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The effect of sintering temperature on the structure and mechanical properties of medical-grade powder metallurgy stainless steels

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ABSTRACT

Nanostructured medical-grade stainless steel powders with the chemical composition of ASTM F2581 were liquid-phase sintered with 6 wt.% Mn–Si additive at different temperatures ranging from 1000 °C to 1300 °C. The effect of sintering temperature on the structure and mechanical properties of the samples was investigated. Structural characteristics like porosity, austenite crystallite/grain size, and retained ferrite were analyzed by optical microscopy, Archimedes densitometry, X-ray diffraction, transmission electron microscopy, and ferritometry. The corresponding results showed that residual porosity in the sintered specimens was reduced by increasing the sintering temperature; in contrast, the crystallite/grain sizes were enhanced. The study of the mechanical properties, including hardness, compressive, and abrasive wear behaviors, of the samples indicated that the optimum mechanical properties were obtained for the sintering temperature of 1150 °C, which were superior to those of AISI 316L stainless steel used as a conventional biomaterial.

1. Introduction

Austenitic stainless steels, especially type 316L, are widely used as biomaterials, because of low cost compared to other metallic biomaterials, good corrosion resistant and mechanical properties, easy processing, and adequate biocompatibility [1,2]. Nickel is the main element stabilizing the austenitic phase in this alloy; however, implants made of this alloy sometimes cause allergy, due to nickel ions released in the body [3,4]. For this reason, nickel-free stainless steels were introduced and are being developed in this critical area [5–7]. Typically, in ASTM standards, two nickel-free medical-grade stainless steels have been recently introduced: ASTM ID: F2229 and ASTM ID: F2581.

One of the processing methods that can be used to fabricate nickel-free stainless steels is powder metallurgy. This route induces porosity in the material, which has been recognized to be desirable for bone implants, because porosity can improve mechanical interlocking between the host bone and implant, thereby reinforcing the stability of the implant. In addition, porosity reduces the mismatch of the elastic modulus of the implant and surrounding bone and thereby improves its fixation [8,9]. On the other hand, it is known that by developing nanomaterials, many mechanical properties like strength, fracture and fatigue behaviors are improved. To merge porous biomaterials into nanomaterials, mechanical alloying is widely regarded as a nanostructured powder processing route [10,11]. The mechanical properties of porous nanostructures are controlled by grain/crystallite size and porosity configuration, suggesting a research area. In this regard, liquid-phase sintering allows a better control of density and porosity for powder metallurgy parts.

Concerning powder metallurgy austenitic stainless steels, it has been shown that the decrease of grain size increases yield and tensile strengths and impeded intergranular fracture [12–14]. Regarding liquid-phase sintering, the addition of a Cu–Sn sintering aid to produce high-strength 465 stainless steels has been tested [15]. According to this study, the maximum sintered density was achieved at 1300 °C with 3% of the sintering aid. However, in the nanometric scale, the grain size which is controlled by sintering has a higher effect on the obtained properties, compared to coarse grained materials. The investigation of the effect of sintering time on the mechanical behavior of nanostructured medical-grade nickel-free stainless steels has shown that the role of sintering time in the obtained density at the sintering temperature of 1100 °C is negligible [9]. It has been also shown that by using 6 wt.% of a Mn–Si alloy at the sintering temperature of 1050 °C, the maximum density and optimal mechanical properties of nanostructured stainless steels were obtained [16]. However, the effect of sintering temperature was not focused on for nanostructured medical-grade stainless steels. In this study, stainless steels with the composition of ASTM F2581 were synthesized by mechanical alloying, and bulk samples were prepared after adding 6 wt.% of the Mn–Si sintering aid. The structure and mechanical properties of the samples were then investigated as a function of sintering temperature.

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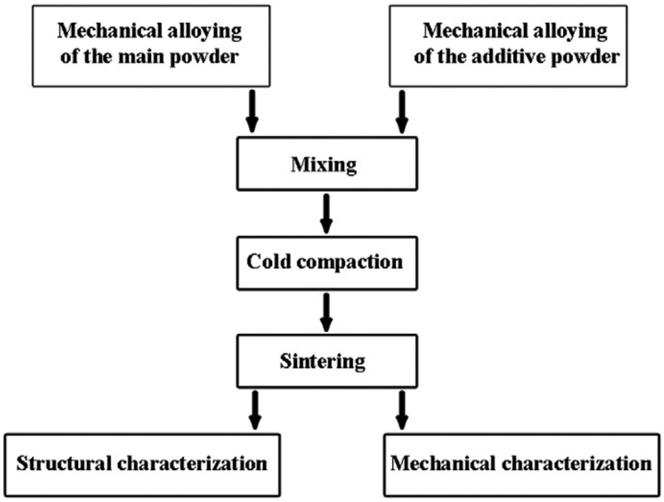

Fig. 1. Schematic representation of this work.

