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Valuation of Mechanical Properties and Microstructural Characterization of ASTM F75 Co-Cr Alloy Obtained by Selective Laser Melting (SLM) and Casting Techniques

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Abstract. Advances in processes using the powder metallurgy techniques are making this technology competitive compared to the other traditional manufacturing processes, especially in medicine area. The additive rapid prototyping technique – selective laser melting (SLM) was applied in a biomaterial of CoCrMoFe alloy (ASTM F75), to study the mechanical properties and microstructural characterization in comparison between the conventional technique – casting. The gas atomized powder was investigated by their physical (as apparent density, bulk density and flow rate) and the chemical properties. The powder was analyzed using scanning electron microscope with energy-dispersed X-ray spectroscopy (SEM-EDS) and X-ray fluorescence. Specimens of standard samples were manufactured using these techniques to evaluate the mechanical properties as uniaxial tensile (yield strength, rupture tensile and elongation), transverse rupture strength and the micro hardness. The mechanical properties showed higher values in the SLM specimens than the casting specimens. Before the mechanical tests the specimens were examined using optical microscope (OM) and SEM-EDS. The micrographs revealed a microstructure with finer morphology in the SLM technique and the dendrites in the casting technique.

Introduction

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

Currently, another form of consolidation with large development is the additive manufacturing (AM) techniques, which has allowed innovation, reducing time and cost in many industrial areas. Rapid prototyping can be manufactured complex and high difficulty shapes to be created removing material, such as milling [4].

At the moment several studies of consolidation of the powder by laser sintering using the Co-Cr alloy are being developed. The mechanical, physical and electrochemical properties of Co-Cr-Mo alloy for dental implants were evaluated for better determination of the production parameters sintered, with the aim of improving the restoration and medical implants [5,6,7].

Manufacturing components in the medical and dental area by powder metallurgy techniques using the AM will provide knowledge of these processing techniques employing alloy Co-Cr in the form of particulate material. These techniques are of great importance knowledge of performance properties, dimensional, mechanical and microstructural of sintered this alloy compared to melted and shaped.

Obtained properties must meet specific characteristics of health care performance. Therefore, the performance analysis of the components manufactured by SLM technology is a relevant subject in the scientific and industrial area, requiring studies to evaluate their performance and viability of manufacturing relative to other manufacturing routes. Based on the ISO 22674:06 standard alloy is classified by up to five possible levels according to the obtained mechanical properties [8]. The aims of this study are compare the mechanical properties and microstructural characterization of standard samples manufactured by the selective laser melting (SLM) and casting tecnhiques.

Materials and Methods

The ASTM F 75 alloy gas atomized used in this study was supplied by HighBond®. The chemical composition of the alloy of gas atomized powder can be confirmed by X-ray fluorescence in the Table 1.

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

Alloy	Content of elements [wt %]			
	Co	Cr	Mo	Fe
Powder	63,858 ± 0,067	28,965 ± 0,042	7,019 ± 0,013	0,159 ± 0,008

The physical properties of the CoCrMoFe powder gas atomized were obtained such as apparent density, tap density and flow hate by funnel Hall [9] [10] [11]. The particle size distribution should be carried out using a 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 specimens were consolidated by investment casting (lost wax casting) and selective laser melting (SLM) techniques. The investment casting specimens were melting at 1380°C to under ordinary atmosphere according to ASTM F75. The standard samples were finished by sandblasting with oxide aluminum to remove burrs. SLM specimens was consolidated in a laser melting machine SLM®280^{HL} (SLM Solutions®, Germany), with manufacture recommendations. The samples consolidated by both processes have not undergone heat treatment. The tensile specimens were manufactured in standard dimensions according to ISO22674:06 [8]. The mechanical tests were performed in a universal testing machine Instron 4400R, at a crosshead speed of 2 mm/min. The elongation property was measured by the gauge length before and after tensile tests.

