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Araştırma Makalesi / Research Article

Effect of Boron Nitride Addition on the Microstructure and Mechanical Properties of PM Steels

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Keywords
PM Steels, Boron Nitride, Microstructure, Mechanical Properties.

Abstract

In this work, the effects of boron nitride additions on the microstructures and tensile behaviours of microalloyed powder metallurgy (PM) steels were investigated. The microstructure of the microalloyed PM steels was characterised by optic microscope, SEM and EDS. Results indicated that the addition of boron nitride in the percentage of 0.1 and 0.2 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 boron nitride content. In addition, the presence of boron nitride in different proportion prevented grain growth during sintering or cooling after sintering. However, a further increase in BN to 0.5 wt.% reduced the yield strength (YS), ultimate tensile strength (UTS), percentage elongation and hardness due to higher amount of precipitation strengthening.

Toz Metal Çeliklerin Mikroyapı ve Mekanik Özelliklerine Bor Nitrürün Etkisi

Anahtar kelimeler
Toz Metal Çelikler, Bor Nitrür, Mikroyapı, Mekanik Özellikler.

Özet

Bu çalışmada, Mikroalaşımlı Toz metal (TM) çeliklerin mikroyapı ve çekme davranışları üzerine bor nitrür ilavesinin etkileri incelenmiştir. Mikroalaşımlı TM çeliklerin mikroyapıları optik mikroskop, SEM ve EDS analizleri kullanılarak karakterize edilmiştir. Sonuçlar; yüzde 0,1 ve 0,2 oranında bor nitrür ilavesinin TM çeliklerin akma dayanımını (YS) ve çekme mukavemetini (UTS) artırdığını göstermiştir. Ayrıca % Uzama oranı bor nitrür ilavesinin artmasıyla artma eğilimindedir. Buna ek olarak, farklı oranda bor nitrür varlığı sinterleme sırasında veya sinterleme sonrasında tane büyümesini engellemiştir. Ancak ağırlıkça % BN oranını 0,5 olduğunda akma dayanımı (YS), çekme dayanımı (UTS), % uzama ve sertlik değeri çökelme sertleşmesine bağlı olarak azalmıştır.

1. Introduction

Steels are material groups which can have superior features such as high strength, low ductile-brittle transition temperature and high toughness by the application of various hardening mechanisms and suitable thermodynamic processes (Erden *et al*. 2014).

Main role of microalloyed elements are to reduce the grain size, prevent recrystallization and contribute to precipitate hardening (Erden *et al*. 2014; Korchynsky, 2001; Sage, 1992). Boron nitride within the structure of cubic crystal is known to be one of the hardest elements after diamond, and it is also a perfect lubricator in high temperature applications when it exists within the structure of hexagonal crystal cage. However, the only good thing with hexagonal boron nitride is not being a lubricator; high thermal conductivity, thermal shock endurance, smooth surface output, refractory characteristics, being inert, low wettability, being a non-poisonous and clean material are its advantages that promoted the use of hexagonal boron nitride (Öztaş, 2012).

Many literatures define powder metallurgy (PM) as the method to manufacture hard-to-produce parts (structures which are small, functional, non-compatible to each other, composite etc.) with high resistance and minimum tolerance (with low diminish) in a more advantageous way compared to other production methods (Schade *et al*. 2012a; Schade *et al*. 2012b; Robert, 1984). Today, although most of the microalloyed steels are produced as flat and pipe product, production of microalloyed steels for forging purpose is increasing. Although it is not widely preferred yet, microalloyed steels are also produced by powder metallurgy method today (Erden *et al*. 2014; Schade *et al*. 2012b).

The purpose of this study is to investigate the effect of BN amount in the steels produced by PM method on the microstructure and mechanical properties. The change in microstructure and mechanical properties of PM steels are examined when 0.1, 0.2 and 0.5 wt. % BN are added to the PM steels sintered at 1150 °C.

2. Material and Method

In this study, steel samples are produced by PM method with desired compositions and the effects of BN, added in different amounts to the steel, on the microstructure and mechanical properties are investigated. Non-alloyed steel and boron steel are produced by mixing the elements given in Table 1.

