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Microstructural and Mechanical Properties of Hot Deformed AISI 4340 Steel Produced by Powder Metallurgy

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Abstract:

In this work, AISI 4340 steel was produced by powder metallurgy. Samples were sintered at 1150°C for 90 min. and then cooled in the furnace. Except to those in the as-sintered condition, one of the produced samples was homogenized at 1150°C for 1 h and then cooled in air. Other samples were deformed at the temperature range of 1150-930°C and deformation rate of 30 and 60% followed by cooling in sand, air and water mediums. The grain size gradually decreased depending on the deformation rate or cooling rate in AISI 4340 steel. Small grains occurred due to completely recrystallization at 60% deformation rate. Density and hardness also increased by the increase in deformation rate. The results showed that hot deformation is a process that is capable of improving the properties of AISI 4340 steel produced by powder metallurgy without rising the addition of alloying elements.

***Keywords**: Microalloyed Steel; Powder Metallurgy; Thermomechanical Processing; Mechanical Properties.*

1. Introduction

Proper material selection is vital for the manufacture of machinery and parts. The life of the materials is desired to be longest together with environmental factors under intense loads and stresses. For parts exposed to high stresses, heat treatable steels are the most preferred steels due to their suitability for hardenability. Hardenable steels should contain more than 0.2% carbon element in their content. AISI 4340 steel, which is a heat treatable steel with alloying elements of 0.40% C, 0.80% Cr, 0.90% Mn, 0.2% Mo, 1.83% Ni, is used in many areas such as aircraft, automobiles, crankshafts, gears where it will be exposed to high stresses. Mechanical properties are improved and new properties are added with heat treatments applied to steel [1].

Although steels contain carbon elements in their content, alloying elements other than carbon are meant when alloyed steel is mentioned. Alloying elements such as Ni, Mo, Cr, Mn, V are used to improve the mechanical properties of steel [2]. Alloying elements in AISI 4340 steel affect the microstructure and mechanical properties [3]. However, it is not always easy to put these elements together. Each element has its own melting-solidification points. Due to many different features of elements, the most suitable alloying method should be selected for

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production. There are many methods for producing alloyed steel. One of them is the powder metallurgy method which is a precise metal forming process into a clear shape of component [2].

Powder metallurgy minimizes material loss depending on the shape of the material to be produced with fewer production steps [4-9]. Complex shaped components that are difficult or impossible to manufacture can be produced with precise size control. It is possible to produce materials with high melting points and difficult to machining. Another advantage in powder metallurgy is flexibility in alloy design and composition control which is not possible with other methods [10]. Many types of materials can now be produced by powder metallurgy processes due to advances in powder metallurgy [11]. Approximately 8000 alloys could be produced from 86 elements, which are considered as metal in the periodic table. However, it may be possible to produce alloys of the order of 10^{25} with different combinations of these 86 elements. Powder metallurgy is emerging as the only method that can make this possible [12].

Production of metal powders is the first step in powder metallurgy. The produced powders are placed in a mold and pressed under a certain pressure after mixing. Then, the pressed powder metal parts are sintered in sinter furnaces. This process is usually the last step of the powder metallurgy method. However, sometimes secondary processes can be requested in final product such as heat treatment and forging [13]. Despite the many advantageous of the powder metallurgy method used during manufacturing, the pore structure in the material cannot be completely eliminated. Therefore, pore structures can be controlled by thermomechanical a process which is applied to the steel after production. Hot deformation, which is the first step in the mechanical processing of many alloys and metals, is carried out at temperatures above the recrystallization temperature. Grains, which generally elongate by changing shape during deformation, crystallize due to high temperature and small grains occur with high-strength structure. With the hot deformation process, the ductility increases and the material can be easily deformed without using high force. As a result of this deformation, herdening does not occur. Also, as high temperatures increase diffusion, a homogeneous structure can be obtained and the porosity in the structure can be eliminated or minimized [14].

It is general information that hardness and toughness of AISI 4340 steel increases after thermomechanical processes. However, literature investigation indicated that the studies on how the microstructure and mechanical properties of powder metallurgy steels which are deformed at different rates and cooled under different conditions are affected are very inadequate. Therefore, in the present study, the effects of hot deformation conditions and cooling mediums on the microstructure and mechanical properties of AISI 4340 steel, produced by powder metallurgy, were investigated.

