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Evaluation of Performance of Cast and Laser-Sintered cr-co Alloys for Dental Applications

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Abstract

Dental prostheses may be built by additive layer manufacturing, specifically by direct metal laser sintering (DMLS). This innovative process allows a high percentage of unmanned work and the direct fabrication of parts from CAD data. Even if , in the product and production development field, these techniques are studied since last '80, up today there is a lack of knowledge about mechanical performance in the medical application. The target of this paper is to investigate the mechanical and functional characteristics, with respect to traditional cast parts. Tensile specimens were built in accordance with ASTM E8M both by DMLS (EOSINT-M270) and traditional lost-wax casting, using the same Cr-Co alloy. An experimental plan was designed to evaluate the effect of all process phases. Tensile performances, hardness, roughness and dimensional measurement, rupture surfaces SEM observation and porosity evaluation were performed .Sintered parts are rougher and proved to have Rockwell hardness values higher confront to cast ones. No significant dimensional variations were noticed among different processes. Some of the cast specimens present defects (macro porosity) that are absent in the sintered ones.UTS of all sintered specimens (~1400MPa) is almost double than of cast ones, whereas are more fragile (εb~5 vs 20%). The porosity in zone free of defects are comparable. Cr-Co specimen produced by DMLS show excellent strength and absence of defects with respect to traditional casting. Low εb values are not critical, since deformation of the final prostheses is limited by the ceramic layer fragility.

Keywords: Direct metal laser sintering, lost-wax technique, Co-Cr alloy, mechanical performance

INTRODUCTION

- *use of Co-Cr alloys in dentistr: state of art*

Metal-ceramic fixed partial dentures (FPDs) have been a standard replacement for missing teeth for many years. Studies on the longevity of FPDs fabricated using high-noble alloys have shown survival rates of 80 to 98%, 81 to 97%, and 74 to 85% after 5, 10, and 15 years, respectively [1-7].

Since the price of gold was deregulated in 1971, gold prices have continued to increase [8]. The cost of dental treatment is important to many patients, and the pursuit of less expensive materials has been an incentive for developing new alloys suitable for metal-ceramic prostheses. To avoid the cost of high-noble metal-ceramic FPDs, other porcelain-compatible metal alloys were developed. In addition to lower cost of the metals, the alloys exhibit a number of properties that decidedly are superior to those of gold-based alloy restorations [9]. A higher modulus of elasticity is another reason for using non-noble alloys, especially in long-span and cantilever FPDs. In this regard, most base-metal alloys have a modulus of elasticity which is almost twice as high as that of noble and high-noble alloys [10].

In recent years, the use of alternative alloys such as titanium and Cr-Co ones has increased worldwide [11-13].

However, the choice of base-metal alloy for prostheses differs around the world. Some countries use nickel-chromium (Ni-Cr) alloys with or without beryllium (Be), Cr-Co is the alloy more widespread in Europe and Japan; alloys containing more than 1% nickel are not used [10-15]. The metal base alloys are inherently less expensive than those based on gold besides studies have shown that the bond strength of porcelain to base-metal alloys may be equal, or greater than, the bond strength to high-noble alloys [19, 20]. But they also have a number of disadvantages:

- the fusion and casting temperatures are appreciably higher than those of precious-alloys this makes it difficult to manipulate these alloys in the laboratory [16];
- different casting methods must be employed[17,18];
- the process is more knowledge-based than traditional one. [9]. The formation of heavy oxides on the surface of the framework complicates soldering and fusing of dental ceramics to the structure.
- fusing porcelain to a base-metal alloy is more technique-sensitive than fusing porcelain to a noble alloy, in vitro

- *Production Processes and New Technologies*

The long-term success of crowns and fixed partial dentures on natural teeth and implants depends on several variables, including but not limited to the accuracy of fit between castings and abutments and teeth, impression materials and techniques, material manipulation, casting and finishing processes and soldering. With conventional castings, the fit depends on the accuracy of the impression and the master production, expansion and contraction associated with the phases of the process [26]. Over the last decade, reverse engineering, computer-aided design, computer-aided manufacturing and additive layer manufacturing(RE, CAD, CAM, and RP) have been employed in medicine and dentistry. [29-39]

