GPa]*	*UTS [MPa]*	*Hardness [HV0,3N/10s]*
ASTM F75	SLM	731,50 ± 40,31	13,73 ± 5,32	276,70 ± 12,63	1136,95 ± 0,92	420,62 ± 21,16
EOS SP2	SLM	788,40 ± 158,12	7,68 ± 0,80	265,58 ±7,24	1327,39 ± 63,40	509,91 ± 20,54
Remanium Star	SLM	578,00 ± 74,03	24,00 ± 4,46	283,36 ± 6,09	1125,37 ± 21,58	502,46 ± 8,90
ISO 22674:15	1	80	18	-	-	
	2	180	10			
	3	270	5			
	4	360	2			
	5	500	2	150		

In Figure 2 can be see the SLM sample examined by OM and SEM. As in OM images (Figure 2a), it is possible to observe the ellipsoid geometry structure of incidence of the laser beam, highlighted by the arrows on the images. This typical laser beam configuration is described by several authors (GIRARDIN et al., 2016; MERGULHÃO et al., 2015a, 2015b; PODESTÁ et al., 2015). It is possible to observe the homogeneous microstructure and absence of second phases in the microstructure. The SLM technique provides formation of ultrafine grains within ellipsoid geometry formed by the fusion laser beam action (Figure 2b-2d).

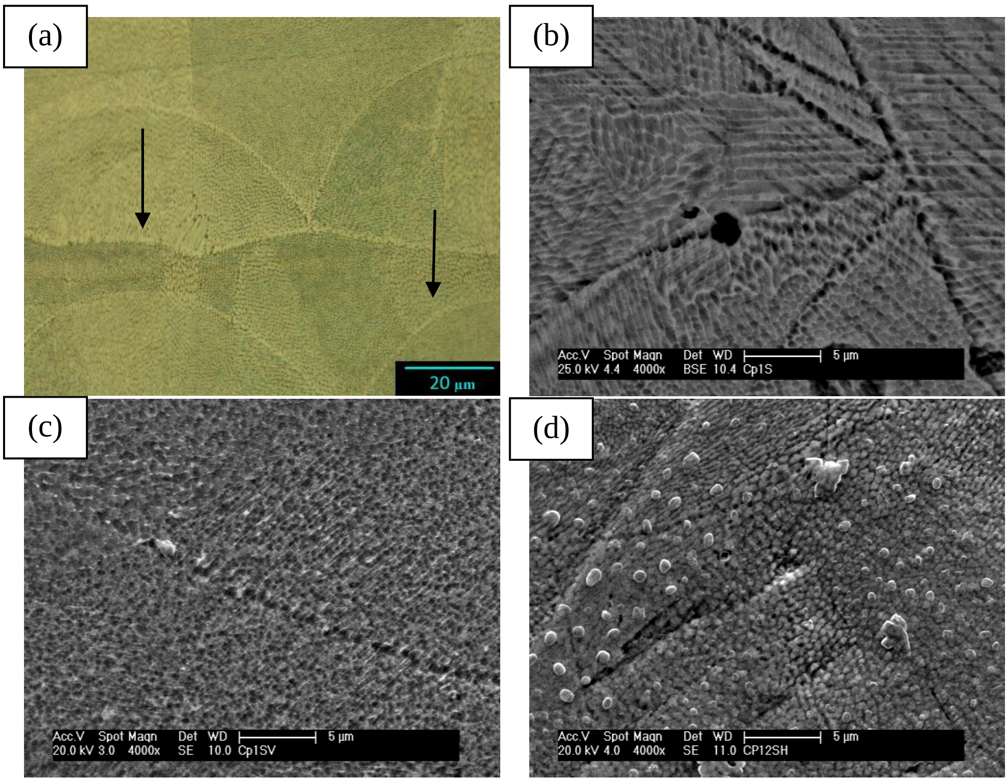

Figure 2: Images of microstructural analysis after etch CoCr alloys. (a) Characteristically OM micrograph SLM specimen (Remanium star alloy); (b) to (d) SEM micrographs of specimens respectively for alloy: ASTM F75, EOS SP2 and Remanium star.

The fractures of the tensile samples were SEM analyzed. In the fracture imaging is showed a homogeneous formation fracture surface of SLM specimens (Figure 3). The formation of "dimples" is homogeneously distributed in the microstructure of the samples, representing a more ductless and higher toughness. The formation of finer dimples is characteristic of a ductile fracture and which features mechanical properties and higher toughness, and it can be see on the SLM sample according to the results shown (Table 2). The type of fracture observed in the samples, according to Takaichi *et al.* (2013), describes the formation of dimples along the fracture surface, as well as cracking of the wedge, was appointed as a possible formation of cleavage fracture over favorable crystallographic planes.

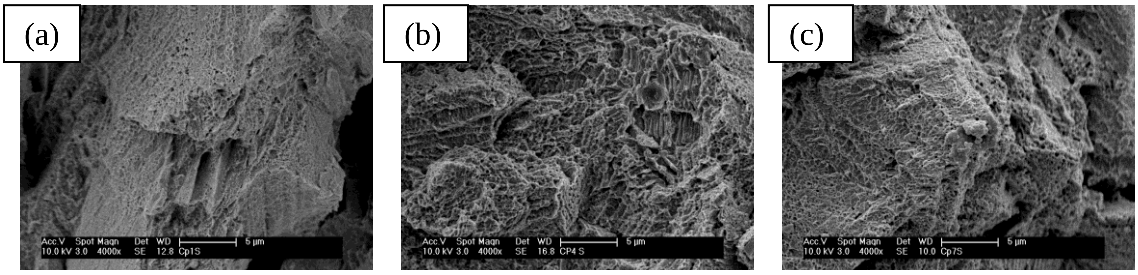

Figure 3: SEM images of tensile fractures. SLM alloys: (a) ASTM F75; (b) EOS SP2; (c) Remanium star.

Conclusions

The SLM technique allowed obtaining samples with great mechanical properties satisfying the standard ISO22674. The microstructural characterization of selective laser melting allowed to obtain samples with improved chemical homogeneity over the molten sample, as discussed in the microstructural characterization of the samples and confirmed by fracture analysis. The processing using laser sintering proved superior to casting processing technique, allowing the use of this technique in the manufacturing area of prosthetics and dental implants.

Acknowledgments

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