2. Materials and Experimental Procedures

In the present study, AISI 4340 steel were produced by powder metallurgy. Iron powder of <180 μm size and graphite powder of 10-20 μm size were obtained from Aldrich. Nickel and molybdenum powder of <150 μm size and chromium powder of <45 μm size were procured from Aldrich. Analysis of powders indicated that the purity of Fe, Ni and Mo was 99.9%. On the other hand, the purity of Cr and graphite was 99% and 96.5% respectively.

The weighing of the powders at the given chemical composition was carried out in a digital precision balance with a sensitivity of 0.0001g. Weighed powders were mixed with a three-axis mixer for 1 h without ball. Mixed powders were pressed at 700 MPa using a press. 8 cylindrical samples with dimensions of Ø32x29mm were produced by cold pressing process. All pressed samples were sintered at 1150°C for 90 min. under argon atmosphere and then cooled to room temperature in a tube furnace. Except to those in the as-sintered condition, one of the produced samples was homogenized at 1150°C for 1 h and then cooled in air. Other samples were deformed at the temperature ranges of 1150-930^{o}C for the deformation rates of 30 and 60% in an open die under normal atmospheric condition and then cooled in sand, air and water mediums.

Temperature before and after hot deformation was measured by using a infrared laser temperature measuring instrument. The microstructures of the produced samples were examined by an optic microscope, scanning electron microscope (SEM) and energy dispersive spectrometry (EDS). The mean linear intercept method was used to determine average grain size of samples. Volume fraction of ferrite, and pearlite were also determined by using point counting method. Vickers hardness measurements were done by using $Hv_{0.5}$ (500 g) load.

3. Results and Discussion

Fig. 1 shows the optical and SEM microstructures of the sintered and homogenized samples. Table I also gives density (%), porosity (%), pearlite (%), ferrite (%) and average grain size values of AISI 4340 steel samples under different conditions. As is seen from Figs 1a and 1b that the sintered and homogenized samples consist of ferrite and pearlite phases with equiaxed grain sizes. In addition, after the homogenization heat treatment, ferrite and pearlite phases are more homogeneously distributed in the structure. However, it is seen from Table I that the homogenized samples (45 μm) have a smaller grain size than the sintered samples (88 μm). This is due to the fact that homogenized samples were cooled faster than sintered samples. At low cooling rates such as furnace cooling, recrystallization and even grain growth occurs before the austenite-ferrite transformation [15,16].

