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Effect of milling time on structure and mechanical properties of porous nickel-free austenitic stainless steels processed by mechanical alloying and sintering

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Abstract

In this paper, the microstructure and mechanical properties of biocompatible Cr–Mn–N austenitic stainless steels produced by powder metallurgy are investigated. Processing is conducted by mechanical alloying of 74Fe–18Cr–8Mn (wt.%) powder mixture under a nitrogen atmosphere, followed by sintering at 1100 °C for 20 h. The prepared materials are characterized by X-ray diffraction, scanning and transmission electron microscopy, suggesting the development of nanostructures with micron-sized spherical pores. It is also found that by increasing the milling time from 48 to 120 h, the densification is retarded; however, the hardness and compressive yield strength of the nanomaterials are enhanced.

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Moreover, the yield strength of matrix is estimated by valid empirical and plasticity-based relations.

Keywords: Ferrous alloy; Austenite; Powder metallurgy; Nanostructured materials; Porous materials; Mechanical characterization

1. Introduction

Recently, nitrogen-containing nickel-free austenitic stainless steels have attracted considerable attention, particularly in biomaterials field. Nitrogen is a strong austenite stabilizer and biocompatible element; hence, it is capable of being an appropriate replacement for nickel that is expensive and toxic in stainless steel biomaterials [1]. Additionally, nitrogen significantly improves mechanical properties [2–6] and corrosion resistance [6–8] of stainless steels. Despite the fact that the effect of dissolved nitrogen on austenite stabilization is several times higher than that of nickel, the nitrogen solubility is limited due to nitride precipitation. To increase the nitrogen solubility, manganese is usually used along with nitrogen [1], developing nickel-free Cr–Mn–N austenitic stainless steels.

In general, nitrogen-containing stainless steels are produced by complex liquid-state methods, especially pressurized electro-slag remelting under high pressures of nitrogen gas [1,9,10]; however, the nitrogen solubility in the liquid phase is limited. Because of this, solid-state processes like mechanical alloying (MA) have been regarded as an alternative to introduce nitrogen into the materials. It is notable that the generation of high densities of defects during MA increases the nitrogen solubility considerably. Nitrogen alloying through MA is accomplished by either milling under a nitrogen atmosphere or milling with proper

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nitrides under an inert gas. Furthermore, MA is a capable process to synthesize nanostructured materials presenting improved properties compared to conventional coarse-grained materials [11,12].

In the recent years, a good number of papers have been published on mechanical alloying of nickel-free stainless steel powders under a nitrogen atmosphere [13–20]. The effect of sintering time on densification and mechanical properties of mechanically alloyed 18Cr–8Mn–0.9N stainless steel has been investigated [21]. The wear and elastic behaviors of austenitic Cr–Mn–N stainless steels prepared by mechanical alloying and sintering have been also focused [22,23]. On the other hand, Cui et al. [24] have studied structure and mechanical properties of high-nitrogen nickel-free austenitic stainless steels with a relative density of 99 % fabricated by powder injection molding, conventional sintering, and solid-nitriding. The present paper aims to investigate the dependence of mechanical properties of porous 18Cr–8Mn–xN austenitic stainless steels developed by mechanical alloying and sintering upon milling time.

2. Experimental procedure

2.1. Sample preparation

Fe (>99.5%, $D_{av.} = 50 \mu\text{m}$), Cr (>99.9%, $D_{av.} = 150 \mu\text{m}$), and Mn (>99.9%, $D_{av.} = 50 \mu\text{m}$) powders supplied by Merck with a nominal composition of 74Fe–18Cr–8Mn (wt.%) were mechanically milled in a high-energy shaker mill. To introduce nitrogen in the powders, milling was conducted under a continuous flow of pure nitrogen gas for durations of 48, 72, 96, and 120 h. The other milling variables are similar to those reported in Ref. [13].

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The as-milled powders were uniaxially cold-pressed to cylinders at a compressive pressure of 1 GPa. To prevent oxidation during sintering and to preserve nitrogen in the structure, the compacts were encapsulated in quartz tubes under an evacuated condition (10^{-5} atm). A study conducted on the sintering behavior of the mechanically alloyed 18Cr–8Mn–0.9N stainless steel has showed that sintering at 1100 °C for 20 h leads to the maximum yield stress compared to other sintering durations [21]. Thus, the densification was performed by sintering at 1100 °C for 20 h and subsequently water quenching to room temperature to achieve an austenitic structure.

