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Mustafa Türkmen
Kocaeli University, mustafa.turkmen@karabuk.edu.tr Kocaeli-Turkey
Hasan Karabulut
Mehmet Akif Erden
Süleyman Gündüz
Karabük University, Karabük-Turkey
hasankarabulut@karabuk.edu.tr; makiferden@karabuk.edu.tr;
sgunduz@karabuk.edu.tr

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ORCID ID	0000-0003-1081-4713	0000-0002-3370-9994

EFFECT OF TIN ADDITION ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF PM STEELS

ABSTRACT

In this work, the effects of TIN additions on the microstructures and tensile behaviors of microalloyed powder metallurgy (PM) steels were investigated. The microstructure of the microaloyed PM steels was characterized by optic microscope, SEM and EDS. Results indicated that the addition of TiN in the percentage of 0.1, 0.2 or 0.5 increases the yield strength (YS) and ultimate tensile strength (UTS) of the PM steels in the sintered conditions. Elongation also tends to improve with increasing TiN content. In addition, TiN prevented grain growth during sintering prior to cooling.

Keywords: Powder Metallurgy, Microalloyed Steel, Tensile Strength, SEM, EDS

1. INTRODUCTION

Tendency towards the production of new types of materials for specific applications is increasing with each passing day because simple material types have not been able to meet the increasing demands in the last 20 years [1]. Two or more of the same or different groups of materials are combined together for the best features or to reveal a new feature. Microalloyed steels are group of steels which include niobium (Nb), vanadium (V) and titanium (Ti) with the range of 0.05% and 0.20 % and have superior properties such as high strength, high toughness, and low ductile brittle transition temperature [2]. One of the properties of microalloyed elements is that they perevent grain growth during sintering and austenitization with carbides and nitrides they have created. TiN are microalloyed precipitates that reduce grain growth, increase satiety, and are found insoluble in the HAZ area of the weld. Moreover, TiN is resistant to high temperatures, and it is widely used for wear protection [3 and 5]. Powder metallurgy (PM) production technology makes it possible to economically produce high-quality, complex parts. This manufacturing technique converts metal powder having different sizes, shapes and packing properties into strong, precise and high-performance parts [6]. In this study, the desired composition PM steel were produced with the powder metallurgy technique by adding TiN in different ratios into Fe matrix and resulting microstructure mechanical properties were compared.

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2. RESEARCH SIGNIFICATION

Powder metallurgy (PM) approaches are advantageous in producing near net shapes of components, and therefore largely increase the utilization ratio of materials and reduce machining cost. Homogeneous and fine microstructure can be obtained by means of this way. PM is a method of producing metal powders prepared in a desired chemical composition by squeezing in a mould and then sintering at high temperature after shaping. The most important advantage of this method is that it is easy to produce parts that are difficult or impossible to produce by conventional production methods such as casting and machining. In addition, it is also possible to produce economical, fast and precise parts from high melting grade metals and alloys. The present study was undertaken to examine the effect of the TiN addition on the microstructure and mechanical properties of sintered PM steel. For this purpose TiN in different weight percentage was added in to several PM steel. Mechanical properties were measured and microstructures were characterized in the sintered condition to assess the role of precipitation strengthening and grain refinement. This study aims to contribute to increasing steel production by powder metallurgy which is expected to have an effect on the international platform.

3. EXPERIMENTAL STUDY

In this study, steel samples were prepared in the desired compositions by PM method. The effects of the varying rates of TiN amount added on microstructure and mechanical properties were investigated. Steel production was carried out by mixing the chemical compounds given in Table 1. After the characterization of the microstructures in produced samples, tensile test was performed. The obtained results are compared with each other. Prior to mixing process, the powders were prepared by using a digital weighing scale with a precision of 0.0001. The powders were stirred with Turbula T2F mixer for 1 hour. Homogeneously mixed powders were pressed with 96 ton pressing capacity-Hidroliksan brand machine at 700 MPa compaction pressure in one direction. Tensile specimens were prepared in accordance with ASTM (E 8M) powder metal material tensile standards and were compressed into blocks with molds.

