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Study of Stainless Steel 316L by Powder Injection Molding for Application as Orthodontic Screw

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Abstract— Micro-powder injection moulding (μPIM) is an advanced and versatile net-shaphing process for the manufacturing of metal and ceramics complex micro components in the in the automotive, optical, fluidic and medical field. In this paper, different milling times (0.5, 1, 2, 4, 6 and 8 hours) of the 316L stainless steel (SS) commercial were studies to obtain an adequate particle size for the micro-powder injection molding (μPIM) process, in order to improve the injection process parameters to manufacturing of a screw prototype for the orthodontics applications. The SS powder obtained by the milling process was mixed with a binder system consisting of high density polyethylene waxes (carnauba and paraffin) and stearic acid. The injection pressure used was 50 MPa with a process temperature of 170oC and the thermal curve for binder removal was obtained from differential thermogravimetric (DTG) analyses that showed a degradation peak at 244.12 °C attributed to paraffin and at 451.75 °C attributed to polyethylene. The milling process resulted in a microstructure more refined and homogeneous, which allowed a lower sintering time (1h) and temperature (1260 °C) compared to usual procedures in the literature. This study allowed the die design of the orthodontic screw through of the injection parameters improvement by μPIM process.

Keywords— Micro-powder injection moulding, Milling process, 316L stainless steel, Sintering, Orthodontic screw.

1 Introduction

Powder injection molding (PIM) is a process metallurgy traditional powder combined with molding plastic injection, which has high reproducibility and is capable of molding complex geometries [1,2,3]. This process includes four processing staged: mixing of powders and organic binders (feedstock), injection molding of the feedstock, debinding to eliminate the binder and sintering [4].

Metallic biomaterials have been used since 1985 when Lane tested a metallic plate for bone fracture fixation due their good strength and corrosion resistance properties. However, metal implants can have poor biocompatibility, which is necessary to promote the growth of natural tissue. Conventional manufacturing processes of metallic biomaterials include casting, thermo-mechanical and powder metallurgy processes and each process has its drawback such as manufacturing costs, surface quality, and metallurgical defects. Casting and thermos-mechanical are appropriate for large metallic parts for powder, shipping and military industries and machining process is required to produce intricate shapes, but surface layer of machined parts experience residual tensile stresses and is often populated with many microcracks. Consequently, these cracks lead towards the failure of implants due to corrosion. However, PIM process can produce more feasible small and intricate shapes [5]. Stainless steel is the most widely used materials for bone fracture fixings being biocompatible, having good mechanical strength, ductility, manufacturing ease and low cost compared to other materials. As orthodontic implant 316L stainless steel, they are used as an alternative to assist in temporary and stable anchorage for the orthodontic movement. They have lower corrosion resistance to body fluids when compared to other metallic alloys [6,7]. The interrelationship between the implantology and orthodontics in orthodontic treatment planning should present safer and more predictable results, for both the patient and the professional. The implantology is the insertion study of materials and devices in order to restore prosthetically the function and the esthetics of the patient, fully and partially edentulous. In turn the orthodontics is the branch related to the condition of facial abnormalities (orthos meaning straight and odontos means teeth). In this respect, the greatest implantology contribution in the advance of the orthodontic technology is to assist the orthodontic anchorage control. There are several companies in the implantology segment using various micro-screw designs with many orthodontic purposes. Almost all orthodontic micro-screws have an orifice in the head for accessories attachment, and others have different slots types or round heads [7]. In this work, the SS commercial powder was studied through of the μPIM process to improvement of the injection parameters well-known in the literature, in order to project a molding to manufacturing of a screw to orthopedics application.

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2 Materials and methods

In this work, a commercial stainless steel 316-L powder from Osprey Metal Powder-(England) was used to obtain a sintered micro-component for odontology applications. The SS powder shape was mostly spherical and a good uniformity

can be seen in Fig. 1. The SS powder milling was carried out in an attritor mill of high-energy in different time (0,5, 1, 2, 4, 6, 8 h) with solid lubricant based on the zinc stearate (1 wt%) and mill steel balls were added to promote high energy impacts, fragmenting the powders placed there to obtain alloys by mechanical synthesis. The rotation used in the mill was 1000 rpm, and the camera was cooled with water to prevent an excessive increase of temperature. The load was prepared with the composition shown in Table 1, with total mass of 1.000g.

TABLE 1
UNITS FOR MAGENTIC PROPERTIES BINDER MIXTURE COMPOSITION USED IN THE INJEC-TION MOLDING PROCESS.