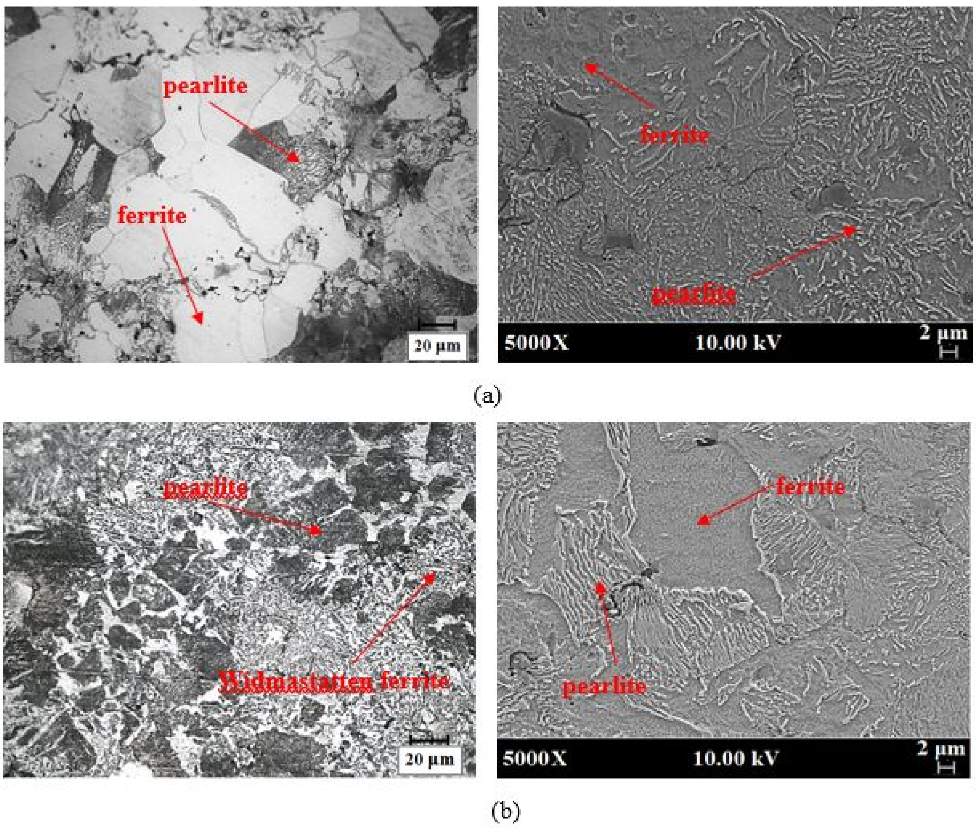


Fig. 1. The microstructure of the AISI 4340 steel a) sintered for 90 min. at 1150°C, b) homogenized for 1 h at 1150°C after sintering.

Tab. I Density (%), porosity (%), pearlite (%), ferrite (%), average grain size and hardness values of AISI 4340 steel obtained under different conditions.

Conditions	Cooling Mediums	Density (%)	Porosity (%)	Ferrite (%)	Pearlite (%)	Grain Sizes (µm)	Hardness ($Hv_{0.5}$)
Sintered	Furnace	90	10	73	27	88	182
Homogenized	Air	92	8	60	40	45	226
30% deformed	Sand	96	4	65	35	51	220
	Air	94	6	58	42	41	238
	Water	96	4	-	-	-	675
60% deformed	Sand	97.5	3	71	29	36	295
	Air	97.30	3	68	32	30	328
	Water	97.33	3	-	-	-	729

Fig. 2 and Fig. 3 show the microstructure of the samples, which were homogenized for 1 h. at 1150°C and then deformed for 30% and 60% in the temperature range of 1150-930°C. Finally they were cooled in sand, air and water.

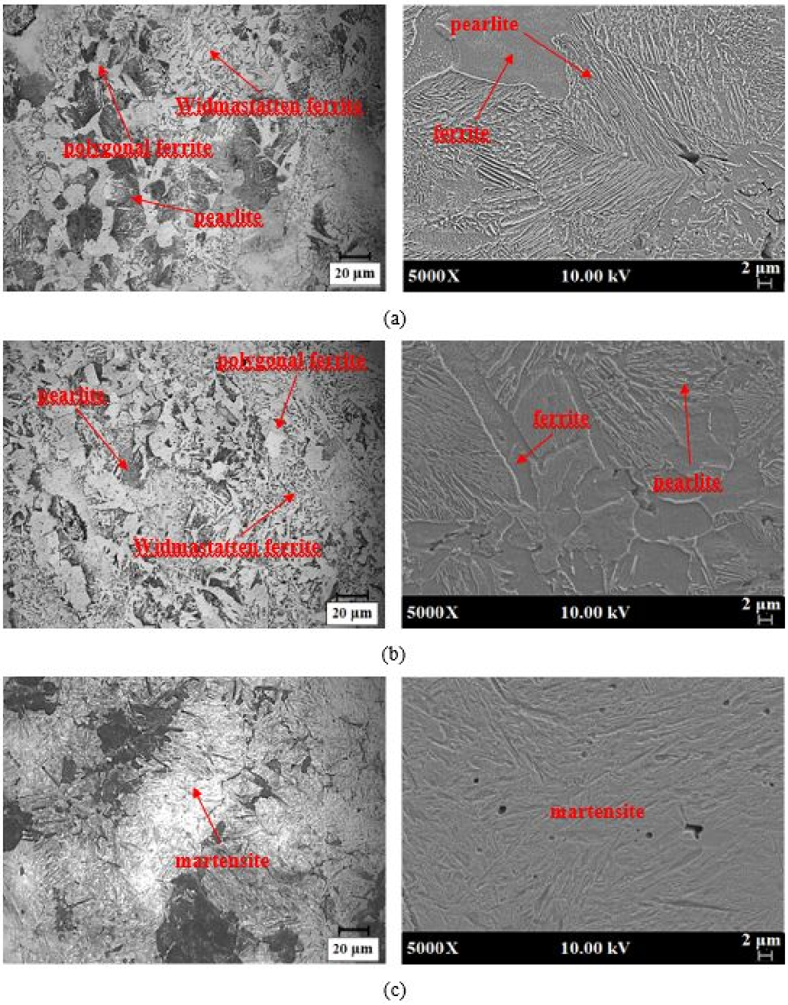


Fig. 2. The microstructure of the AISI 4340 steel deformed at temperatures in the range of 1150-930°C for 30 % and then cooled in a) sand, b) air and c) water.

