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Microstructure and mechanical properties of a new group of nanocrystalline medical-grade stainless steels prepared by powder metallurgy

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ABSTRACT

This paper focuses on the structure and mechanical properties of powder metallurgy stainless steels (Fe–Cr–Mn–Mo–Si–N–C) developed for biomedical applications. The samples were prepared by mechanical alloying and subsequent liquid-phase sintering with a eutectic Mn–Si alloy additive. By changing the sintering aid content, the pore configuration, compressive strengths, and impact properties of the samples were assessed. The Rietveld X-ray diffraction analysis showed after sintering at 1050 °C for 60 min followed by water-quenching, a nanocrystalline austenitic structure was formed in the material. According to the mechanical experiments, by increasing the additive content from 0 wt% to 6 wt%, sintering densification, yield stress, compression strength, and absorbed impact energy were improved, where spoiling occurred when adding 8 wt% additive. Also, as realized from the impact fracture surface features, despite the presence of some unmelted additive particles, the role of the pore elimination in toughness prevailed over that of these particles.

1. Introduction

Metals and alloys are the oldest materials used in surgical purposes to make devices for fracture fixation, joint replacement, external splints, braces, and traction apparatus, as well as dental amalgams [1]. Nowadays, the widely used metallic biomaterials include stainless steels, titanium and its alloys, cobalt-chromium-based alloys, as well as tantalum, niobium, and gold. Stainless steels, typically AISI 316L, are conventionally used in orthopedics, with the main advantages of low cost, good mechanical properties, sufficient corrosion resistance, and easy processing. However, problems have been found with this type of medical-grade stainless steels. The most important problem is the negative effect of metal ions or fretting debris released from the implant due to corrosion and wear. Nickel and chromium are known as potentially harmful elements in the medical stainless steels. Nickel ions act as allergens in the human body, which may cause inflammations like swelling, reddening, eczema, and itching on skins [2,3].

Due to the harmful effect of nickel ions on the human body, nickel-free austenitic stainless steels, generally Fe–Cr–Mn–Mo–N system, are being considered as a potential replacement for conventional nickel-containing alloys. Because of this, with the development of new surgical stainless steels and the modification of ASTM medical standards, the nickel content is decreasing and the nitrogen content is increasing. Currently, in ASTM standards, two nickel-free medical-grade stainless steels are imported: ASTM: F2229 and ASTM: F2581.

To produce nitrogen-containing nickel-free austenitic stainless steels, there are several methods, such as melting processes, solid nitrogen absorption treatment, and powder metallurgy. Powder metallurgy is a continually and rapidly growing technology which includes most metallic and alloy materials in a wide variety of shapes and applications. The high precision forming capability of powder metallurgy generates pieces with a near net shape and complex features, without the need of subsequent machining. In addition, the powder metallurgy process has a high degree of flexibility, allowing the tailoring of the physical characteristics of a product to suit specific properties and performance requirements. In this regard, mechanical alloying is a capable process to synthesize a wide variety of equilibrium and non-equilibrium structures, including supersaturated, metastable crystalline, quasicrystalline, intermetallic, nanostructured, and amorphous powders [4].

It is known that to meet the best mechanical and corrosion behaviors of powder metallurgy parts, high densities are imperative.

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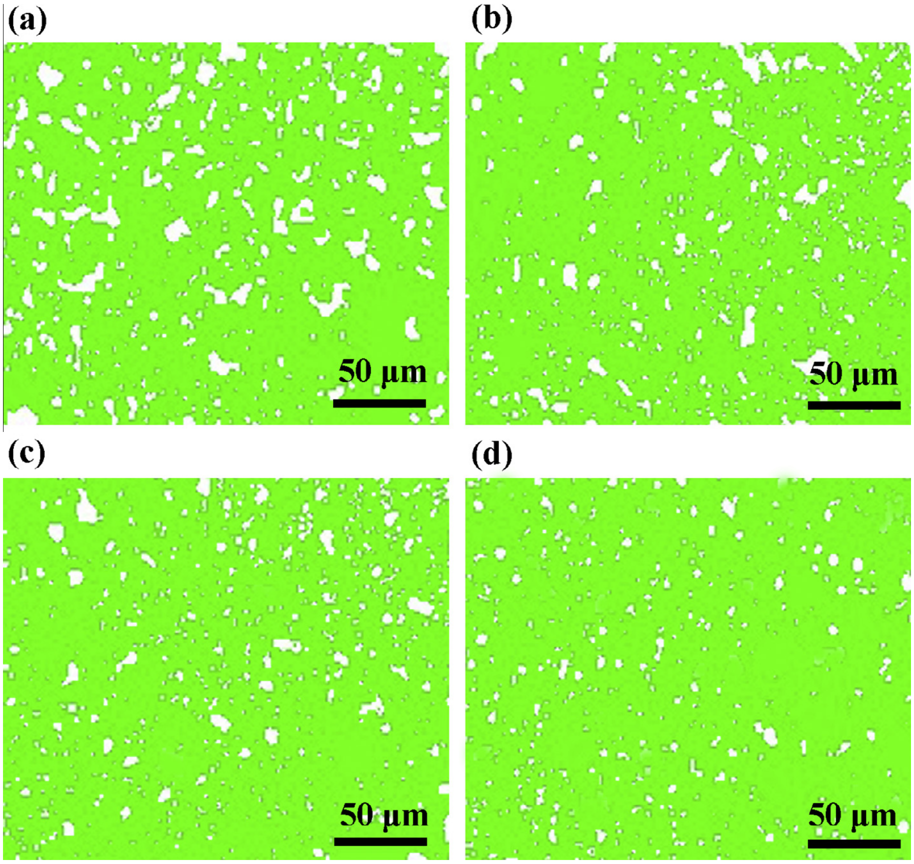