2.2. Materials characterization

The chemical composition of the as-milled powders was determined by a LECO gas analyzer (Corp., St. Joseph, MI) and X-ray fluorescence analyzer (XRF, Philips PW2400). The phase analysis was carried out by X-ray diffraction (XRD, Shimadzu Lab X-6000 with Cu K α radiation). The relative phase content and average crystallite size were identified by the Rietveld method and Double-Voigt approach, respectively. The amorphous phase amounts were also estimated by Rietveld analyzing the XRD data via the procedure detailed in Ref. [13]. Also, the validity of the XRD results was checked by transmission electron microscopy (TEM, JEOL-JEM 2010).

The density of the sintered samples was measured by the Archimedes water immersion method. The theoretical density of the as-milled powder particles was measured as 7.70 g/cm³ by a pycnometer using He gas. The resultant microstructure after sintering was studied by XRD, TEM, and scanning electron microscopy (SEM, JEOL-JSM 5310). For the TEM observations, the selected sintered samples were first cut into discs of 3 mm diameter,

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manually ground to about 35 μm thickness, and then ion-milled at low temperatures. In addition, to assess probable nitrogen loss due to the consolidation process, the LECO analysis was again conducted.

2.2. Mechanical experiments

The Vickers microhardness at pore-free zones and the Vickers bulk hardness were measured on 10 points by applying 50 g and 31.25 kg loads, respectively and the average values are reported. To determine the yield strength of the specimens (with a diameter of 5 mm and a height of 5 mm), uniaxial compression tests were carried out at room temperature with a crosshead speed of 2×10^{-5} m/s using a universal testing machine. To reduce friction at the loading faces, the samples were lubricated with a special grease. At least five replicates were done for each specimen and the average value is reported. Finally, the fracture surfaces after the compression tests were observed by SEM to determine the fracture mechanism.

3. Results and discussion

The chemical composition of the as-milled powders is listed in Table 1, conducted by LECO and XRF. It is seen that the only detectable impurity is oxygen. It is attributed to the natural oxidation of the powders in the atmosphere, which is insignificant and the same for all the samples. Nonetheless, the nitrogen content increases progressively with the milling time due to a solid-gas reaction, approaching 2.13 wt.% after 120 h of milling. It is believed that the chemisorption of nitrogen onto the clean surfaces created by milling is the essential step governing the solid-gas reaction [13,25,26].

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Fig. 1 shows the XRD pattern of the powders milled for 48, 72, 96, and 120 h.

According to the Rietveld analyses, three different phases, namely ferrite (α), austenite (γ), and an amorphous phase, are recognizable. The results of the XRD analyses, consisting of the relative phase amount and crystallites sizes, are summarized in Table 2. It is observed that by increasing the milling time, the amorphous phase content increases. The amorphization reaction can be argued in terms of the high energy given to the powders during milling and the contribution of nitrogen to the increase in the atomic size mismatch and heat of mixing [13–16]. In addition, the nanocrystallization can be explained by severe plastic deformation subjected to the powder particles in the course of mechanical milling [11,13]. It would be worth mentioning that the XRD results were compatible with the TEM observations.

The XRD pattern of the specimens sintered at 1100 °C for 20 h is presented in Fig. 2. The analysis of the XRD results suggests that fully austenitic (γ) structures without any nitrides and ferrite are obtained after the densification process. It is well known that the microstructure of nitrogen-containing stainless steels depends on their nitrogen concentration and final heat treatment [17–19]. Since the austenite phase has larger interstitial sites and smaller interfacial energy compared to the ferrite phase [13], nitrogen that is interstitially dissolved in Fe-based alloys promotes austenitization. On the other hand, in spite of the considerable concentrations of nitrogen, no nitride has precipitated, due to the enhanced nitrogen solubility resulted from the fine structures. Indeed, the XRD results verify that water quenching from 1100 °C are an appropriate heat treatment to develop an austenitic structure for the studied materials. Note that water quenching from 1100 °C prohibits the onset of diffusional phase transformations.