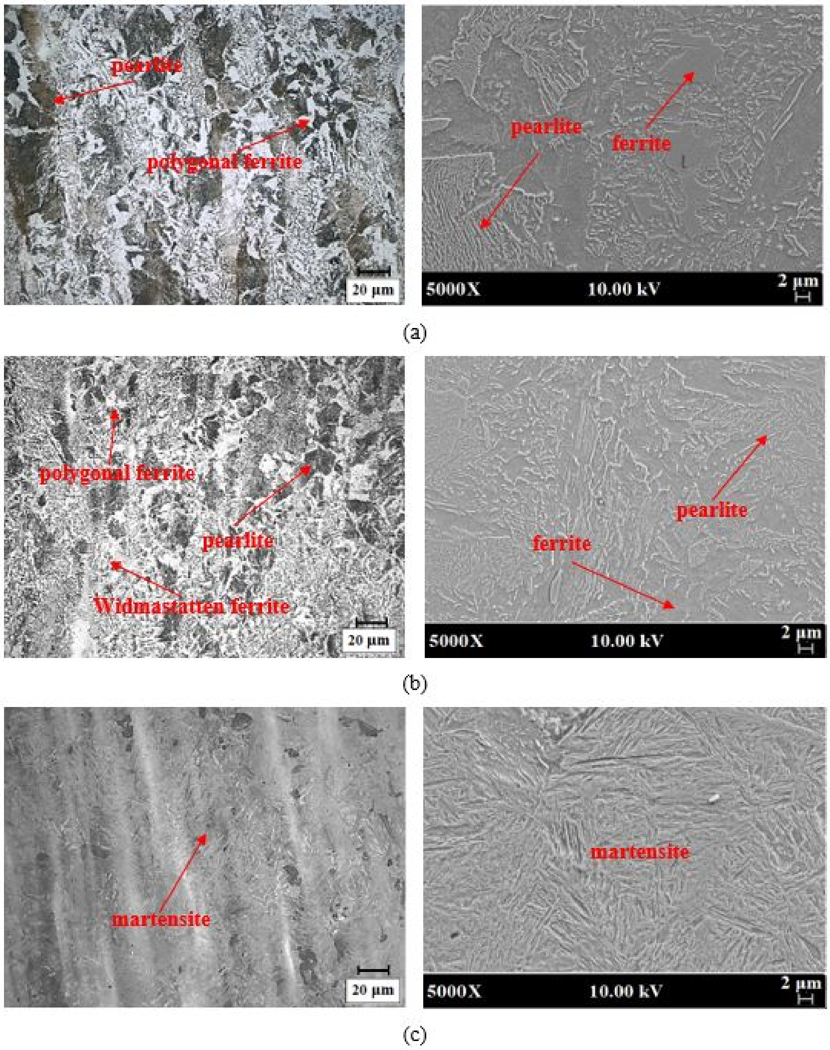


Fig. 3. The microstructure of the AISI 4340 steel deformed at temperatures in the range of 1150-930°C for 60% and then cooled in a) sand, b) air and c) water.

As can be seen from Fig. 2, Fig. 3 and Table I, preeutectoid ferrite was distributed as a thin, continuous network at the prior austenite grain boundaries and the ferrite volume fraction decreased in both 30% and 60% deformed samples as the cooling rate increased. This is usually related to the nucleation and growth rates of ferrite due to the increase in cooling rate [17, 18]. Samples cooled in air contain a lower volume fraction of preeutectoid ferrite than samples cooled in sand since there is not enough time for the formation of preeutectoid phases during cooling in air [19]. In addition, increasing the cooling rate decreases the transformation temperature and the ferrite and perlite phases form as a fine grained at lower temperatures.