Medical and dental applications are suitable to be produced by additive layer manufacturing, due to their complex geometry, strong individualization and high-aggregate price. Moreover, the manufacturing of multiple unique parts in a single production run could enable mass customization[28]

The technique used to produce parts in sintered cobalt-chromium alloy is named DMLS (Direct Metal Laser Sintering). The process melts the metal powder entirely, creating a fine, homogeneous structure, the parts have excellent mechanical properties, high detail resolution and a very good surface quality [28]. DMLS enables the formation of cavities and undercuts which, with conventional methods, cannot be produced or can be produced with great difficulty. Therefore, the technology is ideal for application which require maximum individuality-for example dental crowns. Metal parts of the most complex geometries are built layer by layer, directly from 3D CAD data, fully automatically, in only few hours and without tooling.

The combined action of advantages deriving from the application of additive layer manufacturingprocesses, in both the product development phase and that of tool production translates into good savings in money and delivery times.

The DMLS technique for dental application has the potentiality to substitute the traditional lost wax technique.

Obviously the correspondence between the final production and the clinical situation depends from an accurate impression sent from the clinician. Starting from the same plaster model, the production process, DMLS or lost-wax, have to give a final restoration to fit perfectly the die.

The hypothesis is that the DMLS process is more repeatable, accurate and less operator-sensitive than the lost-wax technique.

MATERIAL AND METHODS

Tensile specimens were produced, following ASTM E8M. The specimens dimensions are showed in Figure 1 (thickness = 3.6 mm).

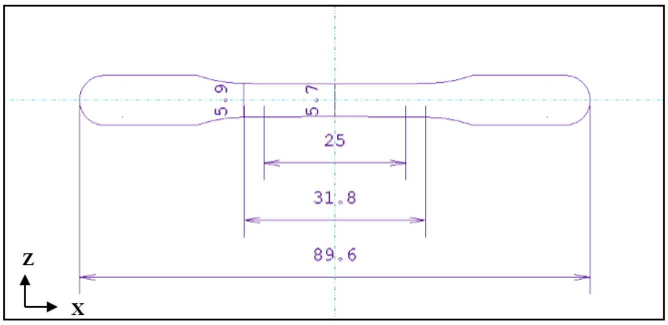


Figure 1: The specimen main dimension

Parts were produced both by DMLS and traditional lost-wax casting. To fabricate the laser-sintered specimens standard parameters were used on EOSINT-M270 (table 1).

Table 1: Parameters used for building the dmls specimens

PARAMETERS	
laser power	200W
laser spot diameter	0.200 mm
Scan speed	up to 7.0 m/s
Building speed	2-20 mm^3/s
Layer thickness	0.020 mm
Protective atmosphere	max 1.5% oxigen

The machine uses a Yb-fibre laser, that sintered the layer with the subsequent strategy of construction: every layer is divided in many squares of 4 mm side. On every square the laser act with wipes directed according to a certain scansion vector. Every layer this direction is turned of 25° respect to the precedent layer. In figure 2 is represented a schematic drawing of this.

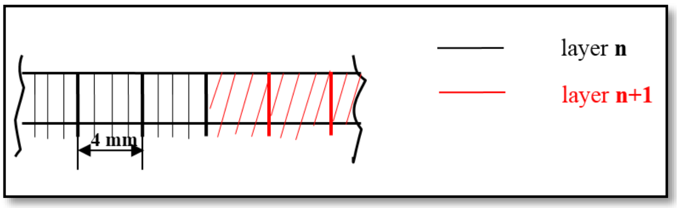


Figure 2: Laser scanning strategy

It has been used the same Cr-Co alloy for both processes, EOS CobaltChrome SP2. It is a fine powder mixture which produces parts in a cobalt-chrome-molybdenum-based superalloy. EOS CobaltChrome SP2 is a Co, Cr, Mo and W based alloy in fine powder form. Its composition corresponds for type 4 CoCr dental material in EN ISO 22674:2006 standard. It also fulfils the chemical and thermal requirements of EN ISO 9693 for

CoCr PFM (porcelain fused metal) of dental materials (Ni content: < 0.1 %, no Cd or Be) and requirements of EN ISO 7504, EN ISO10993-1:2003 and 10993-5:1999 regarding the biocompatibility and cytotoxicity of the dental materials. The powder used for the production of samples has been observed with SEM, with the help of semi-quantitative X-ray microanalysis, as to detect particle size and chemical composition of the product of departure.