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The crystallite sizes determined by the XRD analyses and the relative density of the sintered samples are summarized in Table 3. Typically, the TEM micrograph and related selected area diffraction (SAD) pattern of the sintered sample containing 0.9 wt.% nitrogen is displayed in Fig. 3, confirming the validity of the XRD results. Note that the SAD pattern clearly suggests the nanostructured austenite phase. It is noticeable that even after sintering, the nanometric structure of the material is retained. The sluggish grain growth is attributed to the segregation of nitrogen atoms to grain boundaries and the retarded crystallization of the amorphous phase existing in the as-milled powders. Since the solubility of nitrogen is limited in the crystalline structures, nitrogen atoms tend to segregate toward grain boundaries to decrease strain energy. The accumulation of considerable nitrogen contents at grain boundaries inhibits grain growth by decreasing grain boundary mobility [21–23]. It is also noted that the LECO analyses on the sintered samples revealed that the nitrogen loss during sintering is negligible (less than 0.03 wt.%), attributed to the small internal volume of the sealed quartz capsules. Preserved nitrogen increases the stability of the amorphous phase, retarding its crystallization and affecting the resultant grain size. It should be considered that by increasing the milling time, the amount and stability of the amorphous phase synthesized by MA increase [13–16].

It is observed that by increasing the milling time, the obtained relative density decreases. Firstly, it should be considered that the green densities were measured as 69, 68.5, 68.1, and 67.6 % for the samples milled for 48, 72, 96, and 120 h, respectively. It is well known that the efficiency of compaction is heavily dependent on the hardness of powder particles. Since the amorphous phase content increases by increasing the milling time, the hardness and accordingly resistance to plastic deformation during compaction are enhanced.

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It dictates a decrease in the compressibility of the powders, leading to less green densities followed by a worse densification during sintering. Secondly, it is anticipated that by progression of MA, the distribution of grain and powder particle sizes becomes more narrow, thereby retarding the densification progress. On the other hand, since nitrogen atoms occupy interstitial sites of the amorphous and crystalline phases, an increase in the atomic packing of the structure is realistic by increasing the milling time. It contributes to a decrease in the atomic diffusion coefficients, retarding the densification progress accomplished by diffusion.

Fig. 4 depicts the SEM micrograph taken from the polished specimen containing 0.9 wt.% nitrogen, showing spherical porosities exceeding 10 μm in size. As it is clear, the primary powder particles are not recognizable, implying that a tolerable necking is created between the particles. The presence of porosities is recognized to be suitable for some applications like biomaterials. Porous biomaterials with sufficient mechanical properties are recognized as desired bone implants. The porous implants provide a better fixation to the bone host, via the growth of new bone tissue into pore spaces. Moreover, introducing pores into stainless steel parts results in a decrease in the mismatch of elastic moduli of the implant and surrounding bone, thereby improving the fixation [27]. There are several reports in the literature on the fabrication of porous austenitic stainless steel biomaterials, for instance by MA and conventional sintering [21], selective laser sintering [27], and continuous zone melting under pressurized gases [28].

The mechanical properties of the sintered samples consisting of the microhardness, bulk hardness and yield stress are tabulated in Table 4. It is seen the microhardness of the sintered material, which is not affected by pores in contrast to the bulk hardness and yield stress, is considerable values. It is due to the fine structure and high nitrogen content of the

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materials. It is well established that nanostructured materials exhibit higher hardnesses and strengths compared to coarse-grained materials, as expected from the Hall-Petch equation. Moreover, it has been found that the nitrogen addition to austenitic stainless steels enhances their hardness and strengths. Nitrogen in austenitic stainless steels is an effective element not only in solid solution strengthening but also in grain size strengthening [29]. In other words, the nitrogen addition to austenitic stainless steels increases both the friction stress and Hall-Petch slope. The increase in all the mechanical properties listed in Table 4 by increasing the milling time is also attributed to the effect of nitrogen. Note that as tabulated in Table 3, the grain sizes are a narrow range and cannot only justify the variations in the mechanical properties with the milling time. It can be inferred that here the effect of the nitrogen addition on the bulk hardness and yield stress prevails over the contradictory effect of the relative density shown in Table 3. It has been reported that micron-grained Fe-23Cr-2Mo-1N and Fe-25Cr-1N austenitic stainless steels having 45 % Lotus-type pores show the compressive strengths of 270 and 290 MPa with pores aligned parallel to the compression direction and those of 90 and 180 with pores aligned perpendicular to the compression direction, respectively [28]. In addition, porous coarse-grained biomedical 316L stainless steels with the porosity range of 40 to 50 % fabricated by selective laser sintering have shown compressive yield stresses of 21 to 32 MPa [27].