After 30% and 60% plastic deformation, the cooling of the samples in the sand with the lowest cooling rate causes recrystallization and grain growth of austenite before the austenite-ferrite transformation (Fig. 2a and Fig. 3a). As a result of this the final structure consists of coarse ferrite and pearlite grains [16, 17]. However, the microstructure of the samples cooled in air after 30% and 60% plastic deformation consists of fine ferrite and pearlite grains (Fig. 2b and Fig. 3b). Also, it is seen that Widmanstatten ferrite occured at higher cooling rates than polygonal ferrite formed in the samples cooled in air [20].

The microstructure of the samples cooled in water after 30% and 60% plastic deformation consists of the martensite phase as seen in Fig. 2c and Fig. 3c. Martensite is a very hard and brittle structure formed by diffusionless transformation as a result of rapid cooling. When the cooling rate is increased and exceeded a critical value, carbon atoms cannot find enough time to diffuse from the solid solution. Even if the iron atoms move a little, the carbon atoms supersaturated in solid solution, the lattice structure cannot transform into BCC structure and a different structure is formed. This structure formed as a result of rapid cooling is called "martensite". Martensite is a solid solution with a body centered tetragonal (BCT) structure supersaturated with carbon [21]. Also the presence of 0.8% Cr and 0.2% Mo in AISI 4340 shifts the TTT and CCT diagrams to a longer time and reduces the critical cooling rate resulted in easier transformation of martensite phase [22]. Martensite is not a desired phase because it reduces the toughness of steels [23].

When the samples deformed by 30% and 60% after the homogenization process are compared to each other, the average grain size of the 60% deformed sample is smaller and volume fraction of ferrite is higher under all cooling conditions (see Table I). This is consistent with the results obtained by Smith and Siebert [24] who studied the effect of deformation on the continuous cooling transformation (CCT) diagram in 0.1%C-0.39%Mo-B steel. In a specimen that was not deformed at all, austenite and pearlite noses are located at longer times. Ferrite nose is shifted to shorter time in 50% deformed sample, while nucleation of ferrite increases due to deformation in the nonrecrystallization region. It was also observed that the nucleation of the pearlite at the grain boundaries of the 30% deformed samples was 8 times larger than the non deformed samples. This is attributed to the increase in the densities of the strained regions at the austenite grain boundaries which facilitate nucleation rates. In addition, Inagaki [25] showed that the introduction of highly strained regions at the grain boundaries or near the boundaries of annealing twins increase the ferrite nucleation and, therefore, to the grain refinement. This situation shows that the deformation in the nonrecrystallization region of austenite accelerates the nuclation rate and formation of ferrite. As a result of this, small ferrite grains occurs and the volume fraction of ferrite increases in structure [26].

In addition the results obtained from present study indicated that 30% and 60% deformed samples showed smaller grain sizes than sintered and homogenised samples under all cooling conditions. This also indicates that deformation promotes grain refinement in AISI 4340 steel produced by powder metallurgy. Li [27] investigated the effect of forging ratio on the microstructure of H13 steel, it was indicated that grain size decreased with the increase of forging ratio which provided more nucleation points and storage energy for the recovery and recrystallization of grains. This promoted the grain refinement.

As is seen in Table I that the density (%) values increase with the increasing deformation rate of the samples heat treated and deformed under different conditions. The traditional powder metallurgy method includes powder production, cold pressing and sintering [28]. The structure of the materials produced by this method is porous which may cause a decrease in mechanical properties. Porous structure restricts the use of materials in high strength applications by causing the stresses increment [29]. It is indicated in the literature that the application of hot deformation to the materials produced by the powder metallurgy causes to exhibit better mechanical properties than the materials produced by the casting and forging method [30]. Accordingly, it was determined that the amount of pores in the material decreased with the deformation applied in the present study.

Fig. 4 shows the SEM microstructure of the sintered, homogenized, 30% and 60% deformed samples. When the microstructure of the samples was examined, it was determined that the grain size decreased with the applied deformation and also the porous structure was minimized.

