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Mechanical Properties and Microstructural Characterization of Cobalt-Chromium (CoCr) Obtained by Casting and Selective Laser Melting (SLM)

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Abstract. The aim of this study is the consolidation of Cobalt-Chromium (CoCr) alloy powder using the additive manufacturing - selective laser melting (SLM) and the investment casting techniques. The research of this study has been applied to their biomaterial applied to development of prosthesis and dental implants. The gas atomized powder are spherical (mean diameter equal to 42,74 μm) and was analyzed by their physical and chemical properties. The microstructure of the powder and specimens was evaluated using optical microscope (OM) and scanning electron microscope with energy-dispersed X-ray spectroscopy (SEM-EDS). The mechanical properties were evaluated of standard samples using a tensile (yield strength, maximum tensile, rupture tensile and elongation), three point bending (transverse rupture strength) and micro hardness tests. The mechanical results indicate higher values for the SLM than casting specimens. The micrographs revealed a characteristic morphology of laser been used in the SLM technique and the dendrites in the casting technique. The microstructure of samples made by SLM is thinner than the samples obtained in the cast.

Introduction

Metal powders of cobalt-chromium alloys (Co-Cr) have been used in many sectors of the automotive, aeronautics and aerospace industry, because the high wear resistance and adequate corrosion resistance also are used in surface coating to increase performance in components. Because of its biocompatibility and mechanical properties this material has been also used in the medical and dental components [1]. Advances have occurred in the manufacture processes using powder metallurgy (PM) techniques notably in the health care, making this technology competitive over traditional manufacturing processes [2,3].

Currently, another form of consolidation in development is the Rapid Prototyping (RP) that can be manufactured complex shapes with high difficulty to be obtained by other processes with removal material like in milling [4].

Manufacturing components in the medical/dental area by PM techniques using the RP, as the selective laser melting (SLM), also known in the ceramic and plastic areas by selective laser sintering (SLS), still requires knowledge of this technology employ the Co-Cr alloy in the form of particulate material. Therefore, are still of great importance the knowledge to evaluated the dimensional, mechanical and microstructural properties of laser melting technology. Thus, obtained properties must meet specific characteristics in health performance. The aim of this study is analyze the mechanical properties and microstructural of standard specimens manufactured by SLM and casting.

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Materials and Methods

The gas atomized powder Co-Cr alloy (EOS CobaltChrome SP2) was supplied by HigBond®. The chemical composition of powder alloy was evaluated by X-ray fluorescence (see Table).

Table 1 – Chemical composition of gas atomized powder determined by X-ray fluorescence [wt%].

Alloy	Content of elements [%]						
	Co	**Cr**	**Mo**	**W**	**V**	**Nb**	**Fe**
Powder	62,0 ± 1,0	25,0 ± 1,0	7,0 ± 1,0	6,0 ± 1,0	< 0,2	< 0,2	0,2 ± 0,1

The physical properties of the CoCr powder were obtained such as bulk density, tapped density and flowability by funnel Hall [5,6,7]. The particle size distribution was determined using laser particle size equipment (Cilas granulometer - model 1064). The particle shape and the morphology of the particles were evaluated by scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDS Philips XL30).

The mechanical properties were evaluated by standard specimens consolidated by investment casting (lost wax casting) and selective laser melting (SLM). The casting samples were melting at 1380°C, according to ASTM F75. The standard samples were finished by sandblasting with oxide aluminum. The SLM specimens were performed in a melting machine EOSINT M270 under nitrogen gas atmosphere. The samples consolidated by both processes have not heat treatment. The specimens were manufactured in standardized dimensions for tensile tests (according with ISO22674:06) [8] and three point bending tests (according ASTM B528:12) [9].

The mechanical tests were carried out on universal testing machine Instron 4400R. The crosshead speed was applied at a rate of 2 mm/min at room temperature. The hardness evaluation was realized after mechanical tests using a microdurometer Microfischer HM2000.

The microstructural characterization of CoCr specimens were realized using optical microscope (Olympus - BX51M) and scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDS Philips XL30 and TableTop 3000). The samples were mechanically grinded, polished and thereafter chemically etched in a solution HCl, H_2SO_4 and HNO_3 for 60 to 240 seconds at the temperature of 45 °C

Results and Discussion

The characteristic shape of the obtained powder by gas atomization is seen in Fig. 1a-1d. It should be mentioned that the particle shape influences the packing property, flow rate and compressibility, as well as reports on the powder metallurgy process [2,10,11]. In Fig. 1 was still observed the presence of satellites in the surface particles. The satellites are formed by collisions between the particles during the completion of the atomization process, and the incidence of the effect increases in regard to reduction of the particle size [11]. Through the images in Fig. 1c is possible to check the presence of bright and dark regions of the particles. According to the reference [2], when reactive elements are connecting with the basic elements during the gas atomization is possible to occur the oxidation on the surface of the particles. The analysis carried out by EDS indicated the possible some particles in the powder surface oxidation as shown in the spectrum in Fig. 1e and 1f.