Table 1. Chemical compositions of PM steel specimens.

Compound	Graphite (wt%)	BN (wt%)	Fe (wt%)
Alloy 1	0.45	-	Balance
Alloy 2	0.45	0.1	Balance
Alloy 3	0.45	0.2	Balance
Alloy 4	0.45	0.5	Balance

Iron, graphite and BN powders were used to produce the steel specimens. The size of graphite, iron and BN powders were <20, ≤180, and <1 µm, respectively. These powders were supplied by Aldrich. The purities of graphite, Fe and BN are 96.5 %, 99.9 and 98%, respectively. After microstructure characterization in the produced specimens, tensile test is made and the results are compared to each other. Before mixing process, powder was weighed and made ready on digital precision scale with 0.0001 precision in the ratios given in Table 1. Powders are mixed by Turbula T2F mixer for 1 hour without ball and compacted by a using Hidroliksan brand device with 96 tons of press capacity under 700 MPa uniaxial pressure. Tensile test specimens are pressed in a mold prepared in accordance with ASTM (E 8M) powder metal material tensile specimen standards, and turned into a block. Pre-tensile test image of the tensile sample is given in the Figure 1.

Figure 1. General view of tensile test specimen for Alloy-1 sintered at 1150 °C for 1 h

The pressed specimens are sintered in argon atmosphere at 1150 °C temperature for 1 hour. Density measurements are made after sintering and pore values are determined. The sintered specimens are made ready for metallographic examination using traditional methods (grinding, polishing and etching). Microstructures of PM steel specimens sintered at 1150°C are examined by Nikon Epiphot 200 brand optical microscope with X50-X1000 zoom capacity and Carl Zeiss Ultra Plus Gemini FESEM device. Tensile test is performed by Shimadzu brand tensile device having 50KN capacity with a crosshead speed of 0.5 mm/min. Yield strength (0.2 %), tensile strength and percentage elongation values of the specimens are measured. In Shimadzu brand hardness test device, Vickers hardness is measured by applying 0.5 kg of load. Densities of the specimens are determined according to density determination kit and Archimedes principle. Furthermore, pearlite amount of PM steels are calculated by using metallographic point counting method defined by Gladman and Woodhead (1960). In addition; BC, BN and BCN precipitates are identified by the help of point and line EDS.

3. Results and Discussion

3.1. Microstructure

Micrographs of the specimens in Figure 2 shows that, the structure consists of ferrite and pearlite phases in every alloy and there are partially unclosed pores at grain boundaries. Although it is discussed in many references that porosity affects the strength negatively, it is also reported that if pores are very small and have spherical shape, the strength does not decrease (Erden, 2017; Sarıtaş et al. 2007). It is understood from the micrographs that, the grain size decreases as the amount of BN increases in PM steels. In the alloy containing 0.5 wt. % BN, increased mean grain size is observed. For example; while the average grain size of Alloy 1 is 29.7 µm, it decreases to 20.37 µm and 18.98 µm as the amount of BN is increased to 0.1 and 0.2 wt. % respectively, and it is 34.6 µm when the amount of BN is 0.5 wt. %.

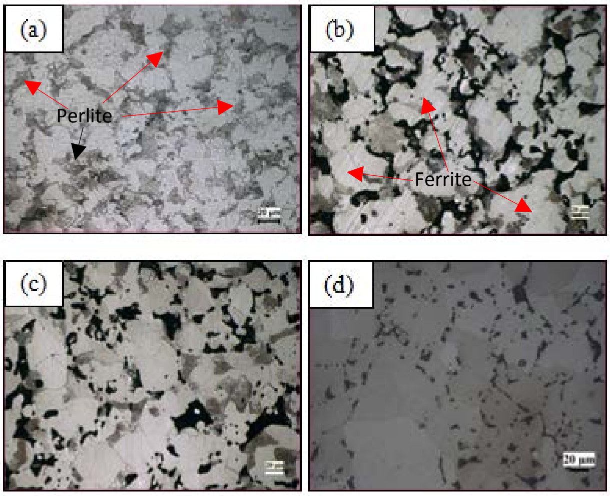

Figure 2. Micrographs of PM steel specimens sintered at 1150º and containing; (a)0 wt. % BN , (b)0.1 wt. % BN , (c)0.2 wt. % BN , and (d)0.5 wt. % (500 x).