After metallographic preparation (grid and polish) the specimens were chemical etched in a solution of HCl, H_2SO_4 and HNO_3 for 60 to 240 seconds at 45º C. Microstructural observations and the composition analyses of the CoCrMoFe specimens were analyzed respectively via optical microscope (Olympus - BX51M) and SEM-EDS Philips XL30.

Results and Discussion

The characteristic format of the powder process fabrication by gas atomization is observed in the Fig. 1. The satellites (appointed by arrows in Fig.1b) can be formed in the surface particles during the cooling process of the spherical powder particles during gas atomization. It is noteworthy that the shape of the particle influences on packing properties, flow hate and compressibility, as well as reports on the powder metallurgy process [2,12,13]. The satellites are formed by collisions, in the cooling column, between the small particles with larger particles during the completion of gas atomization process. The analysis in SEM show that the powders are spherical and presented satellites. The cross-sectioned powder was etched to reveal the microstructure (Fig. 1c is). SEM micrograph shows the dendritic morphology with the primarily arms and ramifications, characterizing the rapid solidification of gas atomization process. The analysis with EDS spectroscopy (Fig.1d) show the chemical elements of the powder composition.

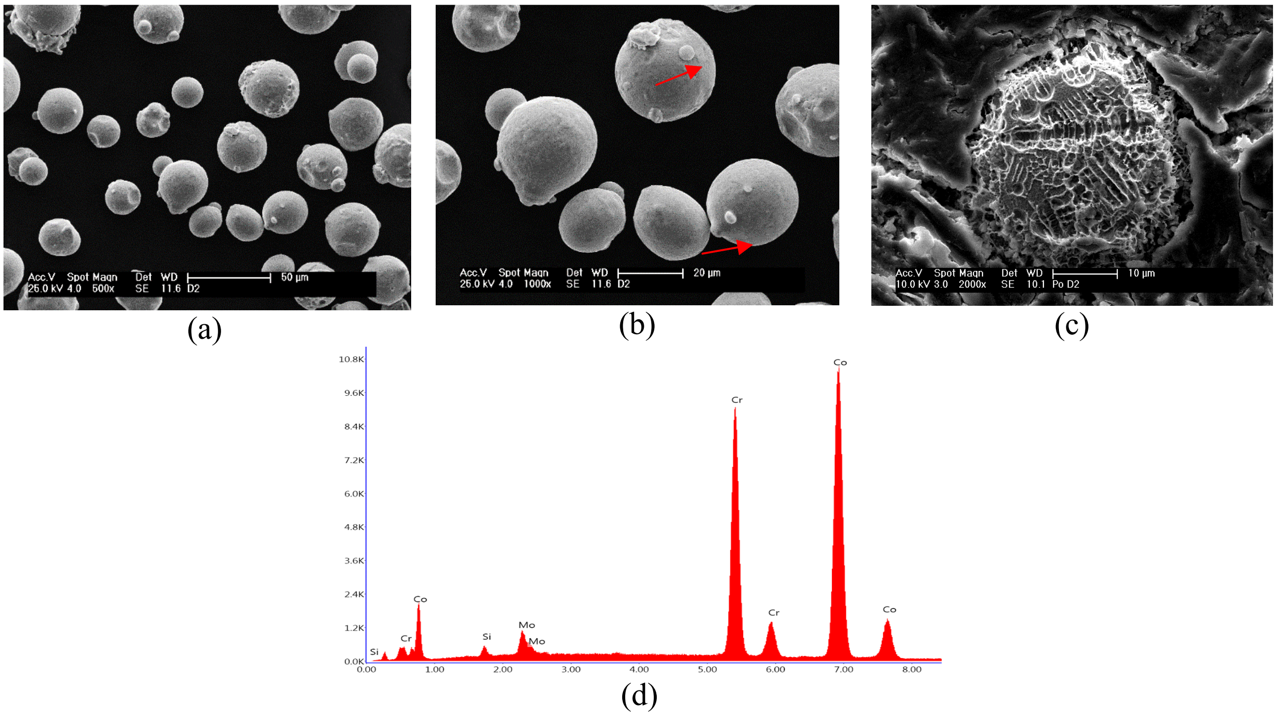


Fig. 1 – a) and b) Micrographs of atomized powder (arrows indicate the satellites), c) cross-section of powder after etch and d) EDS spectroscopy of powder.