The powders for SLM manufacture technique have a mean diameter between 26 to 65 microns to improve the physical properties like as flow time, apparent density and tap density. After evaluating the physical properties of the powder according to standards, the results obtained are shown in Table 2.

The flowability of the powder is an important feature for the SLM consolidation process. The obtained value allows proper placement of particles and is performed efficiently, to eliminate voids and porosity in the final consolidated.

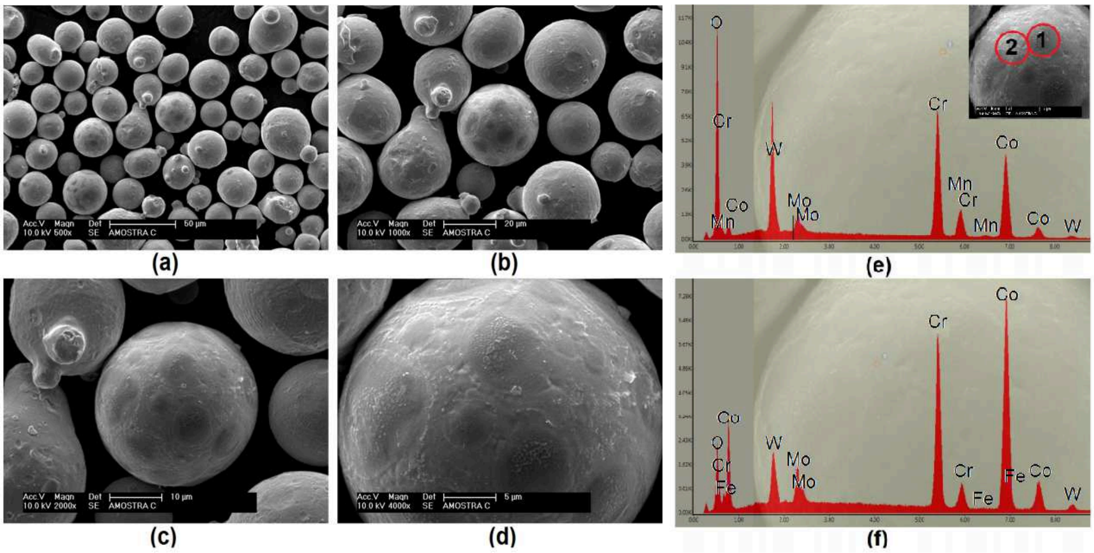


Fig. 1 – a) to d) Micrographs of atomized powder Co-Cr powder, e) EDS spectroscopy of powder – spot 1 and f) EDS spectroscopy of powder – spot 2.

Table 2 – Physical properties of CoCr powder.

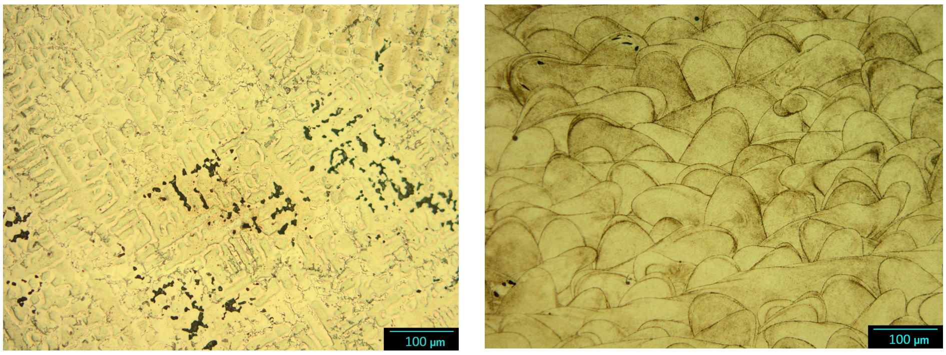
Properties		**Powder**	**Standard**
Granulometric Distribution [µm]	**diameter of 10%**	26,10	
	diameter of 50%	39,52	
	diameter of 90%	64,05	
	medium diameter	42,74	
Flow Time [s/50g]		20,59	**MPIF 03**
Apparent Density [g/cm³]		4,63	**MPIF 04**
Tap Density [g/cm³]		5,17	**MPIF 46**
Relative Density [g/cm³]		8,71	
Specific Surface Area [m²/g]	**One Point**	1,799	
	3 Points	2,644	

The results of the mechanical tests for the specimens consolidated by casting and selective laser melting are present at Table 3. Analyzing the values is possible to verify that in all properties the SLM technique are better than casting technique. In according to standard ISO22674:06 both technique satisfied the type 5 criteria. The minimum values for the standard at the type 5 are 500MPa for Rp 0,2%, 2% for elongation and 150 GPa for elastic modulus. The hardness data appointed for SLM samples were higher values than those of cast samples.