The nominal composition (% by mass) alloy declared by the manufacturer are indicated in table 1[reference]

Table 1: Cobalt Chrome SP2 weight% composition

Co	Cr	Mo	W	Si	Mn	Fe
62-66	24-26	5-7	4+6	0.8-1.5	max 1.5	max 0.7

An experimental plan was designed to evaluate the effect of all process phases, which is shown in table 2.

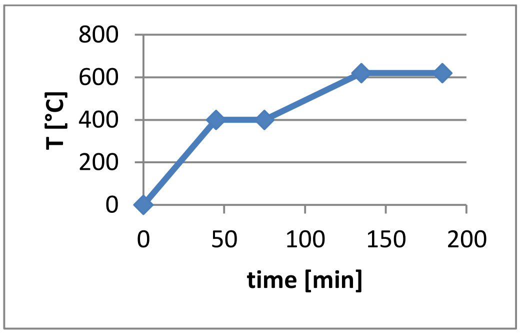
A group of six parts was left as-sintered (DMLS_L) and the others were heat-treated for stress-relief (DMLS)(figure…). Wax parts were built by drop-on-demand, invested in ceramic and burnt away before casting (CAST). Then, two groups of specimens were subjected to the high temperature treatment used in ceramic covering without deposition (DMLS_H, CAST_H). Of course, the experimental result of specimens subjected to this last typology of thermal cycle , just because they follow closely the orthodontic prosthetic treatment, will be of primary importance.

Table 2: Experimental plan scheme for the production of specimens

TECHNIQUE	N. SPECIMEN	TREATMENT		NAME
Direct metal laser sintering	6	as sintered		DMLSTQ
	6	relief heat treatment		DMLSRHT
	6		heat treatment applied with the same parameters used for the ceramic covering	DMLSCHT
Casting technology	6			CASTTQ
	6	As manufactured		CASTCHT

The standard thermal treatment of distension made by the company is composed of four stages (figure 3a), after that the oven door is open and left cooling

Table 5: Thermal cycle used for the standard thermal treatment of distension for the dmls specimens



The ceramic thermal cycle has been carried out by a dental technician appropriately equipped. A standard cycle of this type is used for the deposition of many layer of ceramic: three layer of opaque, two of masses, and one layer of glasure .

Table 5b: Parameters used for virtually baking the ceramic on the H specimens

Stage	Temperature	Time	Vacuum
Opaque 1/2	500	10 min	No
	500->980	80°C/min	Yes
Opaque 3	500	10 min	No
	500->960	80°C/min	Yes
Mass 1	500	6 min	No
	500->920	50°C/min	Yes
Mass 2	500	6 min	No
	500->910	50°C/min	Yes
Glasure	400	5 min	No
	400->900	70°C/min	No

On all specimens (6 for each of 5 groups) thickness was measured through a measuring microscope (Kestrel 200 by Vision Engineering, equipped with Quadra-check metrology software). Roughness was determined on Lm=4,8mm.Ultimate Tensile Strength (UTS) tests were carried out on at least 5 parts for each typology at a speed of 5mm per minute, a load cell with a maximum of 20kN was adopted for cast specimens and one of 250kN for dmls. Specimens were tensile tested obtaining UTS and percent elongation.For every group of specimens the mean and the standard deviation has been calculated on the test results.

After testing, rupture surfaces were observed using the scanning electron microscope (SEM) to investigate failure mechanisms and joining phenomena between the particles.

It was chosen to observe the surface rupture of a sample for each type of process (so five in total), choosing a representative sample that showed characteristics of tensile strength close to the average recorded for that group.