The powders for SLM manufacture technique have a mean diameter less than 50 microns to improve the physical properties like as flow time, apparent density and tap density. The results of physical powder properties are summarized in the Table 2.

Table 2 – Physical properties of CoCrMoFe powder.

Properties		Powder	Standard
Granulometric Distribution [µm]	diameter of 10%	20,88	
	diameter of 50%	31,11	
	diameter of 90%	46,10	
	medium diameter	32,36	
Flow Time [s/50g]		15,88	MPIF 03
Apparent Density [g/cm³]		4,51	MPIF 04
Tap Density [g/cm³]		5,28	MPIF 46
Relative Density [g/cm³]		8,37	
Picnometry Density [g/cm³]		8,30	

The mechanical results of the tests for the specimens consolidated by casting and selective laser sintering are present in the (Fig. 2 and Table 3. Analyzing the values is possible to verify that in all properties the SLM technique are higher values than casting technique. However, casting technique presented lower values for the yield strength (Rp 0,2%). In according to standard ISO22674:06 the SLM and casting techniques satisfied respectively the type 5 criteria and type 3, in all mechanical properties [8]. To compare the levels of the standard, the mechanical properties are Rp 0,2% (MPa), elongation (%) and elastic modulus (GPa) and for the type 5 and type 3 levels the values are shows Table 3.

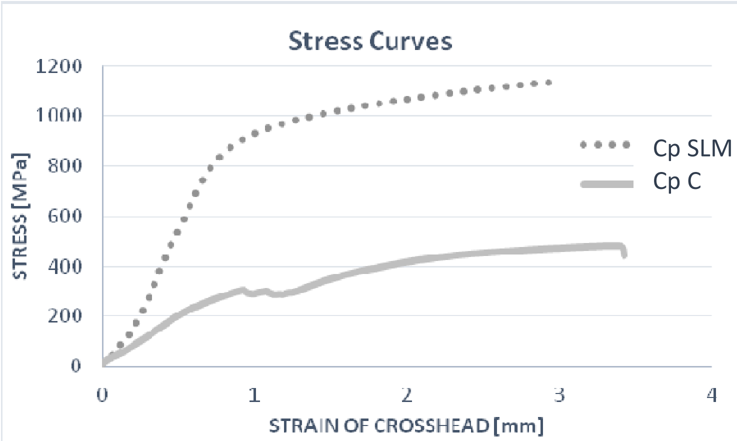
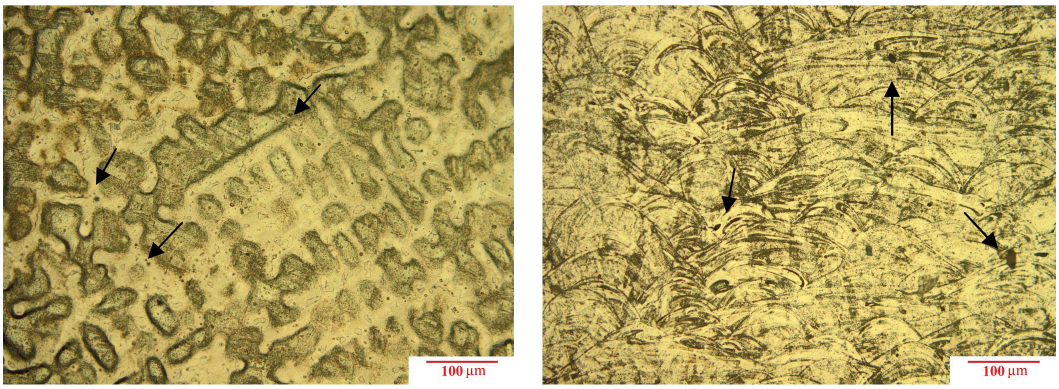


Fig. 2 – Stress curve of CoCrMoFe samples (cast sample - Cp C and selective laser melting sample – Cp SLM).