Fig. 1. Optical image analyzed photos of the sintered samples with (a) 0%, (b) 2%, (c) 4%, and (d) 6% additive.

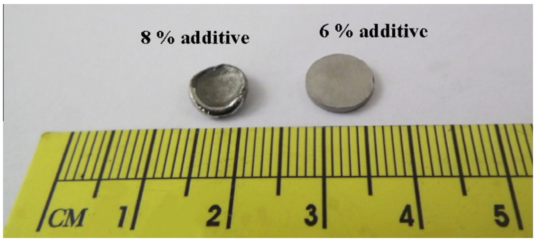

Fig. 2. Macroscopic photo of the samples after sintering.

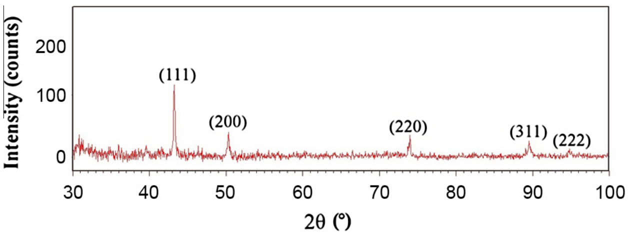

Fig. 3. XRD pattern of the sintered sample containing 6% additive.

To do so, a number of approaches like warm compaction, increasing sintering temperature and time, and using proper additives to activate liquid-phase sintering are under consideration. In the liquid-phase sintering process, the formation of a liquid phase promotes densification via providing a particle rearrangement, faster diffusion rate, and pore elimination [5]. Recently, mechanically-alloyed stainless steel powders with the nominal composition of ASTM: F2581 were liquid-phase sintered with a Mn–11.5 wt% Si additive [5–7]. Also, the suitable corrosion resistance and biocompatibility of this novel material were verified [8]. In this work, the effect of the additive amount on their mechanical properties is investigated by uniaxial compression and impact experiments.

2. Materials and methods

Mechanical alloying was used to synthesize stainless steel (with the composition of ASTM: F2581: Fe–17Cr–10Mn–3Mo–0.4Si–0.5N–0.2C in wt%) and sintering aid (Mn–11.5 wt% Si) powders. The samples with 0, 2, 4, and 6 wt% additive were sintered at 1050 °C for 60 min and were then quenched in water to obtain a single-phase austenitic structure. More details on the mechanical alloying and sintering parameters are available in Refs. [5–8].

To analyze the size and shape of pores in the sintered samples, optical microscopy was used. In order to determine the final microstructure, including the formed phases and crystallite sizes after sintering, X-ray diffraction (XRD, Shimadzu Lab X-6000, Cu Kα) analysis was used, where the results were interpreted by the Rietveld method using the Double-Voigt approach.

To study the uniaxial compressive behavior of the samples, according to ASTM: E9, cylindrical samples with the aspect ratio of 1.5 were used. The compression tests was done at a constant crosshead speed of 0.1 mm/s, with at least three replicates. Additionally, in order to compare the absorbed impact energy of the samples, Charpy impact tests on the stainless steel samples of 5 × 5 × 75 mm in size (ASTM: E23), were conducted. To do so, a sample containing 3 wt.% additive was also prepared and the tests were done on the specimens containing 0, 3, and 6 wt.% additive. Finally, fractured surfaces after the impact tests were studied by a scanning electron microscope, SEM.

3. Results and discussion

According to the Mn–Si phase diagram [9], the used sintering aid was a eutectic alloy with a melting point of 1040 °C. Obviously, at sintering temperatures below 1040 °C, no additive liquation occurs and solid-state sintering merely governs [5–7]. On the other hand, very high temperatures accelerate the undesirable phenomenon