Table 1. Chemical compositions of powder metallurgy steels

Alloy	C(% wt.)	TiN(% wt.)	Fe (% wt.)
Alloy 1	0.25	-	Balance
Alloy 2	0.25	0.1	Balance
Alloy 3	0.25	0.2	Balance
Alloy 4	0.25	0.5	Balance

The pressed samples were sintered at 1150°C for 1 hour in an argon atmosphere. After sintering, the density and porosity measurements were made and their values were determined. Sintered samples were prepared for metallographic examination using conventional methods (sanding, polishing and etching). Microstructures of tensile samples sintered at 1150°C were examined with the X50-X1000 magnification capacity-Nikon Epiphot 200 brand optical microscope. Tensile testing was carried out with 50KN capacity-Shimadzu brand tensile testing device with a tensile speed of 0.5mm/min. Flow strength (0.2%), tensile strength and percentage elongation values of the broken samples after tensile testing were calculated. Vickers hardness value was determined by applying a load of 0.5kg with Shimadzu brand hardness device. The density of the samples was determined according to Archimedes principle using the density measurement kit. Moreover, the volume fraction of pearlite in powder metal steel is calculated using the metallographic point counting method described by Gladman and Woodhead [7]. Moreover, TiC, TiN and TiCN precipitates were determined with the help of EDS point and line analyses.

4. EXPERIMENTAL RESULTS AND DISCUSSION

4.1. Microstructure

Microsturcture of the samples are seen in Figure 1. As it can be understood from the figure, in all the alloys the structure is composed of ferrite and pearlite phases. In the microstructure image in Figure 1, partial closure of the pores in the grain boundaries can be observed. Although in many sources it is stated that porosity negatively affects strength, it has been reported that very small and spherical shaped pores do not reduce strength [6]. From microstructure images, it is understood that in PM steel samples the grain size decreases as the amount of TiN increases. It was obserbed that the average grain size increased in alloy with 0.5% TiN. For instance, whereas the average grain size of nonalloyed PM steel in Fe+0.25C composition is 29.7µm, after increasing the amount of TiN to 0.1 and 0.2 by wt.%, the grain size decreased to 25.8µm ve 24.22µm respectively. It valued 27.16µm when the TiN amount was 0.5 by wt.%.

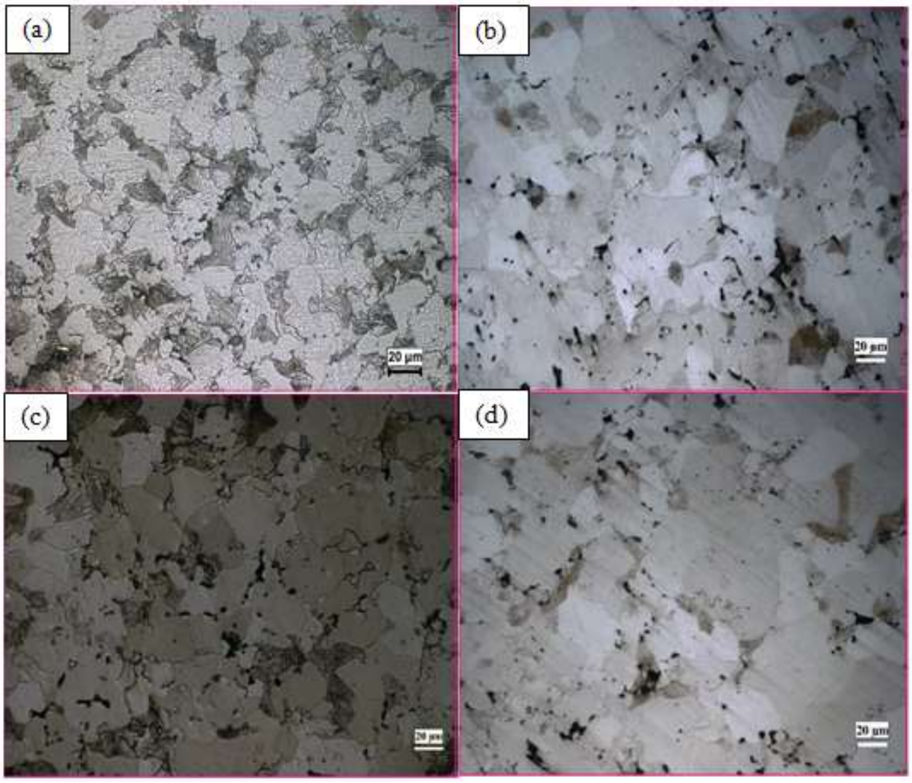

Figure 1. Microstructure images of PM steel samples sintered at 1150°C; (a) Alloy 1, (b) Alloy 2, (c) Alloy 3, and (d) Alloy 4 (500x magnification)

Table 2 also shows that the average grain size decreases with increasing the amount of TiN up to 0.2% by weight. This is due to the precipitation of TiC, TiN ve TiC(N) precipitates during sintering which prevented the growth of the austenite grains [5]. One of the properties of microalloy elements is that they avoid the grain growth during sintering and austenitization with the carbide and nitrides they have created up. The formation of small precipitates during austenitization inhibits the growth of austenite grains, and creates the formation of small ferrite grains during cooling [8 and 10].