Table 3 – Mechanical properties of casting and SLM CoCr specimens. (*NU = not usual)

Mechanical Properties		**Consolidation technique**		**Standard**
		Cast	**SLM**	
Rp 0,2%	[MPa]	545,67 ± 14,57	788,4 ± 158,12	**ISO 22674: 06**
Rupture Stress	[MPa]	820,48 ± 38,54	1312,4 ± 67,67	
Max. Stress	[MPa]	845,98 ± 35,19	1327,4 ± 63,40	
Elongation	[%]	7,29 ± 1,16	7,68 ± 0,80	
Transverse Rupture Stress	[MPa]	NU*	1790,0 ± 91,94	**ASTM B528:12**
Micro Hardness	[HV]	381,50 ± 12,63	551,55 ± 20,03	**ISO 14577-1**

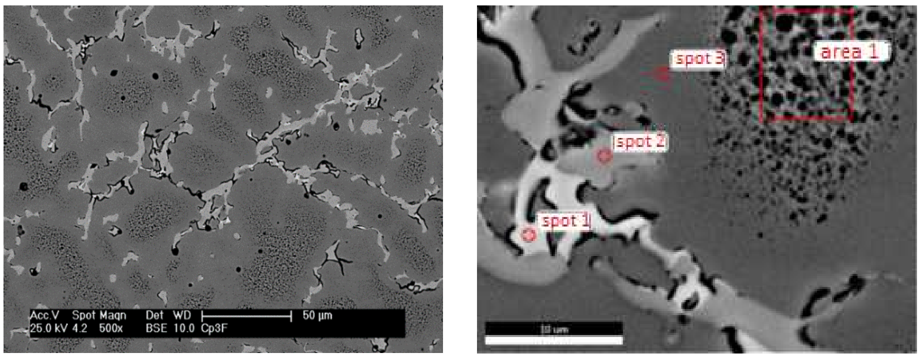
To understand the improvement in the mechanical properties in the SLM specimens in relation of the casting process technique microstructural characterizations were performed, as show in Fig. 2. Is possible to verify the microstructure characteristic in the casting specimens have a dendritic morphology and in the SLM specimens have a weld-like structure. The configuration as-weld indicates the direction of the laser melting scanning (Fig.2 b). As it is possible to check the presence of porous in the both specimens, however at the casting technique the presence of porous is more notable than the laser melting. In this technique the porous are smaller and in small quantity.



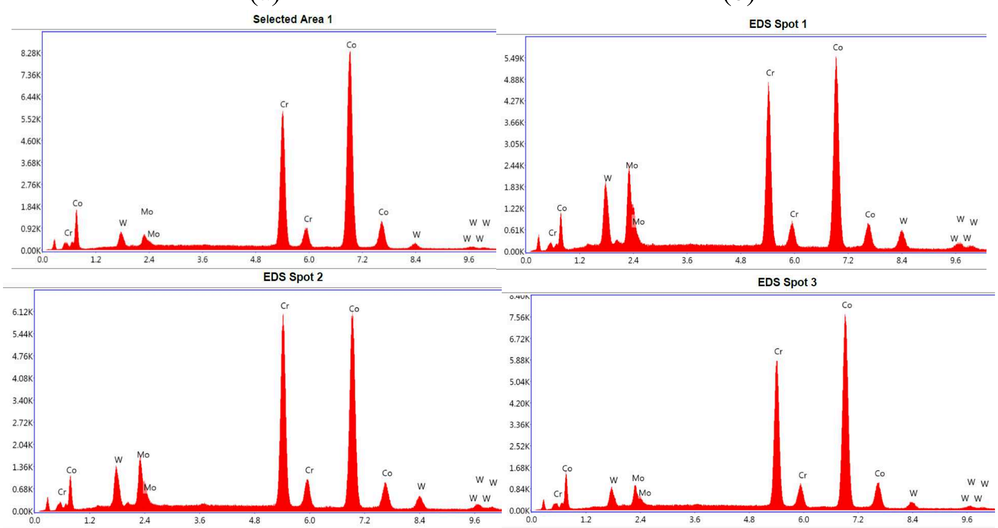
(a) (b)

Fig. 1 – OM images of CoCr specimens after chemical etch, a) as-cast, b) as SLM specimens.