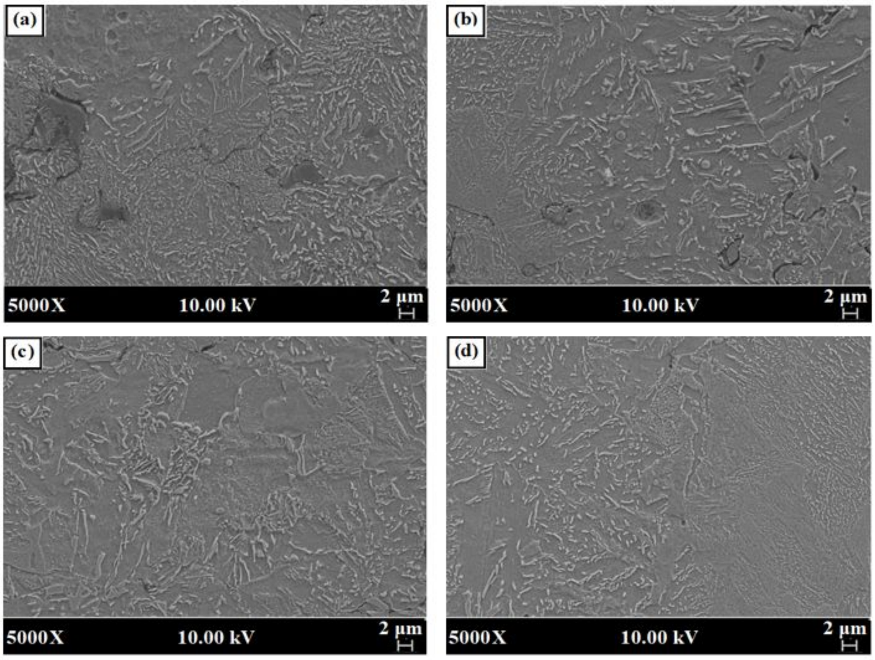
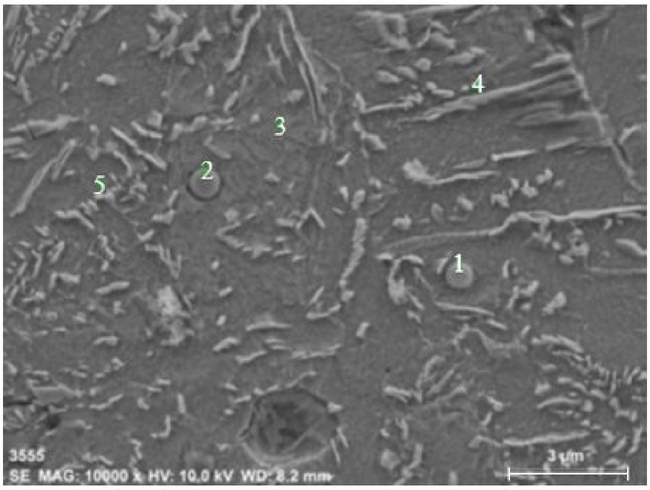


Fig. 4. SEM micrograph of a) sintered, b) homogenized, c) 30% deformed and d) 75% deformed samples cooled in air.



Mass percent (%)

Spectrum	C	Cr	Mn	Fe	Ni	Mo
1	1.91	0.52	33.71	9.44	2.71	51.71
2	1.43	0.12	32.96	10.28	1.48	53.73
3	3.34	0.49	0.00	95.67	0.51	0.00
4	10.48	0.00	0.00	87.21	1.57	0.74
5	7.93	0.56	0.00	89.76	1.56	0.19
Mean value:	5.02	0.34	13.33	58.47	1.57	21.27
Sigma:	3.99	0.26	18.26	44.48	0.78	28.72
Sigma mean:	1.78	0.12	8.17	19.89	0.35	12.84

Fig. 5. SEM micrograph of homogenized AISI 4340 steel sample and corresponding EDS of the indicated particles.

Fig. 5 shows the EDS results of the homogenized sample. When the results are examined, it is seen that precipitates with different sizes are formed. In addition, EDS results show that Fe_3C, CrC(N) and MoC(N) precipitates are formed because these precipitates contain Fe, C, Cr and Mo elements. The accumulation of deformation may initiate the precipitation of carbonitrides in steels with the alloy addition [31-33]. Precipitates are more effective for the inhibition of the grain growth compared to the dissolved atoms [34].