A second observation made at a scanning electron microscope (or SEM - Scanning Electron Microscope) covered polished section surfaces of some samples, chosen as previously described.

Two sections have been observed along different plane of construction: xy and xz

The specimen was also observed at the optical microscope (OM) to calculate data related to porosity. Several micrographs for each magnification were acquired through a CCD camera, made binary and analyzed through a software tool for image analysis to obtain:

- Percentage porosity, calculated as the area fraction of pores out of the overall area;
- The average size of pores

To observe the sections at SEM and OM, the specimens were cut with a metallurgical microcutting, then embedded in a metallurgical epoxide resin and polished till a fine grinding. Final polishing was carried out with a plan cloth and a 1 μm diamond suspension.

RESULTS

- *SEM observation of powder EOS CobaltChrome SP2*

Figure 3a and 3b-3c show the SEM image of powder EOS CobaltChrome SP2 and relative image analysis for the definition of granulometry . The powder is predominantly made up of particles of 10-20 μm and some larger than 60-70 μm. There is also a relevant percentage of particles under 2 μm

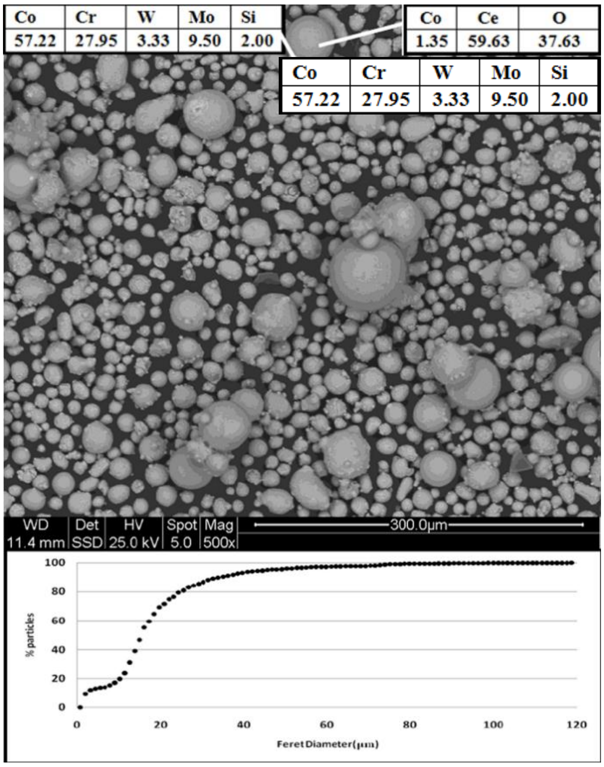


Figure 4: Powder particles of EOS CobaltChrome SP2

Spectra of X-ray microanalysis were acquired on some of the particles (Fig 3d). The results show how both large particles and small ones have the same chemical composition, summarized in terms of weight percentages in table in the figure.

The light portions adhesives on larger particles are composed of almost pure Cerium. Other particles as indicated one have an intermediate chemical composition, probably linked to small cerium deposits on the surface of particles of cobalt chromium alloy.

- *Roughness*

Table 6 report the roughness value obtained, sintered parts are rougher than cast ones (R_a~3.8 vs 2.6μm). Slightly higher roughness of DMLS parts is likely to improve mechanical bonding with the ceramic layer or might be smoothed.

Table 6: Roughness (R_a and R_{max}) of the specimens

	R_a [μm]		**R_{max}**[μm]	
	MEAN	ST.DEV.	MEAN	ST.DEV.
CAST_{TQ}	**2.62**	0.35	**18.10**	1.97
CAST_{CHT}	**2.58**	0.78	**19.01**	4.88
DMLS_{TQ}	**3.58**	0.79	**23.50**	5.24
DMLS_{RHT}	**4.02**	0.98	**23.33**	6.97
DMLS_{CHT}	**3.78**	0.37	**23.26**	5.45

- *Hardness*

The samples obtained by casting process proved to have Rockwell hardness values lower than DMLS ones. The heat treatment seems to not affect the hardness for the casting specimens.

DMLSL, DMLS AND DMLSH showed higher values of hardness of 6%, 8% and 10% if compared with the CAST and CASTH.