Binder	Mass [g]	Weight [%]
High density polyethylene	30	3.0
Carnaubawax	15	1.5
Paraffin	50	5.0
Stearicacid	5	0.5
Powder of 316L stainless steel (Mechanical alloying)	900	90
Total	1000	100

After, the SS mixed with zinc stearate and steel balls were placed in the mill chamber with protective under argon atmosphere [8]. The binder system employed was composed of 3% high density polyethylene, 1.5 %, carnauba wax, 5% paraffin wax and 0.5% stearic acid, with 90 vol.% powder loading. The SS powder milled for 2 h and binder system were mixed in a mixer at 160 °C to prepare the feedstock. Then, the mixer continued working for 6 hours to achieve a good distribution of the powders in the binder systems as well as a good homogeneity of the mixture.

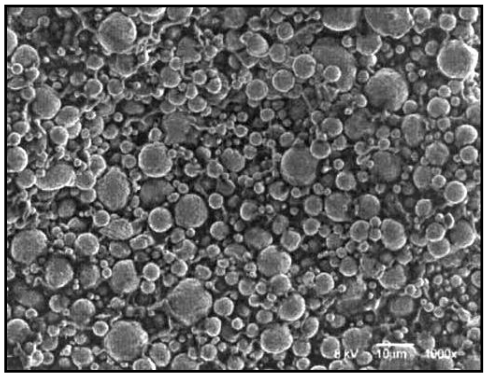

Fig. 1. Micrograph of powder of 316L stainless steel.

The feedstock was pelleted in mill knives and sieved to remove fine particles and to avoid funnel locking problems. Then, the feedstock was used to inject the samples into a mold with prismatic cavity injection molding machine (Arburg - Model 170s). This form allows a number of sample evaluation cited above, as cavity filling, dimensional variation and contraction at 170 °C under the pressure of 50MPa. The temperature was kept homogeneous and for all injection stages were kept at the same temperature. Figure 2 shows the injection die (mold) used for manufacturing samples in the form of prism, mounted on the injection machine. The schematic drawings of the samples and the die used in the injection can be seen in Figure 3.

Fig. 2. Sample die (mold) mounted in injection machine.

Fig. 3. Schematic drawing (a) Sample in the prism form (b) Die (mold) fix (c) Die (mold) mobile.

The binder removal was performed by chemical and thermal extraction, using hexano as solvent at 60 ^{o}C. The extraction cycle was defined from the thermogravimetric data of the binder system components, which in most cases involve heating levels (time and temperature) showed in Fig. 7. The sintering was carried out in an oven at 1260 ^{o}C for 1 hour, using a 5 ^{o} C / min heating rate, followed by a slow cooling. Differential scanning calorimetry (DSC) analysis and DTG were conducted in a TA Instrument DSC equipment, model Q600, with nitrogen gas at a heating rate of 10 ^{o}C/min in order to test the thermal properties of the binder system. The sintered SS was polished and then etched with a suitable etchant to analysis microstructure in optical microscope and the structure and surface were examined by SEM microscopy.

3 RESULTS AND DISCUSSION

Fig 4 shows that the particle size decreases with increasing the milling time until become stable and even at different milling times the size range particle was observed between 4 and 27 μm.

It was also observed that after six hours of milling, there was no average particle diameter significant variation with grinding increasing time. Comparing the milling time with the average particle size, as shown in Figure 4, it is possible to conclude that, from 1.6 milling hour, is obtained a 20 μm average particle size. This particle size is suitable for the powder injection molding process. As shown in Figure 5, it is possible to verify a tendency to decrease in particle size. It is also observed that, with increasing milling time, there is a tendency to form aggregates. The composition injected difficulties in the injection, and it was the most appropriate, with repeatability

values found in the green density, which indicates that the mixture was homogeneous. The mixing time used was 6 hours. The viscosity of the binder-powder mixture is very sensitive to temperature as well as to the particles percentage of the powders in the mixture. At low temperatures, the viscosity is very high, then the molding process is not allowed. Furthermore, at high temperatures, the binder coating that humidifies the powder surface is very thin, which promotes the segregation during injection. Furthermore, it can result binder degradation. The injection pressure is also of great importance at this stage of the process, because when this is appropriate, enables the filling of the cavity without causing distortions and adhesions of the components in the die walls. The green density of the injected samples was approximately 4.25 g/cm^3.