Obviously, the yield strength and bulk hardness of porous materials depend not only on those of matrix but also on the porosity. There are several empirical equations and theoretical models predicting the room temperature strength of austenitic stainless steels. A number of them correlate the yield stress with the chemical composition and grain size of stainless

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steels, including for nickel-free austenitic stainless steels. The following relations can estimate the yield strength of matrix:

$$YS = 257.69 (\text{wt. \% C}) + 479.49 (\text{wt. \% N}) + 5.74 (\text{wt. \% Mn}) + 1.46 (\text{wt. \% Cr}) + 92.58 (\text{wt. \% Si}) - 449.09 (\text{wt. \% P}) + 7354.36 (\text{wt. \% S}) - 0.00107 D + 235.17 \quad [10] \quad (1)$$

$$YS = 145 \sqrt{1 + 15 (\text{wt. \% N})} + \{8 + 38 (\text{wt. \% N})\} / \sqrt{D} \quad [30] \quad (2)$$

$$YS = 127 + 307 \sqrt{(\text{wt. \% N})} + \{7 + 78 (\text{wt. \% N})\} / \sqrt{D} \quad [31] \quad (3)$$

where the yield strength YS is given in MPa and the grain size D is given in nm.

On the other hand, plasticity equations have proposed to account for the effect of porosity on the yield stress of austenitic stainless steels. For uniaxial compression tests, the following equations can be applied [32]:

$$YS_p = \sqrt{\frac{(1 - \Phi)^2}{1 + \Phi/4}} YS \quad (4)$$

$$YS_p = \sqrt{\frac{3(1 - \Phi^{1/3})^2}{3 - 2\Phi^{1/4}}} YS \quad (5)$$

$$YS_p = \sqrt{\frac{3(1 - \Phi)^5}{3 + 2.07\Phi^{1.028}}} YS \quad (6)$$

$$YS_p = \sqrt{2(1 - \Phi)^2 - 1} YS \quad (7)$$

where YS is the yield stress of matrix, YS_p is the yield stress of porous samples, and Φ is the porosity fraction. In the literature, other equations are found to estimate the 0.2 % proof stress of Fe–Cr–Mn–N austenitic stainless steels as a function of only nitrogen content, for example that reported in Ref. [1]. However, since nanometric structures including the structure of the materials studied here affect the strength significantly, equations ignoring the effect of grain size are not considered in this study.

Inserting the chemical compositions and grain sizes listed in Tables 1 and 3 in Eqs. 1, 2, and 3 and the relative densities and the yield strengths of the sintered samples tabulated in

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Tables 3 and 4 in Eqs. 4, 5, 6, and 7, the yield stress of matrix is estimated as shown in Fig. 5. As justified above, the yield stresses are considerable values and progressively increase with the milling time. It is also seen that Eq. 1 accounting for the entire composition and grain size of the alloys presents overestimates for the yield stress of the studied materials; in contrast, Eq. 4 considering only the relative densities presents underestimates. In addition, the yield strengths calculated by Eqs. 3, 5, 6, and 7 are in good agreement with each other compared to the other relations. It is notable that micron-grained Fe-17Cr-11Mn-3Mo-0.78N austenitic stainless steel produced by powder injection molding, conventional sintering, and solid-nitriding, having 99 % relative density, presents a yield stress of 580 MPa and hardness of 222 Hv [24]. For Ni-free austenitic stainless steels containing 0.82 wt.% nitrogen developed by casting under a nitrogen atmosphere, the Hall-Petch equations $YS = 507.6 + 11.7 D^{-1/2}$ and $Hv = 253.5 + 6.7 D^{-1/2}$ have been established for yield stress and Vickers hardness, respectively [33], which is relatively comparable with the results obtained in this work.

The secondary electron micrograph of the fracture morphology of the Fe-18Cr-8Mn-0.9N sample after the compression test is provided in Fig. 6 in two magnifications. The fracture mechanism of microvoid coalescence is observed in the picture in which almost all the particles are discretely apparent. A large number of the particles and considerable particle boundary cracking are recognizable in the fracture surface. This mechanism is in good agreement with the relative density of the materials, demonstrating the domination of the role of pores.

