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RESEARCH ARTICLE

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Influence of Niobium additions on sintering behaviors and mechanical properties of injection molded 420 stainless steel powder

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ABSTRACT

This paper describes the sintering of an injection molded 420 martensitic stainless steel with additions of niobium, with the aim of producing high mechanical properties. And at the same time, microstructural and mechanical characterization of these produced parts was also carried out. At the initial stage, 420 martensitic stainless-steel powders were mixed with a multi-component binder system for preparing feedstock. Then the prepared feedstock was granulated and shaped by injection molding. And then, the shaped samples were subjected to the debinding process. These samples were sintered at different temperatures for various times. Samples sintered under the condition that gave way to the highest relative density were heat treated. Sintered and heat-treated samples were separately subjected to microstructural and mechanical characterization. All analysis showed that using polymeric binder system led to plentiful martensite ratio and carbide precipitates to be occurred in the injection molded samples. Mechanical characterization was performed by hardness measurements and tensile tests.

1. INTRODUCTION

Powder injection molding (PIM) is a powder metallurgy process currently used for the production of complicated and near-net-shape parts of high-performance materials. This technique basically combines the advantages of the plastic injection molding with the versatility of the traditional powder metallurgy, producing highly complex part of small size, tight tolerance, and low production cost. The process overcomes the shape limitation of traditional powder compaction, the cost of machining, the productivity limits of isostatic pressing and slip casting, and the defect and tolerance limitations of conventional casting [1-4]. Mechanical properties of a well-processed powder injection molded material and indistinguishable from cast and wrought material. The PIM process is composed of four sequential steps; mixing of the powder and organic binder, injection molding, debinding (binder removal), and sintering. If it is necessary, secondary operations such as heat and surface treatments after sintering can be performed [1-2].

AISI 420 stainless steel has high chromium (12 to 14%) and medium carbon content (> 0.15%) in its chemical composition [5]. Following heat treatment, the material has useful properties including high strength, hardness and corrosion resistance [6], making it a suitable choice for wear applications [7]. Although there are some experimental studies on PIM of 420 stainless steels, more detailed information is not available in literature. In particular, the effect of the niobium element on 420 stainless steels is unknown.

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Previous works on PIM of 420 stainless steel less discussed mechanical properties and microstructure [8, 9].

Nb has a higher affinity for C than Cr. In low C alloy steels, the addition of Nb has been reported to improve the mechanical properties [8-10]. It was reported that, in presence of Nb, yield strength of high strength low alloy (HSLA) steel increased from 700 to 780 MPa. Nb can also reduce the hardenability of steel because it forms very stable carbides [11], thereby reducing the 4 amounts of C dissolved in the austenite during heat treatment. Another role of Nb in cast and wrought stainless steels is as a stabilizing agent to reduce the tendency to undergo intergranular corrosion. Though 420 stainless steel possessed good corrosion resistance in the heat-treated condition, which depletes the Cr content in solution near the grain boundaries and thus reduces the intergranular corrosion resistance significantly [12-15].

This present work was aimed to investigate the effect of Nb on the sintering behavior and the final properties of powder injection molded 420 stainless steel. Metallographic techniques were employed to sintered tensile bars to investigate the sintering behaviors. Tensile, hardness and corrosion properties of the sintered products were evaluated in heat-treated condition. Powder morphology, molded, debinded, sintered and heat-treated samples were analyzed under scanning electron microscope.

