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MECHANICAL AND WEAR PROPERTIES OF PRE-ALLOYED MOLYBDENUM P/M STEELS WITH NICKEL ADDITION

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Abstract

The aim of this study is to understand the effect of nickel addition on mechanical and wear properties of molybdenum and copper alloyed P/M steel. Specimens with three different nickel contents were pressed under 400 MPa and sintered at 1120 ºC for 30 minutes then rapidly cooled. Microstructures and mechanical properties (bending strength, hardness and wear properties) of the sintered specimens were investigated in detail. Metallographical investigations showed that the microstructures of consolidated specimens consist of tempered martensite, bainite, retained austenite and pores. It is also reported that the amount of pores varies depending on the nickel concentration of the alloys. Hardness of the alloys increases with increasing nickel content. Specimens containing 2 % nickel showed minimum pore quantity and maximum wear resistance. The wear mechanism changed from abrasive wear at low nickel content to adhesive wear at higher nickel content.

Keywords: Powder Metallurgy, Sinter Hardening, Wear, Microstructure

1. Introduction

Because of its advantages compared to conventional methods, powder metallurgy (P/M) is preferred for numerous applications. In the case of complex shaped components and parts with small dimensions, the easy control of the microstructure and low production costs are some of the advantages of powder metallurgy [1-4].

In the last decade, many authors have studied the improvement of mechanical properties of P/M parts with proper alloy

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designs and development of new technologies to reduce production costs [5]. There are a number of alloying elements, such as Mo and Cr, to enhance the mechanical properties of the P/M steels [6]. Mo provides solution strengthening and increases the hardenability of the P/M materials [7-8]. Molybdenum alloyed steels have been fully studied during the 1980s. At the begining, molybdenum steels were alloyed with chromium to enhance mechanical and wear properties. New material groups have beeen developed using Fe-1.5% Mo as the raw alloy. At this point nickel is used for dimensional precision applications and high autoquenching capabilities [9]. These pre-alloyed powders are considered based on compressibility, sinterability and transformation of austenite in order to achieve the desired microstructure. The addition of nickel to Fe-Mo alloys increases sintered density, hardenability and reduces sintering activation energy. One of the main advantages of Ni and Mo is the development of bainitic and martensitic structure when standard conditions of cooling rate are used [7, 10-11]. Martensitic transformation in many steels has recently been studied extensively due to its desirable technological features in industry and technology [12].

Besides the alloying contents, cooling rate from sintering temperature has a strong effect on the microstructure. Conventional powder metallurgy has slow cooling rates, and the sintered materials require heat treatment following densification. The specimens used in this study are produced by the sinter hardening method. Sinter hardening is a highly effective and low cost method for producing high performance sintered materials [13-14]. Using this method both cost, and the mechanical properties, of the products can be arranged for different applications. With lower cooling rates compared to the quenching, sinter hardening provides high dimensional accurateness and minimum distortions on the components [15]. The cooling in the furnace must be rapid enough to obtain martensite and bainite in the microstructure. Depending on the dimensions and chemical composition of the component, the cooling rates can be reached up to 50 ºC/s [13, 16].

This study aims to investigate the effect of nickel on the mechanical and tribological properties of the molybdenum and copper pre-alloyed steels in the same heating and cooling conditions.

2. Experimental

Figure 1 shows representative scanning electron microscope images of the molybdenum and copper pre-alloyed $Fe_2Cu_{1.5}Mo_xNi$ powders. Specimens with 3 different nickel concentration were used for this study (0, 2, 4 %-wt). Chemical compositions of the powders are given in Table 1. The powders were mixed with 0,8 % UF4 graphite and 0,5 % zinc stearate, and then pressed to obtain green compacts.