The analyses using SEM was performed to evaluated the phases contained in the microstructure. In the Fig. 2 is possible analysis the cast specimen with a second phase (white areas), however in the other magnifications are possible verify that has a different areas in the second phase. The semi-quantitative analysis with the EDS (Fig. 2c) and the respectively spectrums, show that are two different compositions in the white area of the microstructure. The spot 1 present a rich area containing Mo and W, and the spot 2 present an area containing more Cr and Co with a small percentage of Mo and W. At the other areas (spot 3 and area 1) is possible verify the matrix, formed by CoCr, but in the area 1 has a presence of microporosity.



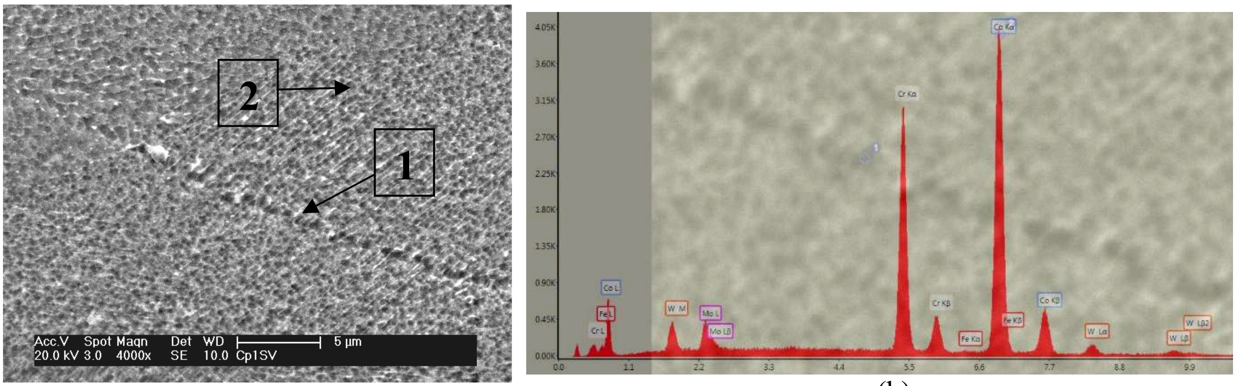
(a) (b)



(c)

Fig. 2 – a) SEM images of as-cast specimen after chemical etch, b) EDS spots and area selected for semi-qualitative analyses and c) spectrum of EDS analyses.

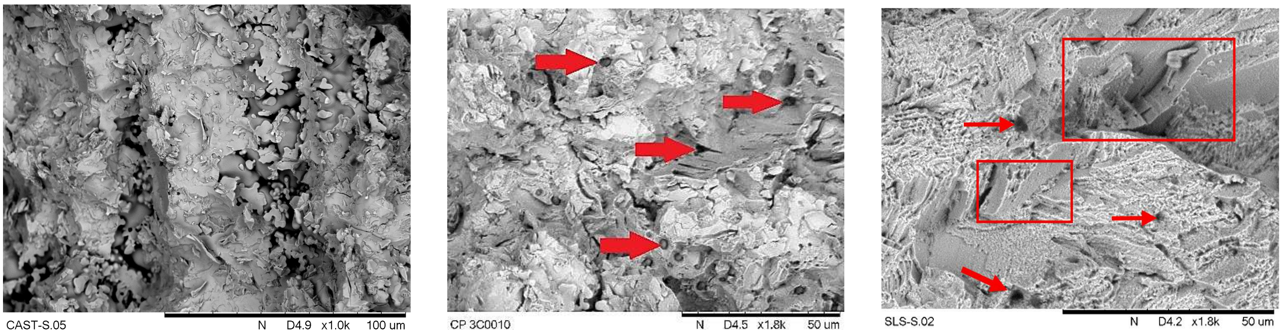
The SLM specimen was analyzed by SEM-EDS, like as show in Fig. 3. The images presented a microstructure formed with small grains characterizing the rapid solidification during the manufactured of the SLM specimen. The semi quantitative analysis in the different areas (matrix area is homogeneous and the boundary of laser melting) show that has not different elements compositions. The SLM specimen presents a homogeneous matrix with fine grains. This microstructure characteristic of laser melting technique allows achieve better mechanical properties than the cast technique.



(a) (b)

Fig. 3 – a) SEM image of SLM specimen, b) EDS spots for qualitative analyses and c) table of quantitative results of EDS analyses.