2. EXPERIMENTAL PROCEDURES

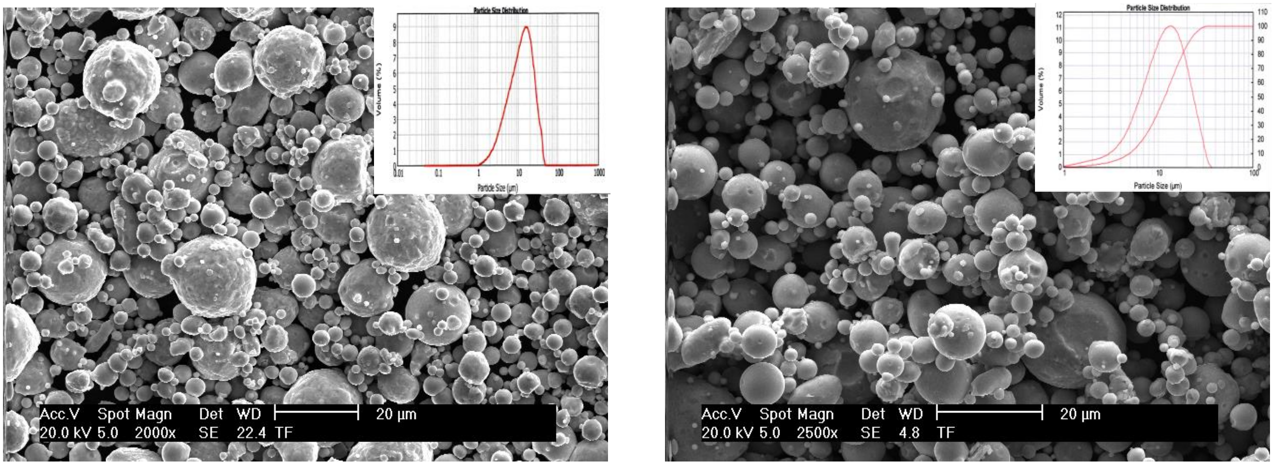
The powder chemical properties and characteristics of gas atomized 420 and 420+Nb stainless steel powders used in this study are given Table 1 and 2. Particle size distributions indicate similar median particle sizes for two type powders. Morphology of the powders, observed using scanning electron microscopy are given Fig. 1(a) and (b). All powders are spherical in shape.

Table 1. Chemical composition of 420 and 420+Nb stainless steel powders.

	Elements (wt.%)											
	Fe	Nb	Mo	Cr	Ni	Mn	Si	P	C	S	O	N
AISI Standart	Bal.	--	--	12-14	--	<1.0	<1.0	<0.04	>0.15	<0.03	--	--
420	Bal.	NA	NA	12.8	NA	0.72	0.79	0.012	0.3	0.01	0.04	0.09
420+Nb	Bal.	1.0	0.56	12.0	0.65	0.9	0.9	0	0.3	0	NA	NA

Table 2. Powder characteristics of 420 and 420+Nb stainless steel powders.

Item	420 SS	420SS + Nb
Vendor	Osprey Co.	Osprey Co.
Production method	Gas atomized, N	Gas atomized, N
Shape	Spherical	Spherical
Particle size (μm)		
D_{10}	4.30	5.07
D_{50}	12.55	11.78
D_{90}	26.64	21.80
Tap density, g/cm^3	4.70	4.90



(a) (b)

Figure 1. Scanning electron micrograph of 420 and 420+Nb stainless steel powder (a) 420 and (b) 420+Nb.

Paraffin wax (PW), carnauba wax (CW), polypropylene (PP) and stearic acid (SA) were used as multi-component binder system to prepare the feedstocks. A multiple-component binder system consisting of PW (69 wt%), PP (10 wt%), CW (20 wt%) and SA (1 wt%) was employed. In order to observe the temperature on rheological behavior of feedstocks. Powder was prepared in formulations containing as 62.5 % vol. Feedstocks were prepared by means of blade mixer. Feedstocks for each volume of component were separately prepared at 180 °C throughout obtaining homogeneous mixing separately. Cooled feedstocks were granulated and then denominated for 62.5 % vol. Rheological behaviour of the feedstocks was measured using a rotational Rheometer Physica MCR51 (Anton Paar. Austria) at shear rates from 10 to 1.000 s-1 at temperatures 120-170°C. The value of viscosity is given by the shear stress divided by the shear rate.

After cooling, the feedstock was pelletized by hand. These feedstocks were injected using a 12.5 MPa specially made injection-molding machine to produce standard tensile test specimens. During which the melt temperature of 180 ºC, the mold temperature of 35 ºC and cycle time of 20 second were used to produce green tensile samples. Debinding was conducted in a two-step solvent/thermal operation. Green parts were solvent debound at 60 ºC for 4 h in hexane, followed by thermal debinding step at 1.8 ºC/min to 600 ºC for 1 h and pre-sintered at 4 ºC/min to 900 ºC for 1 h in pure Ar. The samples were sintered in an atmosphere controlled vertical recrystallized alumina tube furnace. The sintering cycle applied to the samples was as follows; samples were heated to 1200 ºC at a rate of 10 ºC/min and held at 1200 ºC for 5 min., then the samples were heated to various sintering temperatures of 1250 ºC, 1300 ºC and 1350 ºC at a rate 5 ºC/min and they were held at each temperature for 1 hour in Ar.