Table 2. Relative density, porosity, mean linear intercept grain sizes and volume fractions of pearlite phase in microalloyed PM specimens

Alloy	Relative Density (%)	Porosity (%)	Ferrite (%)	Perlite (%)	Grain Size (μm)
Alloy 1	92	8	79.4	21.6	29.7
Alloy 2	93.29	6.71	79.45	20.25	19.25
Alloy 3	92.87	7.13	76.80	23.20	18.43
Alloy 4	87.92	12.08	77.29	22.71	21.85

With an increase in the amount of of TiN from %0.2 to %0.5 by weight, average grain size has been observed to increase. The reason for this can be considered as the formation of big TiN precipitates in the grain boundaries [2 and 5]. Because the resulting big precipitates could not sufficiently prevent grain growth, this caused the average grain size to growth.

4.2. Mechanical Properties

Figure 2 shows the stress-strain diagrams of the sintered samples. Table 3 also shows the yield strength, tensile strength, percentage elongation and hardness values. When TiN amount increased up to 0.2% by weight, an increase was observed in the yield strength, tensile strength, percentage elongation and hardness values. Carbide, nitride and carbonitride precipitates formed by the titanium element provide small-grained material by preventing austenite grain size and the recrystallisation of austenite. More grain boundaries exist in small-grained structure, and these grain boundaries block the movement of the dislocation; therefore, strength increases. Decrease in grain size contributed to % elongation of the material [2, 4, 9 and 10].

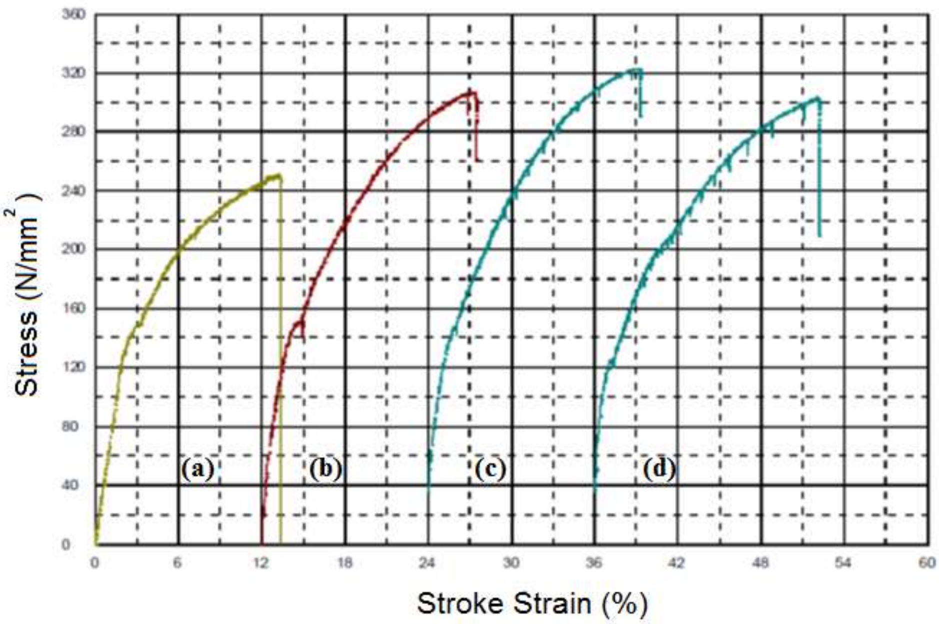

Figure 2. Variation of stress-strain curves of PM steels with different percentage of TiN: Alloys 1 (a), 2 (b), 3 (c), and 4 (d)

Table 3. Yield strength (YS), Ultimate tensile strength (UTS), Elongation ad Hardness (HV) of microalloyed PM steels

Alloy	Yield strength (YS)(MPa)	Ultimate tensile strength (UTS)(MPa)	Elongation (%)	Hardness ($HV_{0.5}$)
Alloy 1	144	252	13	68
Alloy 2	151	307	15	102
Alloy 3	148	324	15	112
Alloy 4	128	304	16	98