As regards the sintered samples, induced heat cycles on the samples have led to increased levels of hardness. The values of the largest ever were found on samples DMLSH.

Table 10: Hardness of specimens

	HRA	
	MEAN	ST.DEV.
CAST_{TQ}	**68.6**	1.6
CAST_{CHT}	**68.6**	1.8
DMLS_{TQ}	**72.8**	1.8
DMLS_{RHT}	**74.2**	1.9
DMLS_{CHT}	**75.8**	1.5

- *OM specimens observation*

Some of the cast specimens present defects (macro porosity) that are absent in the sintered ones

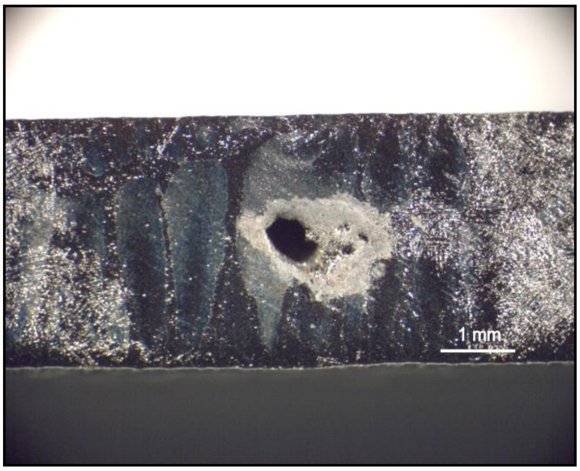


Figure 5: Macro porosity observed on cast specimens

- *Tensile Test*

The specimens have been subjected to tensile strength test following E8 standard. Results are showed in table 7

Table 7: Tensile strength and elongation of specimens

	UTS [MPa]		ε_b[%]	
	MEAN	ST.DEV.	MEAN	ST.DEV.
CAST_{TQ}	**817**	10.69	**24.08**	1.63
CAST_{CHT}	**751**	16.85	**19.49**	1.18
DMLS_{TQ}	**1339**	14.63	**8.98**	0.84
DMLS_{RHT}	**1425**	11.58	**6.22**	0.42
DMLS_{CHT}	**1441**	19.82	**4.72**	0.29

The tensile strength of DMLS specimens (~1400MPa) resulted almost double respect to the cast ones. Unlike cast parts, the heat-treatment increases UTS for DMLS. Laser-sintered specimens are also much more fragile than cast ones (ε_b~5 vs 20%).

- *SEM observation of rupture surface*

It follows some micrographs detected by scanning electron microscope with the fracture surface. The observations were conducted on samples selected according to criteria set out previosly. In all images the horizontal direction is the specimen width and the vertical is the thickness. In particular for DMLS, this means that the horizontal there is the Z axis, or the growth direction perpendicular to the layers, and vertical is the Y axis, or the direction orthogonal to the scanning laser.

CAST-type specimens.

The structure is quite homogeneous. It was not detected the presence of evident porosity. It can be noticed the formation of dendritic structure, typical of the materials obtained for casting. In figures 5a snd 5b the dendritic architecture is still visible on the surface of rupture.