The casting and SLM standard samples after mechanical tests were analyses in the fracture area by SEM (Fig. 4). Analyzing the fractures it is possible verify the formation of dendrite microstructure (characteristic of the casting process) and the porosity (red arrows) distributed in the molten sample (Fig. 4a). The presence of porosity (red arrows) on fracture, consequently influenced in decreasing the rupture resistance of the material. However, even in this condition it is possible to verify, in the SLM sample (Fig. 5b) the presence of dimples, which represent a more ductile region. This formation can be verified homogeneous and distributed form in the microstructure of the SLM than cast sample.



(a) (b)

Fig. 4 – SEM images of fractured standard samples after mechanical test, a) fracture of casting sample and b) fracture of SLM sample.

In selective laser melting process is observed small amount of pores (red arrows), which consequently influence of positive way to increase the tension rupture. However, it is possible to observe (red rectangle) a quasi-cleavage fracture that shows characteristics of both cleavage and plastic deformation [12]. Is important to describes that quasi-cleavage does not configure and describe a fracture, the term quasi-cleavage can be used for describe a distinct fracture and not represent a separate fracture mode [12]. This unique appearance fracture is observed in SLM CoCrMo samples [13,14] and in CoCrW [15].

Conclusions

The mechanical properties as yield stress, rupture stress, maximum stress, elongation and hardness in the SLM technique are better than casting technique.
The microstructures in the samples represent the characteristics phases in the manufacturing processes. The casting specimens are characterized by the dendritic morphology and the SLM specimens are characterized with a very fine grains and great homogeneity of the microstructure by the rapid melt and localized solidification of the laser melting process.
The analysis by OM and SEM show that the microstructure presents more pores in the casting technique than the SLM technique.
The SLM fracture represents a ductile fracture, showing the presence of dimples. This is one of the evidences in the low values at the results of uniaxial tensile tests in the casting samples.

References

[1]T. Narushima, et al.: JOM Vol. 4 (2013), p.65.
[2] A.J. Yule, J. Dunkley: *Atomization of Melts*. (Claredon Press Oxford, 1994).
[3] N. Moro, A.P. Auras, Metalurgia do pó e o futuro da indústria. www.norbertocefetsc.pro.br, 2007. Acesso em: 21 Fevereiro 2014.
[4] M.F. Oliveira: Aplicações da Prototipagem Rápida em Projetos de Pesquisa. [S.l.]: Unicamp, 2008. .
[5] Metals Powder Industries Federation. Standard methods for determination of apparent density of free-flowing metal powders using the Hall apparatus. Pinceton: MPIF, 1985. (MPIF Standard 04).
[6] Metals Powder Industries Federation. Standard methods for determination of flow rate of free-flowing metal powders using the hall apparatus. Princeton: MPIF, 1988. (MPIF Standard 03).
[7] Metals Powder Industries Federation. Standard methods for determination tap density of metal powders. Princeton: MPIF, 1985. (MPIF Standard 46).
[8] International Organization For Standardization. Dentistry — Metallic materials for fixed and removable restorations and appliances. Geneva: [s.n.], 2006. (ISO 22674:2006(E)).
[9]American Society For Testing Materials. (ASTM B528-12). Standard Test Method for Transverse Rupture Strength of Powder Metallurgy (PM) Specimens. West Conshohocken: 2012.
[10] R.M. German: *Powder Metallurgy Science*. (Metal Powder Industries Federation 2. ed. 1994).
[11] ASM Metals Handbook. Powder Metal Tecnologies and Applications. Vol. 7 1999.
[12] ASM Metals Handbook. Fractografy. Vol. 12 1992.
[13] A. Gatto, S. Bortolini, L. Iuliano, Characterisation of Selective Laser Sintered Implant Alloys: Ti6Al4V and Co-Cr-Mo. 20th CIRP Design Conference, Nantes (FRA), 19-21 April 2010. 729-736.
[14] A. Takaichi, et al.:Journal of the mechanical behavior of biomedical materials Vol. 21 (4) (2013), p. 67.
[15] Y. Lu, et al.:Materials Science and Engineering C Vol. 49 (8) (2015), p. 517.
[16] International Organization For Standardization. Metallic materials - Instrumented indentation test for hardness N. Moro, A. P. Auras, Metalurgia do pó e o futuro da indústria. www.norbertocefetsc.pro.br, 2007. and materials parameters - Part 1: Test method. Geneva: 2002. (ISO 14577-1).
[17] American Spciety For Testing Materials ASTM. Standard Specification for Cobalt-28 Chromium-6 Molybdenum Alloy Castings and Casting Alloy for Surgical Implants. West Conshohocken: Copyright © ASTM International, 2012. (ASTM F75-12).