Table 2 shows that the grain size decreases as the ratio of BN increases up to 0.2 wt. % This situation takes place as the BN and BC(N) precipitates which are formed during sintering inhibit the growth of austenitic grains (Ollilainen *et al.* 2003). Microalloying elements inhibit the grain growth during austenitization or sintering with carbides and nitrides they form before. Formation of small precipitates during austenitization inhibits the growth of austenitic grains and leads to formation of small ferrite grains during cooling (Gladman, 1997; Sarıtas *et al*. 2007; Ollilainen *et al*. 2003; Xiang-done *et al.* 2013; Bakkali *et al*. 2008).

Table 2. Density, porosity (%), ferrite-pearlite, average grain size values of PM steel specimens.

Compound	Relative density (%)	Porosity (%)	Ferrite (%)	Pearlite (%)	Average grain sizes (µm)
Alloy 1	92	8	79.4	21.6	29.7
Alloy 2	91	9	81.7	18.3	20.4
Alloy 3	91	9	77.8	22.2	18.9
Alloy 4	90	10	95	17.2	34.6

When BN amount is increased from 0.2 to 0.5 wt%, it is observed that the average grain size increase The reason may be the large BN precipitates formed at grain boundaries (Erden, 2016; Schade *et al*. 2012a; Schade *et al*. 2012b). Accumulation of precipitates in grain boundary caused an increase in the amount of pores. Thus, since the formed large precipitates are unable to inhibit the grain growth sufficiently, they lead to a growth in average grain size.

3.2. Mechanical Properties

Figure 3 shows the stress - strain diagram of the sintered specimens while Table 3 gives their yield, strength, elongation and hardness values. It can be seen that increasing BN amount to 0.2 wt. % at 1150 °C sintering temperature increases the yield and tensile strength, elongation and hardness. Carbide, nitride and carbonitride precipitates, formed by boron element, inhibit grain growth and recrystallization of austenite, and thus provide the material with small grains. Since there is more grain boundary in the small grained structure and these grain boundaries block the dislocation movement, strength is improved. Reducing the grain size also contributes to the elongation of material. Besides, the formed precipitates contributed to development of yield strength and tensile strength with various strength developing mechanisms such as precipitate hardening and dispersion hardening (Cuddy and Raley 1983; Ljewellyn and Hudd 1998).

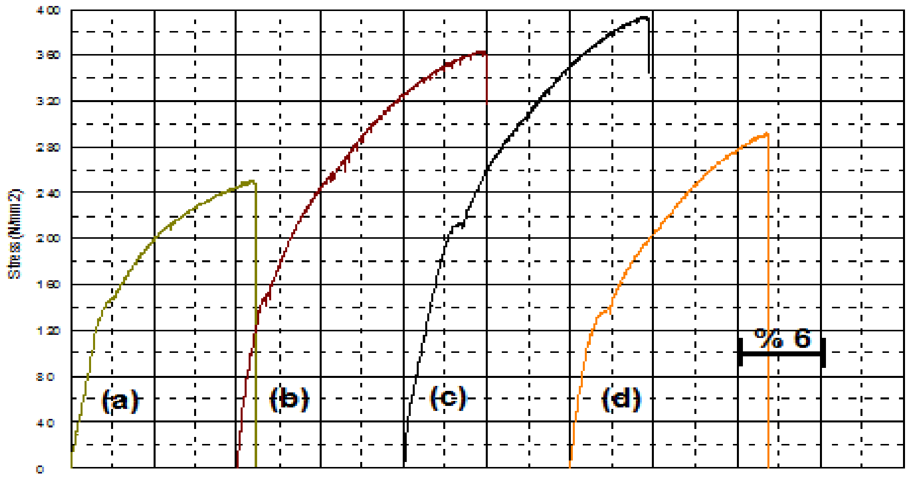

Figure 3. Stress-strain diagrams of PM steels sintered at different temperatures; a) 0 wt. % BN, b) 0.1 wt. % BN, c) 0.2 wt. % BN, d) 0.5 wt. %.