Densities of the sintered tensile bars were measured by the Archimedes method. The samples were cut from the tensile bars, mounted, ground, and polished to a 0.3 μm and 0.5 μm surface finishing using standard metallographic procedures. A Kalling's reagent was used to etch the samples for optical metallography. Finally, the sintered tensile bars were heat-treated the following two steps. The heat treatment consisted of a solution treatment in argon for 1 hour at 1030ºC, followed by an air quench and aging treatment for 1 hours at 440ºC with a cooled in air. All tensile tests were performed using Zwick-Z250 mechanical tester at constant crosshead speed of 25.4 mm/min (25 mm gauge length). The hardness tests were performed using an Instron-Wolpert Dia Testor 7551 at HRC scale. An average of five values of all mechanical measurements was reported. The fractures were examined using a scanning electron microscope (FEI-Srion).

2. RESULT and DISCUSSION

The evaluation of the feedstock rheological properties is based on the viscosity and its shear rate, temperature and powder type sensitivity. The lower the value of the viscosity, the easier it is for a feedstock to flow. It can be found from Fig. 2. that the viscosity decreases at all shear rates. With the increase of temperature, viscosity of feedstock decreases for two types powder. PIM feedstock is conducted under pressure and temperature. It is desirable that the viscosity of the feedstock should decrease quickly with increasing shear rate during molding. This high shear sensitivity is especially important in producing complex parts. In addition, the surfaces of the 420+Nb powders are smoother. Therefore, they are better connected with binders. As a natural consequence, the viscosity values were lower compare to 420 stainless steel powder. The results indicate that the feedstocks possess pseudo-plastic rheological behavior and increasing temperature leads to a decrease in the viscosity.

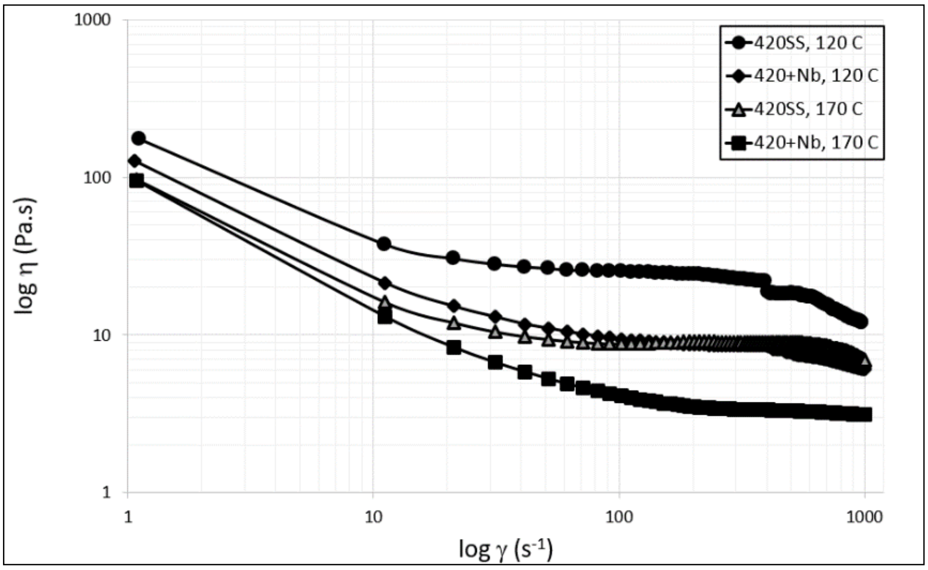


Figure 2. Temperature-dependent viscosity versus shear rate of different type 420 and 420+Nb stainless steel powder.