Table 3 – Mechanical properties of CoCrMoFe specimens fabricated by casting and selective laser sintering technique. (*NU = not usual)

Mechanical Properties		Consolidation technique		Standard ISO 22674: 06	
		Cast	SLM	Type 5	Type 2
Rp 0,2%	**[MPa]**	276,20 ± 43,60	731,50 ± 40,31	**500**	**270**
Rupture Stress	**[MPa]**	391,03 ± 88,91	1127,91 ± 0,15	**-**	**-**
Max. Stress	**[MPa]**	453,62 ± 75,91	1136,95 ± 0,92	**-**	**-**
Elongation	**[%]**	8,37 ± 4,45	13,73 ± 5,32	**2**	**5**
Elastic Modulus "E"	**GPa**	291,21 ± 15,22	276,69 ± 12,63	**150**	**-**
Micro Hardness	**HV**	365,74 ± 16,15	420,62 ± 21,16	**ISO 14577-1**	

To understand the mechanical properties improved in the SLM specimens in relation to the casting process technique was carried out the microstructural analysis by OM and SEM-EDS. Is possible to check the presence of porous in the both specimens (Fig. 3a and 3b), however at the casting sample the porous is smaller (microporous), but in large quantities. The porous in the laser melting are uneven (a little larger but in small quantity), occurred by problems of dispersing the powder in the bed layer and the presence of satellites/porous in the particulate. The microstructural analysis is describe a dendritic arms and ramifications with different solidification orientations (Fig. 3a). SLM specimens shows a characteristic morphology (weld-like structure) of laser bean melting, and is possible to check the layers formed during the manufacture process ((Fig. 3b).

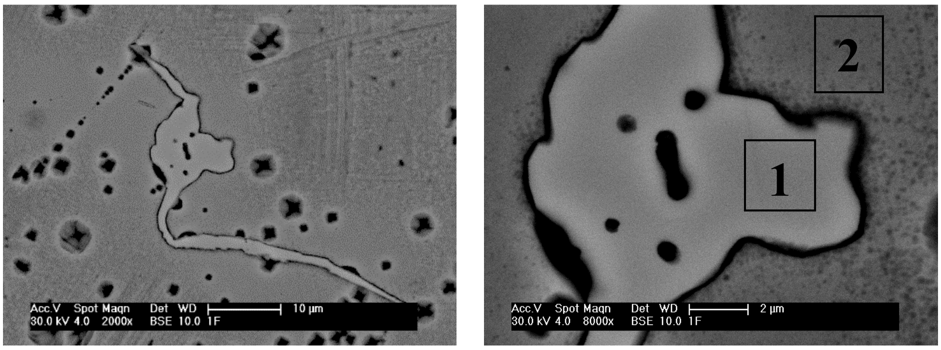


(a) (b)

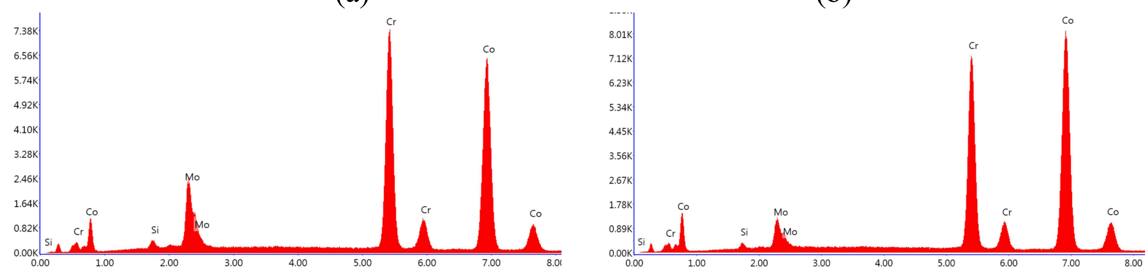
Fig. 3 – Optical micrographs of CoCrMoFe specimens after chemical etch (arrows indicate the porous) - a) as cast sample and b) as SLM.