4. Concluding remarks

The study is summarized as following:

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1. Nitrogen alloying of the Fe–18Cr–8Mn powders was accomplished via mechanical milling under a nitrogen atmosphere, due to a solid–gas reaction governed by the chemisorption of nitrogen onto the clean surfaces created by milling.

2. The structure of the as-milled powders consists of an amorphous phase, ferrite and austenite nanostructures, revealed by XRD and TEM evaluations.

3. The densification process at 1100 °C for 20 h followed by water quenching provided porous fully austenitic nanostructures with micron-sized spherical pores, which is promising for biomaterial applications.

4. The obtained relative density decreased by increasing the milling time.

5. The mechanical properties of the consolidated materials were enhanced by increasing the milling time.

6. The yield strength of matrix was estimated by valid equations based on empirical and theoretical models.

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Figures:

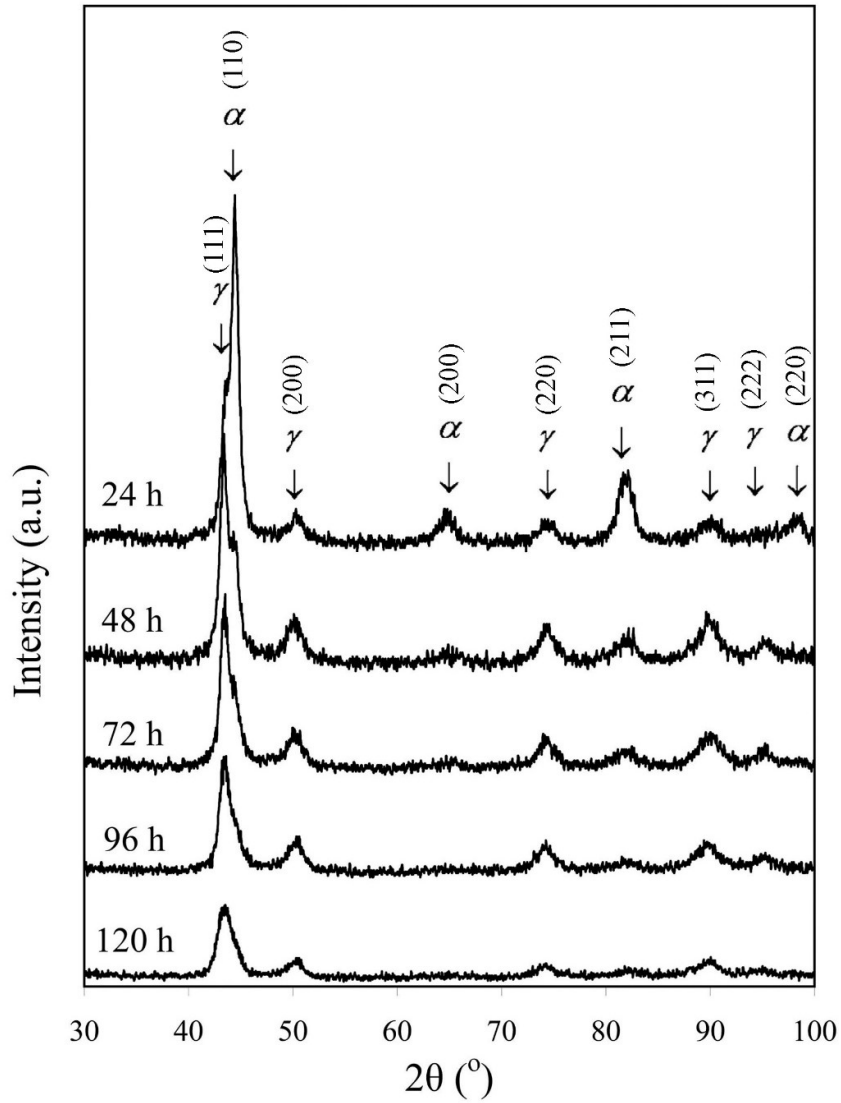


Fig. 1. X-ray diffraction trace of the powders milled for the different durations.