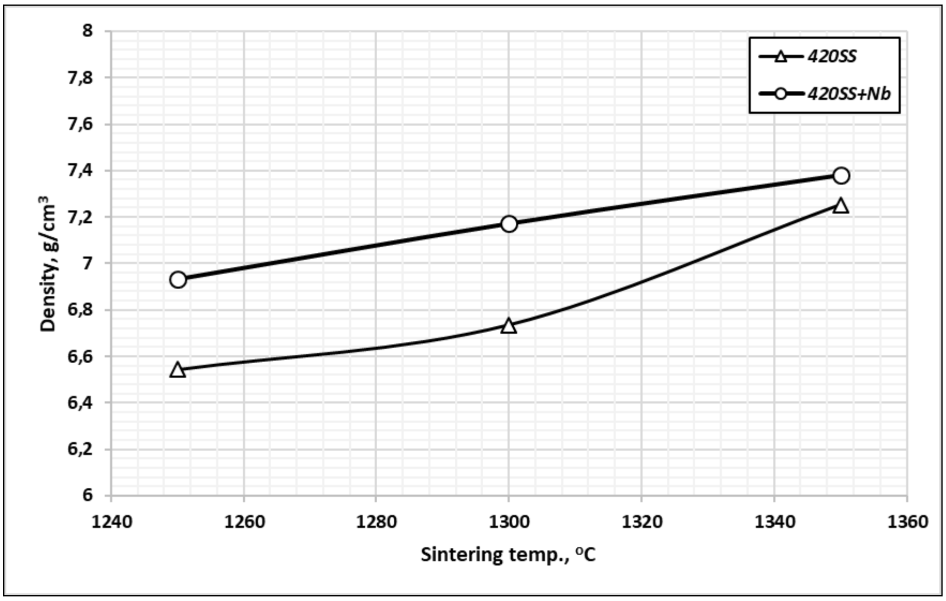


Figure 3. Sintered density of the samples sintered at different temperatures

Fig. 3 shows the change in sintered density values for two different types of 420 stainless steel powder depending on the increasing sintering temperature. In both types of powder, sintered densities of samples increased depending on the increasing sintering temperature. Compared to sintering densities at all sintering temperatures, 420 stainless steel samples showed lower density values than 420+Nb stainless steel samples. The sintered densities of the samples sintered were 6.54 g/cm^3 for 420 stainless steel samples and 6.93 g/cm^3 for 420+Nb stainless steel samples at 1250 ºC, respectively. With increasing sintering temperature, sintered densities reached to 7.25 g/cm^3 for 420 samples and 7.38 g/cm^3 for 420+Nb samples at 1350 ºC, respectively. In general, increased sintering temperature and Nb addition has improved sintering behavior.

Metallographic analysis was carried out on samples from each type samples for different sintering temperatures. Images of the polished microstructures for two types samples are shown in Fig. 4. At lower sintering temperatures, polished surfaces were present more pores. But at higher sintering temperatures, the pore quantity is low. This is a natural result of increased sintering temperature. At the same time, when Nb addition and non-added samples were compared, it was found that there was less pores in Nb added samples. Images of the etched samples microstructures for two types samples are shown in Fig. 5. The microstructure showed needle-liked structures that were dispersed throughout the etched microstructure representing a martensitic structure in the heat-treated conditions. The microstructures exhibited by the two materials types are quite different. The 420SS without Nb displays predominantly intergranular eutectic precipitation surrounding large grains with little intragranular carbide. The 420SS containing 1%Nb exhibits a much finer and uniform grain size with extensive intragranular precipitation of NbC. Finally, the Nb alloy-based samples shows residual porosity, intermediate grain size and a lower concentration of intragranular NbC precipitation reflecting its lower Nb content. Martensite forms through austenite-martensite transformation in the range from 700 to 300°C when the cooling is rapid.