Table 1
Some structural parameters of the samples sintered at different temperatures (experimental error for ferritometry was less than 2%).

Sintering temperature (°C)	Ferrite content (%)	Crystallite size (nm)
1000	3	51
1050	0	[a]
1100	0	[a]
1150	0	63
1200	0	[a]
1250	0	[a]
1300	6	82

[a] Not measured.

2. Materials and methods

2.1. Sample preparation

The summary of this research is schematically shown in Fig. 1. Mechanical alloying was used in order to produce stainless steel powders. For this purpose, powders supplied by Merck with the composition of Fe–17Cr–10Mn–3Mo–0.4Si–0.5N–0.2C in wt.% (ASTM F2581) were milled in a planetary ball mill with a rate of 500 rpm and a ball-to-powder weight ratio of 20:1 under an argon atmosphere for 48 h in a tempered steel bowl. 4 bearing steel balls of 20-mm and 12 bearing steel balls of 8-mm diameters were used in this work. After alloying, 6 wt.% of Mn–11.5Si pre-alloy powder was added in order to improve the sintering behavior of the stainless steel powders. The pressed samples were then encapsulated in evacuated quartz crystals, then sintered for 60 min at temperatures of 1000 °C to 1300 °C, and immediately cooled in water for keeping the high-temperature austenitic structure at room temperature.

2.2. Structural characterization

Medical-grade stainless steels should have a fully austenitic structure. The existence of magnetic phases (ferrite) in the samples, which is detrimental in medical-grade stainless steels, was checked by ferritometry. To ensure the austenitic formation and to determine its crystallite sizes, X-ray diffraction (XRD, Shimadzu lab X-6000, Cu Kα, step size of 0.02, and step time of 6 s) was used, where the results were interpreted by the Rietveld method. A transmission electron microscope (TEM, FEI-Tecnai G2F30) was also used to further analyze the structure of a selective sample. For the TEM sample preparation, a selected sintered specimen was cut into a disk of 3 mm in diameter, ground to approximately 100 μm in thickness, and then electropolished. As the porosity level and density of powder metallurgy samples significantly affect their properties, the size and shape of porosity were also analyzed by optical microscopy. The porosity percentage of the samples was calculated by the Image Analyzer software; also, the density of the samples was measured by the water Archimedes method.

2.3. Mechanical characterization

Materials developed for medical applications, especially for orthopedic purposes, should present adequate mechanical properties. In this study, aspects of mechanical properties, including hardness, compressive, and wear behaviors were studied as a function of sintering temperature. Rockwell hardness measurements were conducted on the sintered samples on at least five randomly-located points of the surface, then the average values were reported. For studying the compressive behavior of the samples, compression tests were done at room temperature, based on ASTM-E9. Finally, pin-on-disk wear tests were also performed on the sintered samples at an applied load of 10 N and a velocity of 0.03 m/s to the sliding distance of 400 m. In the wear sliding experiments, 440C stainless steel pins of 65 HRC in hardness were employed as the slider. The friction coefficient and wear weight loss were measured and the worn-out surfaces were studied by a scanning electron microscope (SEM, JEOL-JSM 5310). Each sample was tested three times and the average value of wear weight loss was reported.

3. Results and discussion

Based on the ferritometry results (Table 1), which is sensitive to magnetic phases (particularly ferrite in stainless steels), the sintering temperatures of 1050, 1100, 1150, 1200, and 1250 °C develop non-

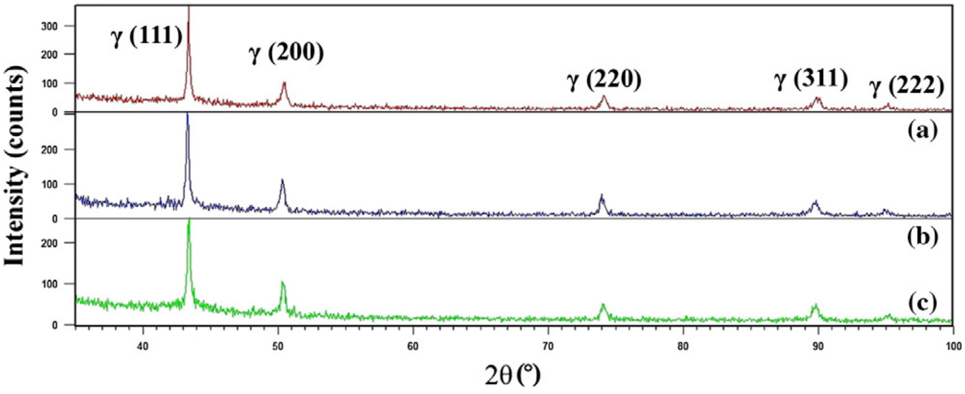

Fig. 2. XRD pattern of the samples sintered at 1000 (a), 1150 (b), and 1300 °C (c) (γ refers to austenite).