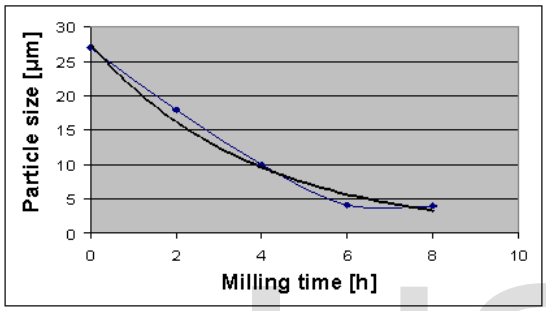

Fig. 4. Particle size distribution of the processed powder.

The binder removal time most suitable for this system was 3 hours, immersed in solvent hexane. The samples subjected to chemical extraction maintained their original form and no changes occurred. The mechanical resistance and stiffness decreased, causing ductile characteristics in the material. It could be related with particle size (20 µm), because small particle sizes are necessary to contribute to structural details, higher aspect ratio and better shape retention of microstructures, improve the isotropic behaviour and to contributes for a finish surface [9]. Figure 6 shows the chemical extraction curve of the binder, using hexane as a solvent. In general, the chemical extraction is performed to reduce the extraction time for next stage (thermal extraction). The piece immersed in the solvent (hexane) dissolve one or more binder components, but not attacks, at least one of them, which is responsible for retention of shape and, subsequently, is removed in thermal extraction.

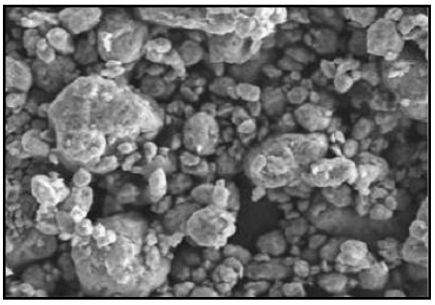

Fig. 5. Micrography of the millied powder

A thermogravimetry analysis (Figure 6) was performed in the injection load used (not shown). From the thermal curve obtained it was possible to define the cycle of thermal extraction where the polymer degradation takes place. It was noted that there are two temperature regions where loss mass occurs. The first region shows a peak at 264.12 °C, indicated by the derivative (DTG), attributed to wax and paraffin degradation. The other region, which peak occurs at 451.75 °C, is assigned to the polyethylene degradation. These parameters are used in the sintering curve.

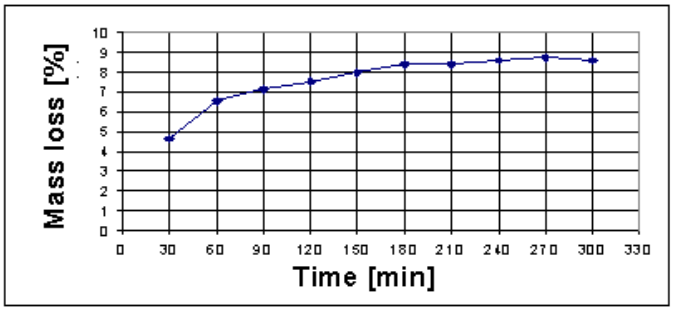

Fig 6. Chemical extraction curve using hexane as a solvent in the binder removal at temperature of 60 °C.

The sintering was performed in the temperature of 1260 °C by an hour, as shown in the Figure 7. It is important to note that it is possible to use a longer time in the sintering, since this product type has a brown density lower, when compared with products obtained by the powder metallurgy conventional process. Therefore, it requires longer sintering times or higher temperature. In this case, it was not necessary to use high temperatures or higher times in the sintering, due to the microstructure refinement that favored the material sintering at a temperature of 1260 °C.

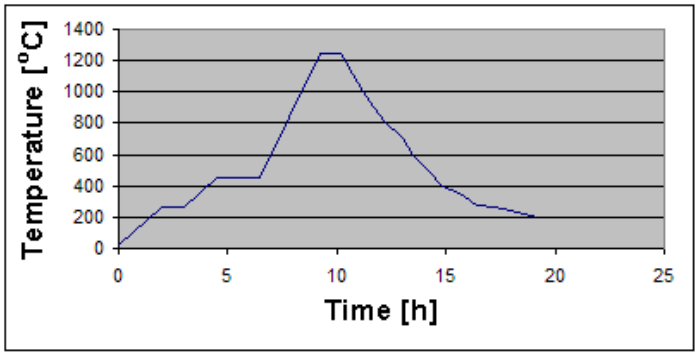

Fig 7. Thermal curve for binder removal and sintering.