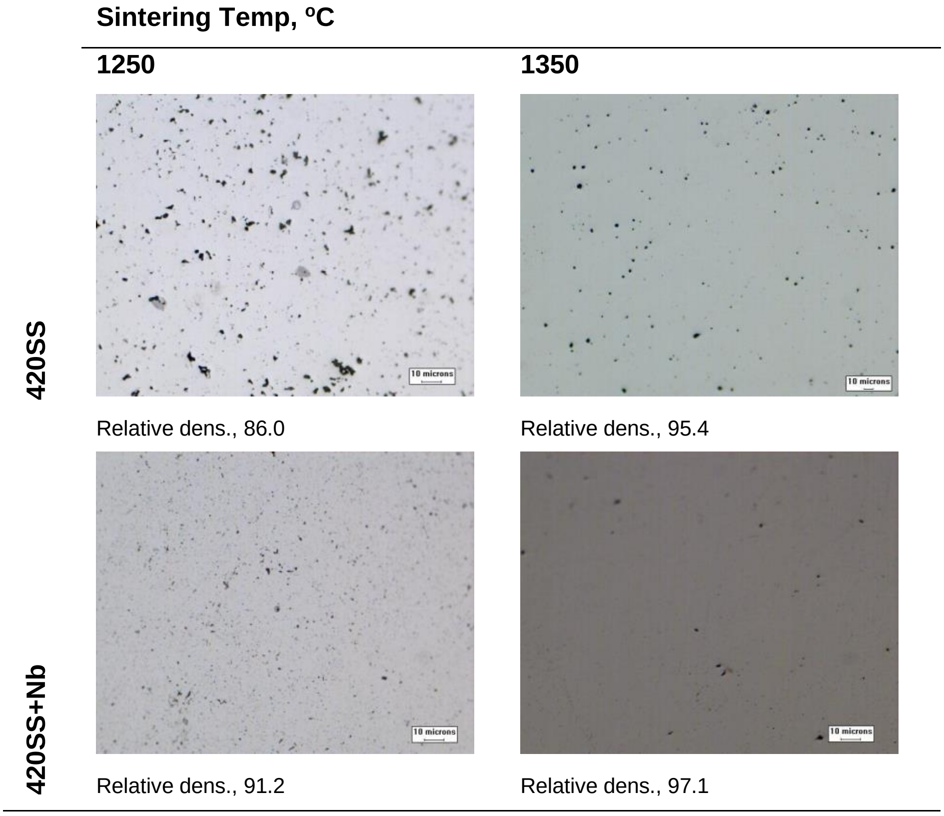


Figure 4. Microstructures of as-polished samples sintered at 1250 ºC and 1350 ºC for 1 h.

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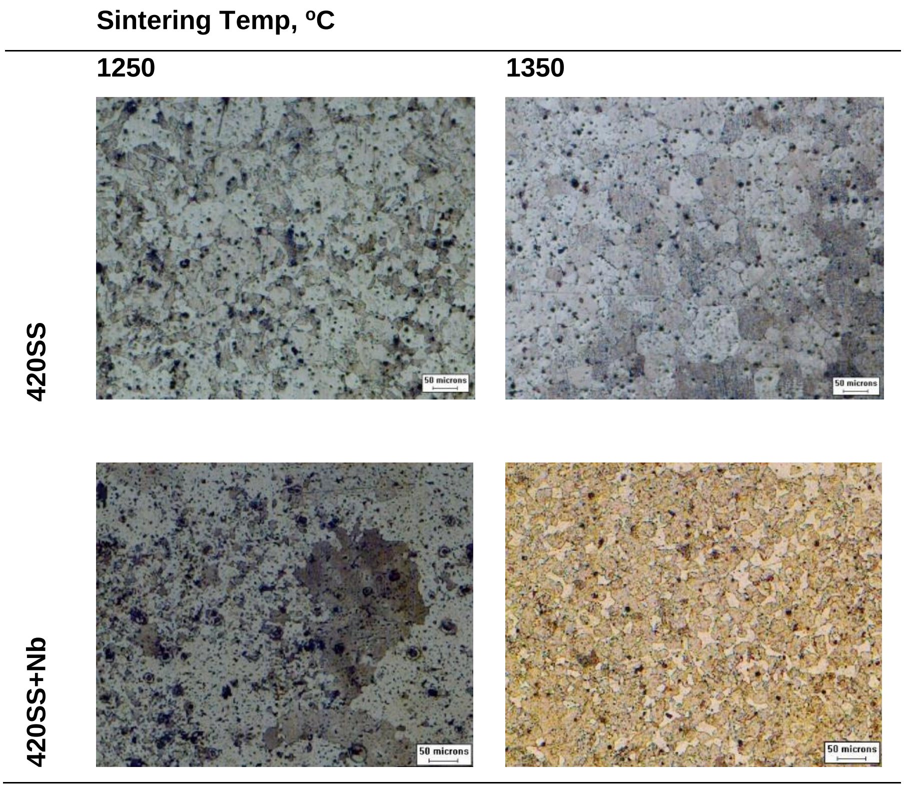


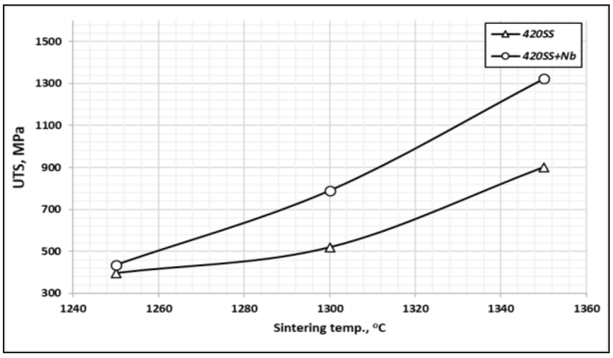
Figure 5. Microstructures of as-ethected samples sintered at 1250 ºC and 1350 ºC for 1 h.