Table 3. Yield strength, tensile strength, percentage elongation and hardness values of PM steel specimens.

Compound	**YS (MPa)**	**UTS (MPa)**	**Elongation (%)**	**Hardness (0.5HV)**
Alloy 1	144	252	13	68
Alloy 2	153	365	17	110
Alloy 3	214	396	17	119
Alloy 4	140	293	14	86

It is considered that precipitates such as BC(N) inhibit the growth of grains during sintering and lead to formation of small austenite grains and consequently increase the strength of material. Erden et al. (2014) produced Ti microalloyed steel by PM method in their studies. They performed the sintering process by keeping the steel specimens at 1150°C for 60 minutes and determined that, as the Ti ratio increased (0.1%-0.2%), there is an increase in yield and tensile strength values. They attributed this increase to formation of precipitates such as TiC(N) during sintering and cooling after sintering. The authors revealed in their study that precipitates such as TiC(N) prevented the growth of grains during sintering and thus caused formation of small austenite grains and consequently increased the strength of materials. In other studies, (Erden *et al*. 2014; Schade *et al*. 2012a; Schade *et al*. 2012b), it is reported that carbides and nitrides which are formed in microalloyed steels improve hardness and strength. In the same studies, it is stated that solid solution hardening remained in low ratios due to precipitation of carbides and nitrides. Increasing the amount of alloy to 0.5 wt%, reduces the yield, tensile, elongation and hardness values. This is due to decrease in strength at high BN levels and formation of BC(N) precipitates at grain boundaries (Schade *et al*. 2012a; Schade *et al*. 2012b). Furthermore, this may be attributed to generally decreasing density at 0.5% alloy addition. Properties such as strength, ductility and conductivity are based on density i.e. porosity and pore structure (Sarıtas et al. 2007). Furthermore, higher precipitation of BN in the PM steel causes over-hardening of precipitate and makes the material fragile and decrease its strength.

3.3. SEM EDS Analysis

SEM pictures of the 0.1 wt. % BN specimen given in Figure 4 shows the existence of precipitates with different sizes. Point EDS analysis results indicate that BC(N) precipitates occur since such precipitates contain boron, carbon and nitrogen elements. Fe_3C precipitates also occur in PM steel since they include iron and carbon. It is known that these precipitates prevent austenite grain growth and recrystallization (Irvine and Pickering 1963). It can be said that such precipitates prevent austenite grain growth and recrystallization, and increase the strength of material by precipitation hardening (Kostryzhev et al. 2014).

Microalloying elements in the solution slightly effects the recrystallization of austenite. Blocking the grain boundary movement by precipitates is much more effective than dissolved atoms (Korchynsky, 2001).

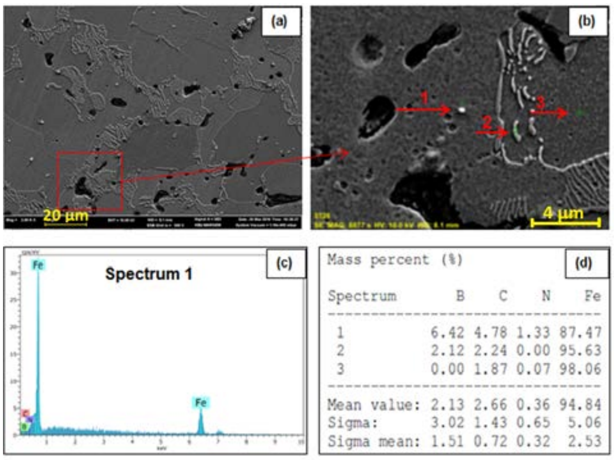

Figure 4. SEM pictures of PM steel specimen sintered at 1150°C and contains 0.1 wt. % BN; (a) X 2000, (b) Point EDSs, (c) Spectrum 1, (d) Point EDS results.