This is the accepted manuscript (postprint) of the following article:

E. Salahinejad, R. Amini, M. Hadianfard, *Effect of milling time on structure and mechanical properties of porous nickel-free austenitic stainless steels processed by mechanical alloying and sintering*, Materials Science and Engineering: A, 527 (2010) 5522-5527.
<https://doi.org/10.1016/j.msea.2010.05.008>

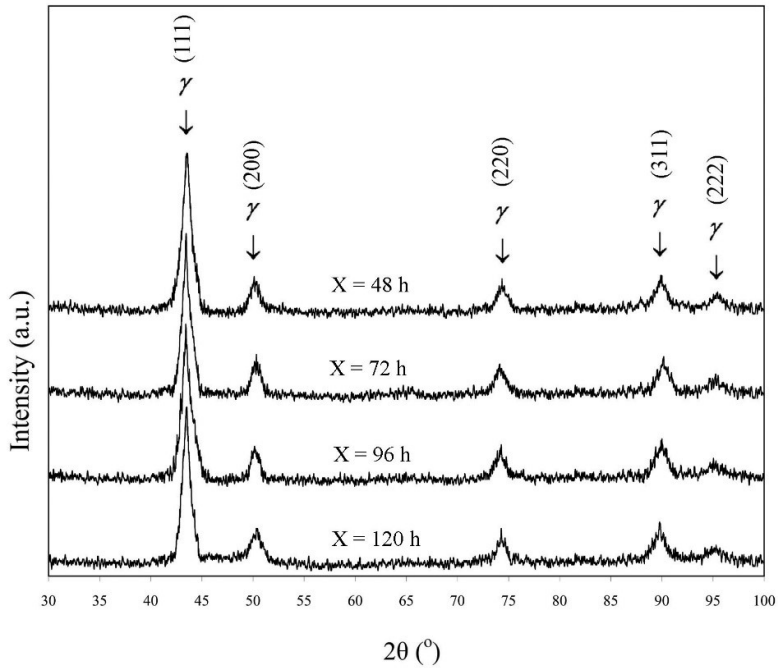


Fig. 2. X-ray diffraction pattern of the specimens sintered at 1100 °C for 20 h (X = milling time).

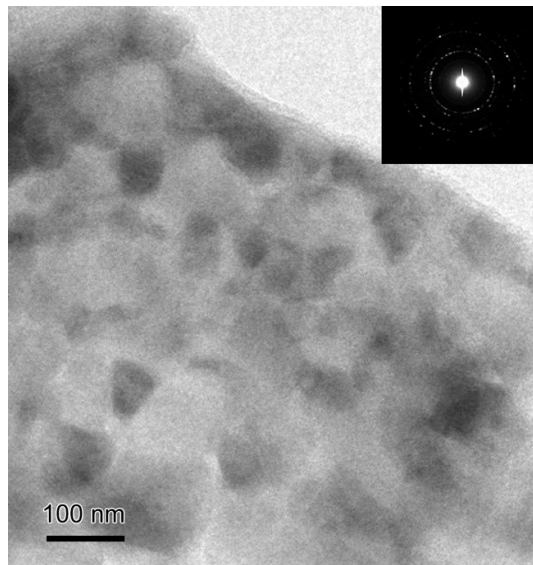


Fig. 3. Transmission electron microscopy micrograph and selected area diffraction pattern of the sintered Fe-18Cr-8Mn-0.9N sample.

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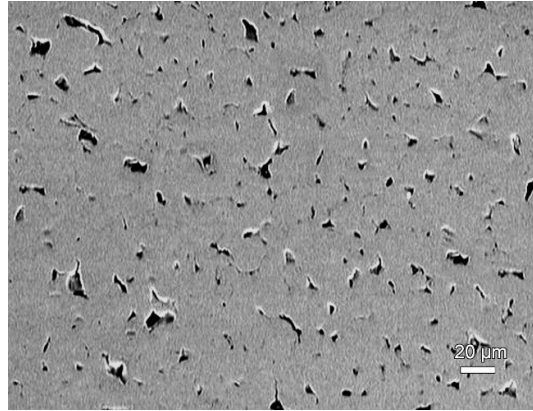


Fig. 4. Scanning electron microscopy micrograph of the sintered Fe-18Cr-8Mn-0.9N specimen.