Table 2. Mechanical properties of sintered and heat treated 420 and 420+Nb stainless steel samples

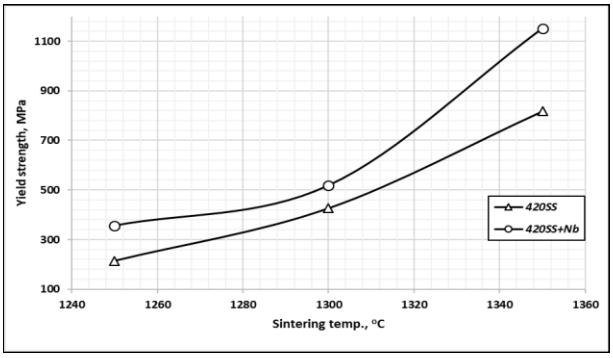
Sample (at 1350 °C-1 h.)	**Sintered density gr/cm^3**	**Ultimate tensile strength MPa**	**Yield strength MPa**	**Elongation %**	**Hardness HRC**
420 SS	7.25	901	817	1.6	47.2
420+Nb SS	7.38	1323	1152	2.9	49.3
420 (MPIF 35) [12]	7.40	1380	1200	<1	44.0

Microstructures of sintered samples shows tempered martensite in all cases with fine chromium and niobium carbides dispersed throughout the matrix. The carbides formed at the grain and grain boundaries caused an increase in mechanical properties.

The mechanical properties of the samples that were processed under different sintering temperatures are shown in Table 2. Graphs of comparison of mechanical properties results were shown in Fig. 6. The effect of Nb additions on the ultimate tensile strength, yield strength, elongation and hardness of 420 and 420+Nb stainless steels in sintering temperature at 1250-1350 ºC are shown in Fig. 6. Ultimate tensile strength, yield strength, elongation and hardness increase with Nb additions and sintering temperature. The maximum ultimate tensile strength of 1323 MPa, yield strength of 1152 MPa, elongation of 2.9 and hardness of 49.3 HRC was reached with samples added Nb sintered at 1350 ºC for 1 h. Depending on the increase in sintering temperature, grain size increased. At the same time, chromium and niobium carbides were formed at high sintering temperatures. Due to grain size increase and carbide formation, mainly sintered densities and mechanical properties increased.



(a)



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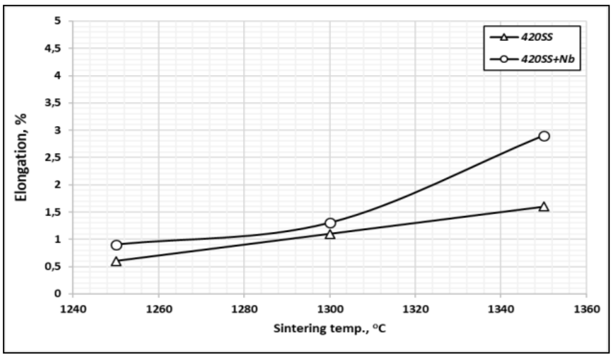

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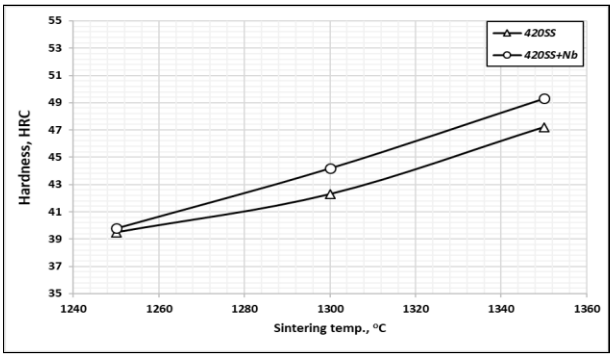

(d)

Figure 6. Effect of sintering temperature and Nb addition of mechanical properties of 420 stainless steel. (a) Ultimate tensile strength, (b) Yield strength, (c) Elongation, (d) Hardness.

More importantly, the formation of martensite phase is the main reason for the increase in mechanical properties. The mechanical properties of the Nb added samples exhibited higher values when compared with the addition and addition of Nb.

3. CONCULUSION

This study concluded that the pre-alloying of 1 wt.% Nb significantly affected the properties and microstructure powder injection molded 420 stainless steel in the sintered and heat-treated conditions. In conclusion, experimental results show that the 420 and 420+Nb stainless steels materials can be produced using PIM techniques. The addition of Nb elements provided some benefit in terms of densification, strength, elongation and hardness. The maximum sintered density achieved in this investigation was 7.38 g/cm3 for a 420+Nb materials. Tensile strength of 1323 MPa, elongation of 2.9% and hardness of 49.3 HRC were achieved for 420+Nb addition. The conditions used for processing these materials lead to good conformity, allowing the improvement of some mechanical properties.

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