Figure 5 shows the line EDS results of PM steel specimen that is sintered at 1150 °C and contain 0.1 wt. % BN, taken from the matrix and precipitates.

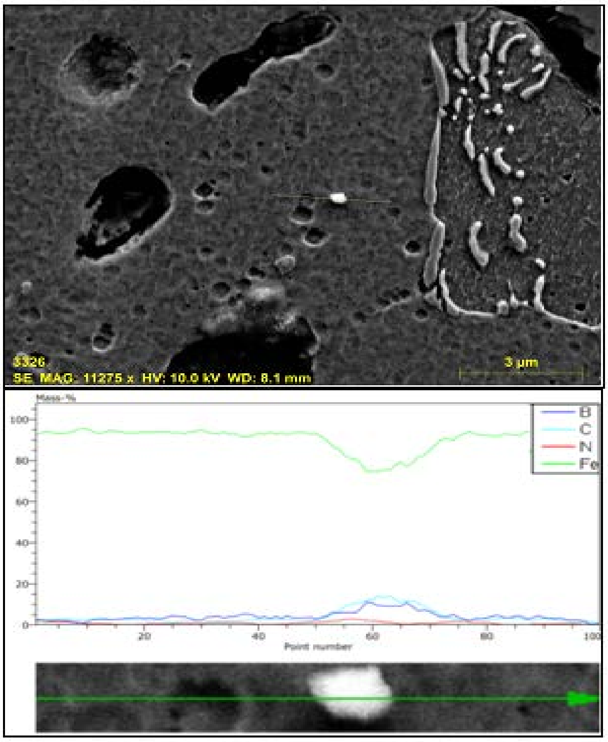

Figure 5. Line EDS results of PM steel specimen that is sintered at 1150 °C and contains 0.1 wt. % BN, taken from the matrix.

The results show a difference between the type and amount of element along the matrix and the line crossing the precipitate. It is observed that the matrix phase is rich in iron whereas round shape precipitate is rich in boron element. Moreover, there is a sharp increase at the crossing point of the line coming from the matrix with the precipitate. Point and line EDS analyses results obtained from this study reveal that precipitates such as BC, BN and BC(N) are formed in PM steels.

For example, Gündüz et al., studies shows the XRD precipitate peaks of the filter residue of Alloy 3 (Fe-0.25C-0.075Nb-0.075Al) (Gündüz at al., 2016). This study, in an alloy containing aluminium and without titanium or niobium, AlN precipitation occurs in the austenitic or ferritic regions. Several investigations report a fine precipitation (≪1 µm) with a large number density of nitride particles for steels containing between 29.96 mg/L and 299 mg/Lnitrogen (Radis and Kozeschnik, 2010). This precipitation is known to have significant effects upon recrystallization and austenite grain growth (Irvine and Pickering, 1963).

The conclusion indicate a difference between the type and amount of element along the matrix and the line crossing the precipitate such as BC, BN and BC(N) are formed in PM steels.

4. *Conclusion*

BN alloyed PM steel with different amount of BN (0.1 – 0.5%) is produced by cold pressing and sintering in argon atmosphere, and the following results were obtained from this study.

1. Fe matrix PM steels containing BN can be produced by powder metallurgy method. The solid solution hardening and precipitate hardening occurring during the sintering or during the cooling after sintering increased the strength of steel.

2. PM steels with 0.1 and 0.2 wt. % BN added have smaller grain structure compared to non-alloyed PM steels. This is due to inhibition of grain growth by carbides and nitrides which are formed by the alloy elements. In microstructure examinations of specimens having 0.5 wt. % BN ratio, it is observed that the pearlite ratio is very low (17.2 %) in their structure. Furthermore, a growth in the grain size was observed. The reason for that may be the formation of BC(N) precipitates in grain boundaries.

3. In general, as the amount of BN increased to 0.2 wt. %, an increase is observed in yield strength, tensile strength, elongation and hardness values of the steels. This is the result of formation of precipitates such as BC(N) during sintering and during cooling after sintering. Such precipitates prevent the growth of grain during sintering and lead to formation of small austenite grains; and consequently improve the strength of the material.

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