Cast and SLM specimens was analyzed by SEM-EDS, like as show in Fig. 4 and Fig. 5, with backscattering electron (BSE) images after chemical etching. Fig. 4 is possible to identify the cast specimen with a second phase (white area) in the matrix. The semi quantitative analysis with the EDS and the respectively spectrums (Fig. 4c and d) shows that the composition of white area (point

1) is rich in Mo element and the matrix (point 2) are composed by Co-Cr elements, with a small percentage of Mo. The phase (point 1) shows the confirmation of carbide ($M_{23}C_6$) presence, rich in chromium and molybdenum.



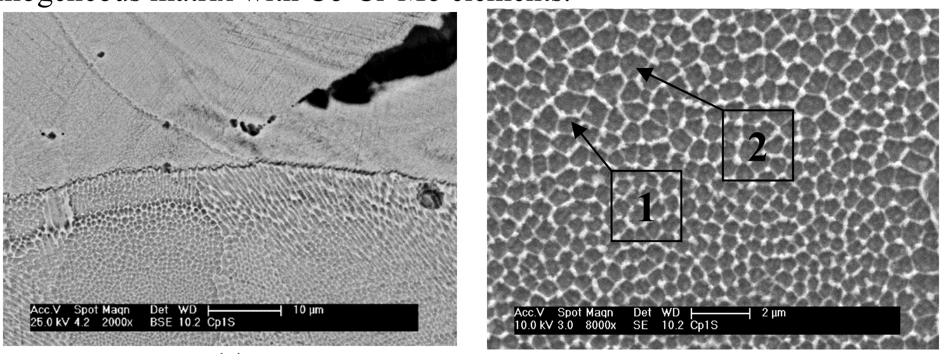
(a) (b)



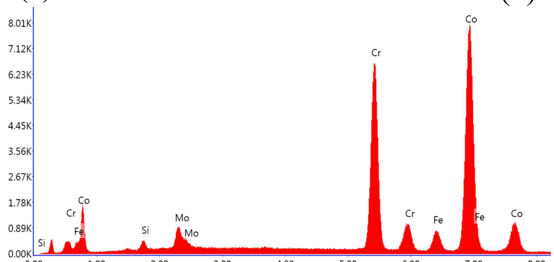
(c) (d)

Fig. 4 – a) SEM image of cast sample, b) high magnification of sample with points of EDS analyses, c) spot 1 and d) spot 2 spectrums of EDS analyses.

SLM specimen, as show in Fig. 4, presented a microstructure formed with small grains characterizing the rapid solidification during the SLM manufacturing process. The semi quantitative analysis in the fine grains shows that has not different elements compositions. SLM specimen presents a homogeneous matrix with Co-Cr-Mo elements.



(a) (b)



(c)

Fig. 5 – SEM images of SLS sample, a) and b) BSE image of cast sample etched and the points of EDS analysis, c) spectrum points1 and 2 of EDS analyses.

The morphology formation of the laser melting sample was also observed after electrolytic attack [14]. As well as possible confirm that the fine grains are oriented in direction of the laser scanning. This characteristic microstructure of laser melting technique allows achieve better mechanical properties than the cast technique [14].

Conclusions

The particles in the study produced by gas atomization showed spherical particle geometry with satellites and internal porosity, which can compromise the additive manufacturing process.
The SLM technique allowed obtaining samples with superior mechanical properties of the cast technique. In this case, the yield strength and elongation were respectively 69,73% and 39,04% greater than the yield strength and elongation obtained in the cast samples.
The microstructural characterization of the casting samples showed formation of rich carbide chromium/molybdenum and the dendritic arms and ramifications.
The selective laser melting allowed obtaining samples with improved chemical homogeneity over the molten sample by the rapid and localized solidification.
The processing using laser sintering proved superior technique to casting processing, allowing the use of this technique in the manufacturing area of prosthetics and dental implants.

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