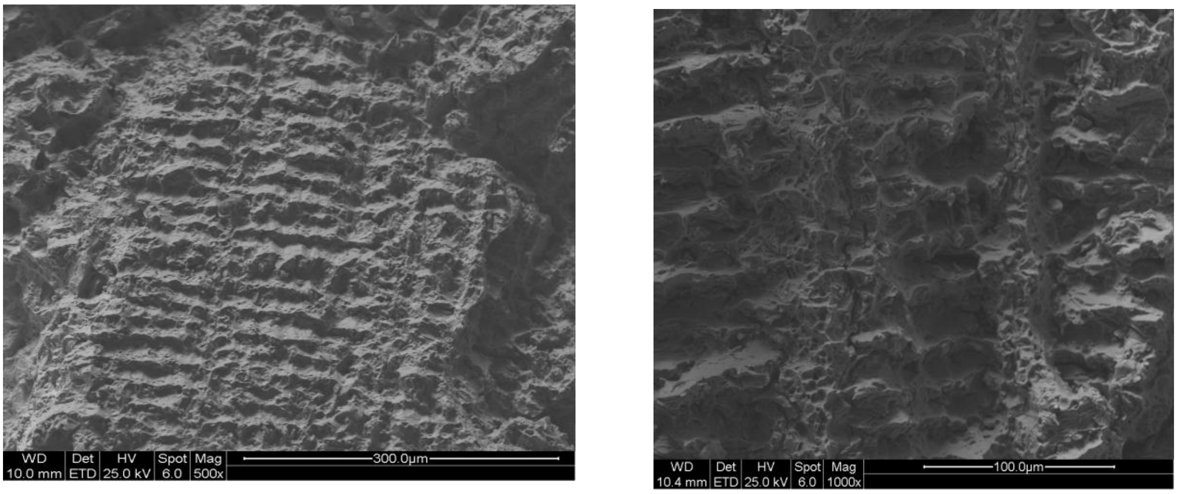


Figure 6a and 6b: SEM micrographs of cast_04 rupture surface

Even on the surface rupture of the sample subjected to heat treatment there were no porosity.

DMLS-type specimens.

The samples present a fracture surface rather homogenous, the starting powder is not more recognizable, unhomogeneosity linked to the additive construction are not visible and structure is extremely compact. The failure occurs for cleavage along clean floors, arranged at 45 degree respect to the direction of load application.

It was observed the presence of cavities of different entities, the largest ones are present figure in micrographs of 6c and 6d, between 50 and 100 μm .

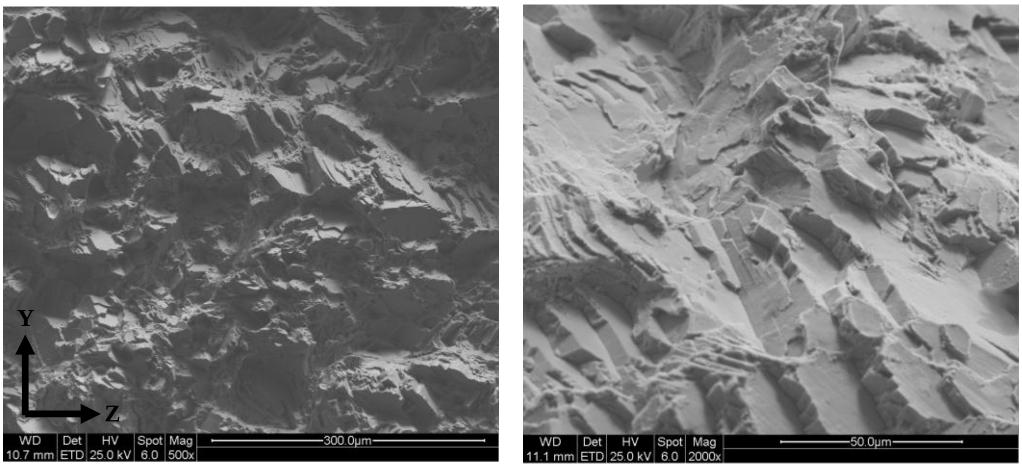


Figure 7a and 7b: SEM micrographs of dmls_06 rupture surface

- *Porosity*

It is necessary to distinguee between porosity and the presence of defects, the porosity is a characteristic of the system, for this reason it has been evaluated in zones without defect (such as macropores or grouping of micropores).

Table 8 shows the results of the porosity calculation in the sections of the different specimens. In a preliminary phase samples were observed as a whole to assess the possibility of share the same in different areas, calculating for each of them porosity.