After sintering, the consolidated material density was 7.80 g/cm^3. This value indicates that the temperature and sintering time used were those necessary to obtain a component with good sintering, as shown in the metallography analysis in the Figure 8, that shows the microstructure of 316L stainless steel, sintered with brown gas for 1 hour at 1260 °C temperature. In the microstructure observed it was possible to find that the material obtained from this process had a grain size more refined and more homogeneous in microstructure. During annealing treatment (1250 °C) is expected a significant grain growth [10,11] and one of the possible could be due to the

presence of the oxides in the debound sample [11]. Figure 9 shows the injected and sintered sample.

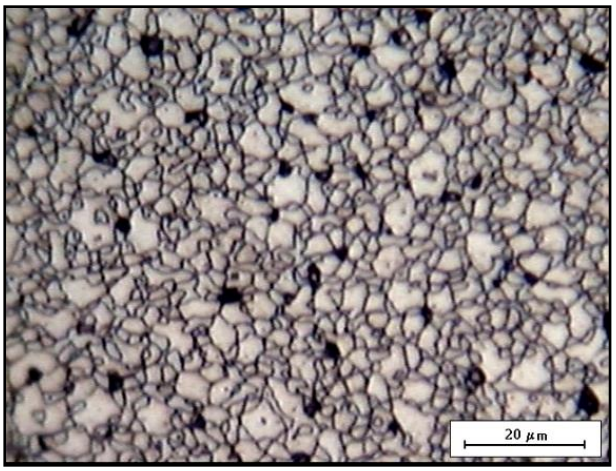

Fig 8. Micrography of 316-L stainless steel (M.A. 2 h) sintered in brown gas during 1 h at 1260 °C (500×).

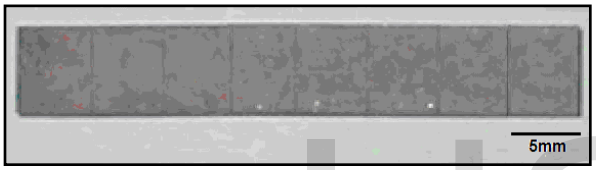

Fig 9. Samples in the prism form injected and sintered

Figure 10 shows the dimensional design of orthodontic screw to be developed. For the dimensions for de screw design, it was necessary to consider the variation in volume that the injected components are submitted after the stages of extraction of the binder and sintering. Considering the total volume, it was estimated a 30% reduction, where each dimension has a relative variation. The data obtained from the manufactured samples were crucial to obtain the orthodontic screw. Thus, the steps for preparing the powder and the load, injection temperature and pressure, thermal and chemical processes to binder removal, the time and sintering temperature, were identical to those used in the manufactured samples. The contractions have remained within the levels proposed in the literature and observed in manufactured samples.

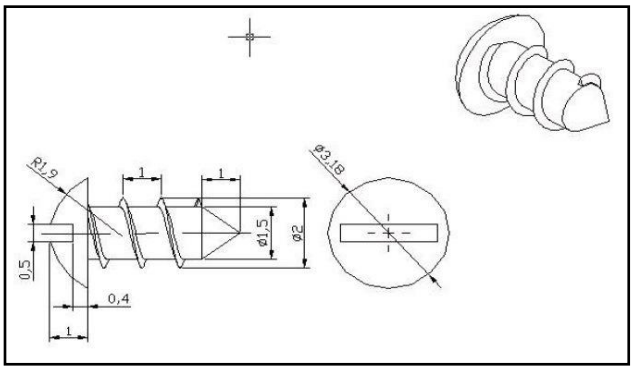

Fig. 10. Screw dimensional design for mold (die) project.

4 Conclusions

In this study the powder 316L stainless steel was milling with different time (0,5, 1, 2, 4, 6, 8 h) to obtain the particle size required for the injection of a orthodontic screw by µPIM process. The particle milling equilibrium is achieved after 6 h of processing and there are significant changes with increasing grinding time and the particle size decreases with increasing grinding time up to a welding balance. From 1.6 h of milling was obtained a mean particle size of 20 □m, which is suitable for the powder injection molding process. The increasing milling time promoted a refinement of the microstructure. Due to hardening of the particles to be a function of milling time was possible to note fractures and a greater degree of disarrangement as consequence of deformation. The powder injection molding process allowed to obtain sintered components with high mechanical properties and complex geometric shapes. The load developed showed good results of injection and an easy extraction of the binder and sintering, reaching the sintered density near the theoretical value due to the mixture homogeneity and consistency.

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