It is thought that precipitates such as TiC(N) cause the formation small austenite grains by preventing grain growth during sintering and as a result they increase the strength of the material. For instance, Erden et al. [2] have produced Ti micro-alloy steel with PM method in their work. Sintering process was carried out at 1150°C by holding it 60 minutes, and they determined that the yield and tensile strengths increased as Ti amount (0.1% to 0.2%) increased. They associated this result with the formation of precipates such as TiC(N) during sintering and cooling after sintering. Authors showed in their work that precipates such as TiC(N) caused the formation of small austenite grains by preventing grain growth during sintering and as a result of this the strength of the materials increased. In other studies [1, 9 and 10], it is noted that carbide and nitride formed in micro-alloy steel caused an increase in hardness and strength. In the same studies, it is also expressed that solid solution hardening remains at low rates due to carbide and nitride precipitation [3 and 7]. In addition, a high proportion TiN in steel causes excessive precipitation hardening and therefore makes the material more brittle and causes a decrease in strength.

4.3. SEM-EDS Analysis

From the SEM image of sample having 0.2 TiN compositions given in Figure 3, it was found that precipitates having different sizes emerged. Moreover, EDS point analysis results showed that these precipates are TiC(N) because they contain titanium, carbon and nitrogen elements, and also Fe3C formed because they contain iron and carbon. It can be said that these precipitates prevent the grain growth of austenite and recrystallization, and increase the strength of the material by precipitate hardening [9 and 11]. The effect of microalloying elements in the solution on austenite recrystallization is very weak. Preventing grain boundary movement with precipitated particles has much more influence than soluble atom [12].

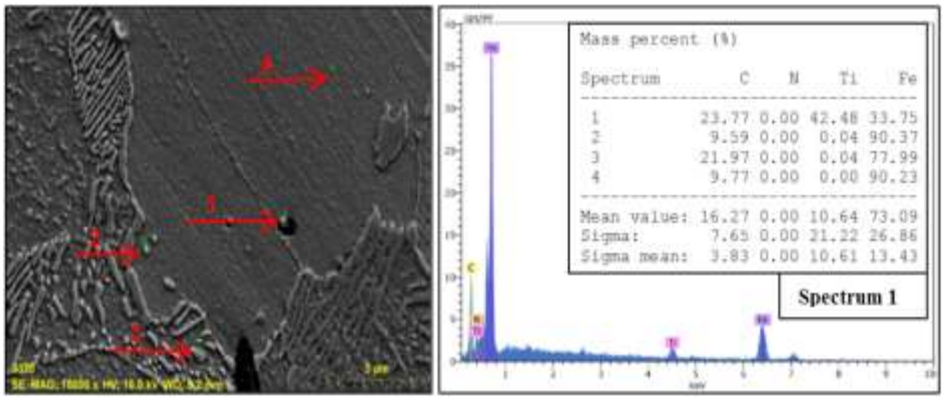

Figure 3. SEM image of PM steel sample (Alloy 1) a) X 10000, b) Point EDS results and spectrum 1

Figure 4 shows line EDS analysis results taken from matrix and precipitate of the PM steel sample containin %0.2 TiN sintered at 1150°C. From the line EDS analysis of PM steel containing %0.2 TiN, it can be seen that there are differences in element types and amounts along the line intersecting the matrix and the precipitate It was found that the matrix phase is rich in iron whereas the round shaped precipitate is rich in titanium element. Moreover, there is a sharp increase in titanium amount at the where the analysis line from matrix cuts the precipate.