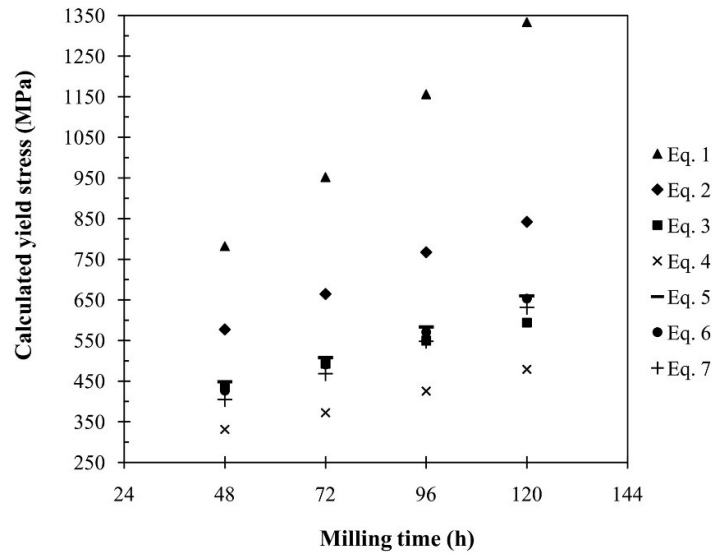


Fig. 5. Yield stress of the sintered materials calculated from the equations vs. the milling time.

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E. Salahinejad, R. Amini, M. Hadianfard, *Effect of milling time on structure and mechanical properties of porous nickel-free austenitic stainless steels processed by mechanical alloying and sintering*, *Materials Science and Engineering: A*, 527 (2010) 5522-5527.

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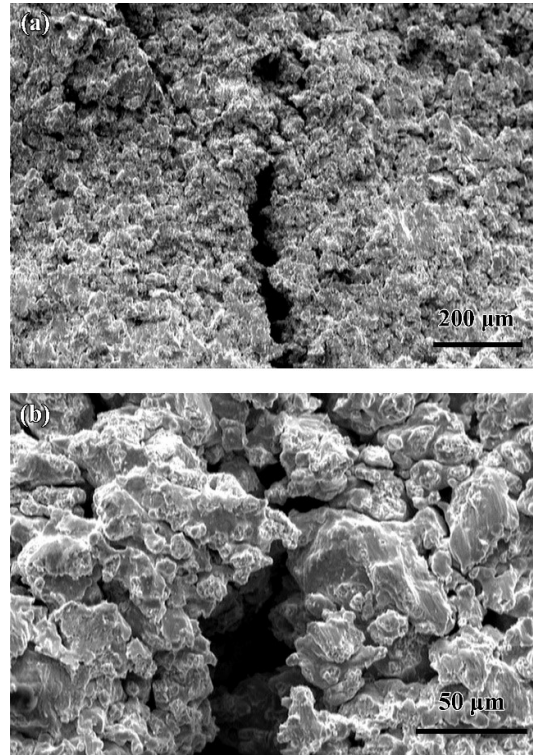


Fig. 6. Secondary electron microscopy micrograph of the fracture morphology of the Fe–18Cr–8Mn–0.9N specimen in two magnifications.

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Tables:

Table 1. Chemical composition of the as-milled powders (wt.%)

Milling time (h)	Fe	Cr	Mn	N	O	C
48	72.948	17.643	8.069	0.973	0.334	0.033
72	72.971	17.505	7.827	1.330	0.334	0.033
96	72.763	17.408	7.705	1.760	0.335	0.029
120	72.530	17.350	7.629	2.130	0.331	0.030

Table 2. X-ray diffraction results of the as-milled powders, consisting of the relative phase amount and crystallites sizes

Milling time (h)	α -phase percentage	γ -phase percentage	Amorphous percentage	α -crystallite size (nm)	γ -crystallite size (nm)
48	22.9	37.8	39.3	14.8	11.0
72	17.7	38.6	43.7	10.0	8.1
96	11.7	33.9	54.4	7.8	6.2
120	5.3	27.3	67.4	7.0	5.1

Table 3. Austenite crystallite size and relative density of the sintered samples

Milling time (h)	γ -crystallite size (nm)	Relative density (%)
48	90	86.0
72	88	84.8
96	87	83.9
120	85	83.1

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Table 4. Mechanical properties of the consolidated samples

Milling time (h)	Microhardness (Hv)	Hardness (Hv)	Yield stress (MPa)
48	345	200	280
72	372	206	310
96	413	211	350
120	495	221	390