All samples were sintered at 1120 ^{o}C for 30 min then cooled in the same furnace. The furnace was equipped with a controlled cooling zone to accelerate cooling from the sintering temperature, to obtain a martensitic and bainitic structure. Following the sintering and hardening process, densities of the compacts were measured using

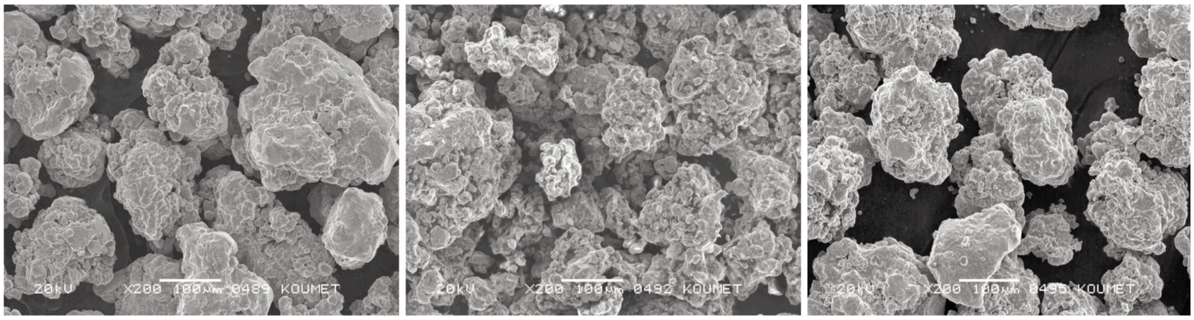

Figure 1. SEM micrographs of the powders, a) nickel-free, b) 2 % nickel and c) 4 % nickel

Table 1. Chemical compositions of the powders

Material	Fe	C	Mo	Cu	Ni
Nickel free	95,30	0,79	1,50	2,28	0,05
2 % Nickel	93,00	0,84	1,53	2,30	2,23
4 % Nickel	91,10	0,80	1,51	2,13	4,34

Archimedes' principle, and pore characterization was made by using Quantimet 501 image analyzing software taking 10 samples for each alloy group. To determine defect sensibility of the specimens, 3 point bending tests were applied. For each alloy group 10 specimens were tested in bending tests, and the Weibull statistic, which is an emerging technique to characterize the mechanical reliability of sintered materials, was used to evaluate the results [17, 18] . Wear tests were carried out on a ball on disc type tribometer under dry sliding condition in air atmosphere at room temperature using AISI 52100 steel ball 5 mm in diameter. All specimens were mechanically polished before wear tests. The normal loads for the tests were 20 and 40 N. The ball speed during wear tests in all cases was 300 rpm and the sliding distance was 1000 m. During the tests friction coefficient values for specimens were recorded continuously. The specimens were thoroughly cleaned with alcohol before and after the tests, and then dried with a hot air blower. The weight loss during wear tests was measured using a Precisa XB220A balance with the resolution of ± 0,1 mg. The Fisherscope HV100 hardness measurer was used for determining hardness of sintered specimens. Leitz light microscope and Jeol 6060 scanning electron microscope were used in microstructural investigations.

3. Results

3.1 Microstructural Characterization

Nickel concentration in the specimens higly effects the density of the sintered specimens. Figure 2 shows the variation in the pore distribution of polished samples. Densities of the specimens were measured as 6,8, 7,7 and 7,8 gr/cm^3 respectively for 0, 2 and 4% nickel containing alloys. Quantitative analysis results of the polished surfaces are shown in Table 2. Results showed that the maximum pore size is 54 µm, 20 µm and 34 µm for the alloys with 0, 2 and 4 % nickel, respectively. The Nickel-free sample also has the highest degree of porosity with 13 vol.-% as compared to other compositions. The sample with 2 % nickel showed the lowest porosity ratio.