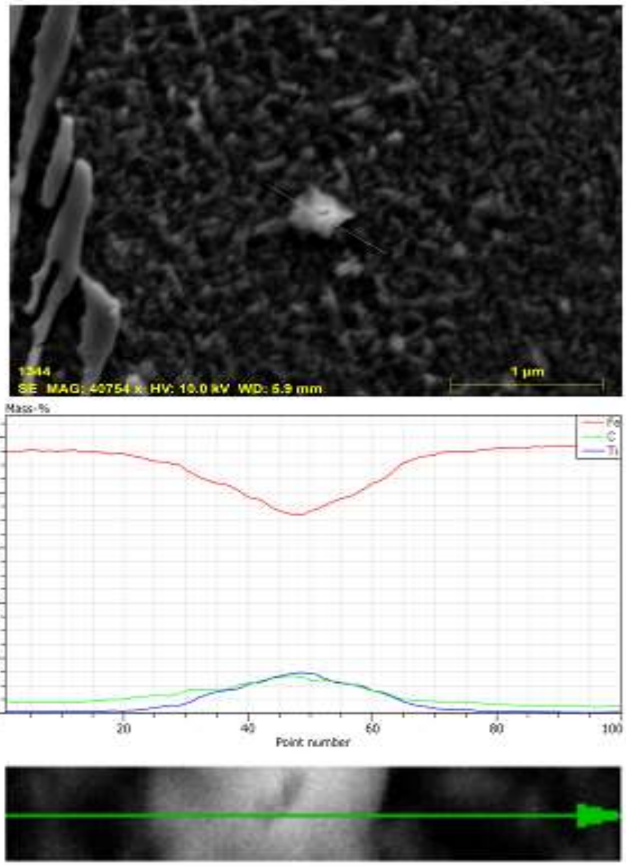

Figure 4. EDS line scan for Alloy 3 (Fe-0.25C-0.2TiN)

In this study, point and line EDS analysis results show that precipates such as TiC, TiN ve TiC(N) are formed in PM steel with titanium.

5. CONCLUSIONS

TiN alloyed PM steel with different amount of TiN (0.1-0.2 and 0.5wt.%) was produced by applying cold pressing and sintering in argon atmosphere and the following results were obtained.

- Fe matrix composite materials containing TiN can be produced with powder metallurgy. Precipitation of TiN occurs during sintering or during cooling after sintering. These precipitates increase the strength of the steel.
- Unlike nonalloyed steel, PM steels which was added 0.1% and 0.2% TiN by weight exhibited a smaller grain size. This is due to the fact that carbide and nitride formed by alloy elements block the grain growth.
- Overall an increase is observed in the tensile strength, percentage elongation and hardness values of PM steels as the

amount of TiN increased up to 0.2 wt.%. This is a result of the formation of Ti(CN) precipates that occur during sintering or cooling after sintering.

- It is seen that post-sintering density of the nonalloy and TiN added composite materials is around 91%. After sintering, densification showed a slight increase.

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NOTE

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REFERENCES

1. Karaduman, B., (2010). Production and Characterization of Titanium Matrix Composites Reinforced Titanium Carbide, M.Sc. Thesis, Istanbul Technical University, Institute of Science and Technology, 1-15.
2. Erden, M.A., Gündüz, S., Türkmen, M., and Karabulut, H., (2014). Microstructural Characterization and Mechanical Properties of Microalloyed Powder Metallurgy Steels, Materials Science and Engineering A, 616, 201-206.
3. Gladman, T., (1997). The Physical Metallurgy of Microalloyed Steels, the Institue of Materials, England, 1, 341.
4. Sage, A.M., (1992). An Overview of the Use of Mikroalloys in HSLA Steels with Particular Reference to Vanadyum and Titanium, Processing, Properties and Applications, Proceedings of the Second International Conference on HSLA Steels, Beijing, China.
5. Karabulut, H. and Gündüz, S., (2004). Effect of Vanadium Content on Dynamic Strain Ageing in Microalloyed Medium Carbon Steel, Materials and Design, 25, 521–527.
6. Sarıtaş, S., Türker, M., and Durlu, N., (2007). Powder Metallurgy and Particulate Materials Processing, Turkish PM Publications, 05, 2-34, 206-237, 379-410.
7. Gladman, T. and Woodhead, J.H., (1960). The Accuracy of Point Counting in Metallographic Investigations, Journal of the Iron Steel, 194, 189-194.
8. Misra, R.D.K., Tenneti, K.K., Weatherly, G.C., and Tither G., (2003). Microstructure and Texture of Hot Rolled Cb-Ti and V-Cb Microalloyed Steels with Differences in Formability and Toughness, Metall. Mater. Trans. A, 34A, 2341–2351.
9. Schade, C., Murphy, T., Lawley, A., and Doherty, R., (2012). Microstrucure and Mechanical Properties of Microalloyed PM Steels, International Journal of Powder Metallurgy, 48(6):51-59.
10. Schade, C., Murphy, T., Lawley, A., and Doherty, R., (2012). Microstructure and Mechanical Properties of PM Steels Alloyed with Silicon and Vanadium, International Journal of Powder Metallurgy, 48(6):41-48.
11. Xiang-done, H., Xin-ping, M., and Sheng-xia, L., (2013). Effect of Annealing Temperature on Recrystallization Behavior of Cold Rolled Ti-microalloyed steel, J. of Iron and Steel Res Int., 20(9):105-110.
12. Korchynsky, M., (2001). The Use of Vanadium in Steel, Proceedings of the Vanitec Symposium, Beijing, China.