Table I also shows the hardness values of sintered, homogenized, 30% deformed and 60% deformed samples under different cooling conditions. As is seen from Table 1 that homogenized sample (226 Hv) showed higher hardness than sintered samples (182 Hv). This increase in hardness can be attributed to the grain refinement occurring in steels with increasing cooling rates. The cooling rate of homogenized samples cooled in air is higher than the cooling rate of sintered samples cooled in the furnace. An increase in cooling rate lowers transformation temperature and ferrite and pearlite occur at lower temperature resulted in finer ferrite and pearlite grains [22].

The hardness results also indicated that water quenched samples deformed for 30% and 60% showed higher hardness than sand or air cooled samples. This is because of the higher amount of free carbon in martensite phase. It was also observed that air cooled samples showed higher hardness compared to the sand cooled samples because of higher cooling rates which give a fine dispersion of small MoC(N) or CrC(N) formed in AISI 4340 steel. [18]. The hardness results of steels produced by powder metallurgy also indicated that the highest hardness was achieved by 60% deformed samples, and followed by 30% deformed, homogenised and sintered samples. The highest hardness of 60% deformed sample is caused by increased nucleation site. It is to be noted that the most frequent nucleation sites differ depending on the amount of deformation; when the amount of deformation is small the γ grain boundary is the most important while, with an increasing amount of deformation, the annealing twin boundary and deformation band become more and more important sides for transformation [26]. This gives finer structure dispersed pearlite and precipitate particles, thereby increasing the hardness

It is also seen from Table I that the relative density of AISI 4340 steel increases with the increasing the deformation rate and the percentage porosity decreases accordingly. The mechanical properties of parts produced by powder metallurgy are also related to the amount of porosity. Since pores in steel causes the accumulation of stresses which contribute to crack propagation [30-33]. Formability and toughness, which are much more dependent on density, increase in cases where density is high [39-43]. It was observed that the amount of porosity is minimized with increasing deformation rate. As a result of this, hardness of AISI 4340 steels showed an increase with rising the deformation rate.

4. Conclusion

In this work, microstructural and mechanical properties of hot deformed AISI 4340 powder metallurgy steel were investigated. The following conclusions obtained from present study are as follows:

- After sintering and homogenization processes, it is possible to produce AISI 4340 steel with high density and hardness as a result of hot deformation in the austenite region under normal atmospheric conditions.
- The highest hardness was achieved by 60% deformed samples, and followed by 30% deformed, homogenised and sintered samples.
- A fine-grained microstructure was obtained in AISI 4340 steel with increasing deformation rate causing deformation of the grain boundaries, increasing the nucleation rate and volume fraction of ferrite.
- The amount of porosity, which is one of the disadvantages of powder metallurgy,

decreased with increasing the deformation rates and the steels became more homogeneous with the increase in density.

- Deformed AISI 4340 steel showed finer microstructure and better mechanical properties then nondeformed AISI 4340 steel for all deformation and cooling conditions. This can be attributed to the grain refinement occurring in steels with increasing deformation rate and also to the increase in the amount of MoC(N) or CrC(N) formed in AISI 4340 steel.
- Hot deformation is a process that is capable of improving the properties of AISI 4340 steel produced by powder metallurgy without rising the alloying element additions.

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Сажетак: *У овом раду, челик AISI 4340 је произведен металургијом праха. Узорци су синтеровани на 1150°C у трајању од 90 минута, затим су охлађени у пећи. Осим оних у синтерованом стању, један од произведених узорака је хомогенизован на 1150 °C током 1 часа, а затим је охлађен на ваздуху. Остали узорци су деформисани у температурном опсегу 1150-930°C са стопом деформације од 30% и 60% након чега је уследило хлађење у медијуму песка, ваздуха и воде. Величина зрна унутар челика AISI 4340 постепено опада у зависности од степена деформације или брзине хлађења. Ситна зрна су настала услед потпуне рекристализације при стопи деформације од 60%. Густина и тврдоћа се такође повећавају повећањем стопе деформације. Резултати су показали да је врућа деформација процес који је способан да побољша својства челика AISI 4340 који је произведен металургијом праха без повећања додавања легирајућих елемената.*

Кључне речи: *Микролегирани челик, металургија праха, термомеханичка обрада, механичка својства.*