Table 8: Porosity of specimens

SAMPLE	**SECTION AND MAGNIFICATION**	**AVERAGE SIZE[μm²]**		**AREA FRACTION[%]**	
		MEAN	SD	**MEAN**	SD
CAST_{TQ}	XY *200X*	**2.36**	0.46	**0.2**	0.07
	XZ *200X*	**3.61**	1.88	**0.25**	0.1
CAST_{CHT}	XY *200X*	**2.74**	2.05	**0.46**	0.21
	XZ *200X*	**3.3**	1.96	**0.2**	0.07
DMLS_{TQ}	XY *200X*	**33.04**	55.54	**0.54**	0.55
	XZ *200X*	**147.56**	114.3	**2.68**	1.18
DMLS_{RHT}	XY *200X*	**1.46**	0.28	**0.1**	0
	XZ *200X*	**28.59**	34.11	**1.13**	1.17
DMLS_{CHT}	XY *200X*	**2.66**	2.25	**0.14**	0.05
	XZ *200X*	**85.26**	57.18	**2.43**	1.59

The cast specimens present a rather uniform porosity with an average size of pores contained, between 0.2 and 0.4 % and the maximum size of pore is 3.6 μm^2. It can be noticed a slight tendency to increase porosity after the heat treatment. On the other hand it must be remembered that some cast specimens are affect from phenomena of macroporosity with very large pore, as described previously.

The DMLS specimen present a porosity that varies from 0.1% to near 3%, the pore sizes are very different, covering an average range for each micrograph experience ranging from 1.17 μm^2 to 144 μm^2. The porosity of DMLS specimens is similar to CAST specimens and often is major, therefore the remarkable difference in mechanical resistance of the samples is probably due to the joining mechanism induced by the processes.

- *SEM observation of polished sections*

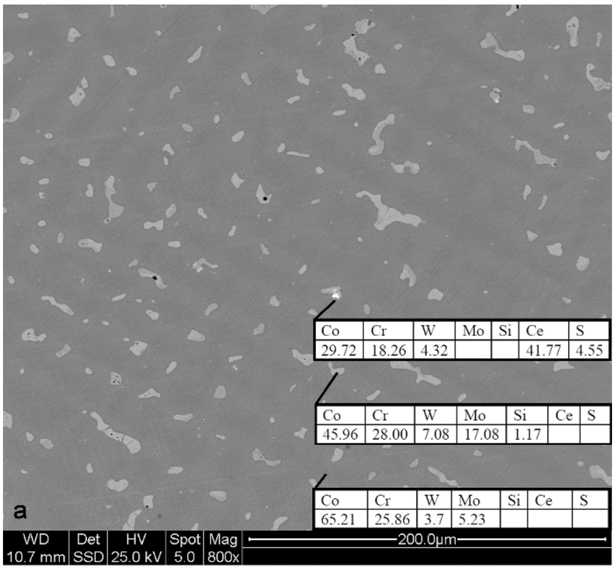
It follows some micrographs detected by scanning electron microscope with the polished section surface. The observations were conducted on samples selected according to criteria set out in material and methods.

CAST type specimens.

The micrographs (figure 7)show that the area is covered with darker shares distributed to spot leopard, which is probably a different composition zones. This is characteristic of both families of samples of type CAST. For a more detailed analysis of appearance see out of those areas held by the scanning electron microscope.

The EDS analysis indicates that the darker zone (spectrum 1) and the lighter zone (spectrum 2) are the same Cr-Co alloy with different percentage of chromium, cobalt, tungsten and molybdenum.

Whereas the white point are cerium, like is visible in spectrum 3; Cerium is not present in spectrum 1 and 2, so it can be concluded that is not melted in the alloy, but it remain solid during the casting process. The chemical composition is resumed in terms of weight percentage next to the figure.



Co	Cr	W	Mo	Si	Ce	S
29.72	18.26	4.32			41.77	4.55

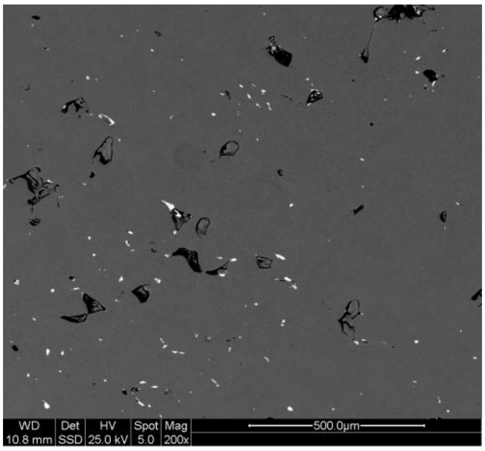
Co	Cr	W	Mo	Si	Ce	S
45.96	28.00	7.08	17.08	1.17		

Co	Cr	W	Mo	Si	Ce	S
65.21	25.86	3.7	5.23			

Figure 8: SEM micrographs of cast polished section

DMLS type specimens

The micrograph (figure 8)shows zone with very low porosity and homogeneous, and zone where there is an higher porosity, in this zones are visible with points that EDS analysis confirm to be cerium. Therefore cerium is not amalgamated neither for DMLS specimens, and it is probably correlated to the porosity



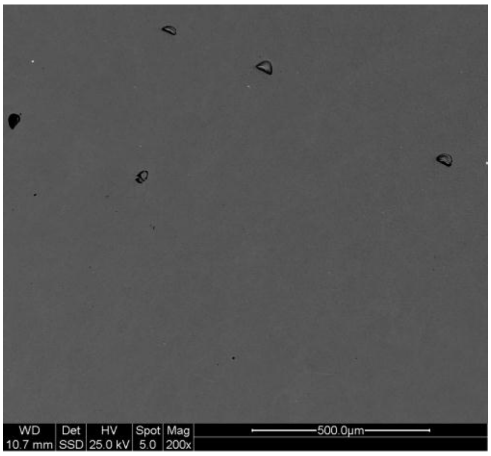


Figure 9a and 9b: SEM micrographs of dmls polished section

DISCUSSION

DMLS technique has been compared to investment casting, focusing on dental applications for prostheses production. The same Cr-Co alloy powder has been used for both processes and mechanical, physical, chemical and microstrucural properties have been compared.

The casting process resulted less predictable in term of quality, some specimens showed superficial macroporosity that can be ascribed to a non sufficient molten metal supply in the solidification phase. The result has been confirmed by the study of porosity on the polished section of the specimens. In fact the cast parts, even if they substantially present low porosity zones like DMLS, show high porosity areas, due to the shrinkage of the liquid alloy during solidification and cooling of the same. The first results is that the DMLS process could guarantee a better repeatability, because in the realised specimens such defects were not found.

DMLS showed to produce parts more strong and rigid with respect to the casting process. Therefore, at least at a theoretical level, they present excellent characteristics of affinity with the aesthetic ceramic material that covers the fixed prosthesis, since the strain modes of the two materials are quite similar. The heating treatment, in the case of the sintered specimens, further increases the tensile strength while it causes a strong decrease in the value of percent elongation.

The tensile test results were confirmed by the SEM observation of fracture surfaces: the different mechanical properties can be related to different failure modes. Whereas in the cast specimens the dendritic structure is still visible, fracture in DMLS samples is caused by scaling along cleavage planes, inclined at 45° to the direction of load application.

Observing the DMLS specimens it can be noticed that the different layers are no more visible, this indicates that the particles are strongly joined together not only within each layer but also between different layers. Isotropy in the build direction is a rarity in additive layer manufacturing techniques.

Microstructural differences concerning chemical composition have been detected with the observation of polished sections at SEM. In the cast samples the phenomenon of segregation has been observed, caused by the solidification of dendrites before and a surrounding matrix afterwards with different chemical compositions. DMLS samples are more homogeneous e don't present this phenomenon.

The material physical and mechanical properties mainly depend on its microstructure, so the observed differences at a microstructural level cause the macroscopic performances of the specimens obtained by the two processes.

The presence of Cerium has been detected, which is not declared by the producer in the nominal composition. Cerium was often found unbound, just dispersed within the alloy. This aspect is more evident on the sintered samples.

CONCLUSION

DMLS of Cr-Co alloys produces excellent strength and absence of defects with respect to traditional casting. Low ε_b values are not critical, since deformation of the final prostheses is limited by the ceramic layer fragility.

DMLS process is very reliable and repeatable, the experimental results are totally repetitive.

It can be concluded that, as to the possibility of using DMLS technology for the realization of prostheses in Cr-Co alloy, from a mechanical point of view the DMLS technique has higher performance respect to the investment casting.

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