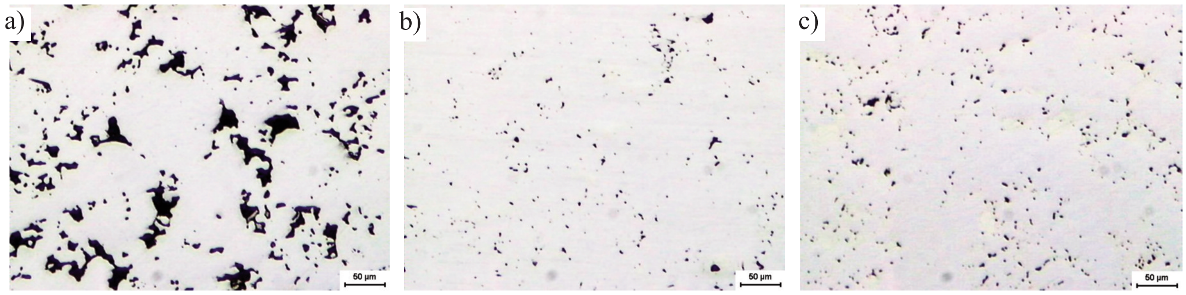

Figure 2. Polished microstructures of specimens, a) nickel free, b) 2 % nickel and c) 4 % nickel

Table 2. Pore characterization results of polished surface of the samples

Sample	Porosity (Vol. %)	Equal Spherical Diameter (µm)	Max. Pore Size (µm)
Nickel free	13.1	7.6	54
2 % Ni	2.9	3.1	20
4 % Ni	8.1	4.7	34

All specimens were etched with 3 % nital (3 % nitric acid + 97 % ethyl alcohol) reagent to reveal the microstructures. Etched microstructures of specimens are given in Figure 3. It is difficult to determine the phases of each of the microstructure, due to the close colors and fine morphology. For this reason we also measured the microhardness of each phase. The results clearly demonstrated that the hardness of bainite and martensite (bright zones) was 395 and 569 HV0.1, respectively. After the correlations, the microstructure of the nickel-free specimen consists of 75 % bainite and 10.4 % martensite, which are generally located around the pores. In the specimen with 2 % nickel, the microstructure mostly consists of bainite (86 %) and some martensite (10,6 %). In the specimen with highest nickel concentration, the martensite and bainite amounts were measured as 76,3 % and 15,5 %, respectively. The austenite phase becomes more stable with increasing nickel content and retained austenite amount increases. The amount of retained austenite was measured by using the magnetic saturation method, which is useful when the metallographic techniques give an imprecise result [19-20]. The results was 1,5, 0,5 and 6,9 % austenite phase, respectively, for specimens with 0, 2 and 4 % nickel.

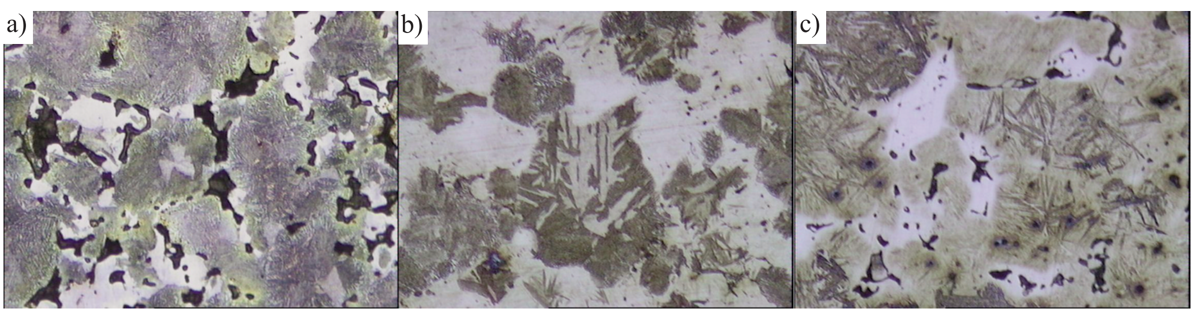

Figure 3. Etched microstructures, a) nickel-free, b) 2 % nickel and c) 4 % nickel.

3.2 Hardness and Toughness

The variation of porosity with nickel addition also has an important effect on the hardness. Density and hardness relation of tested specimens is given in Figure 4. With the addition of 2 % nickel to the alloy, density of the sintered specimen increases significantly. However increasing nickel content to 4 % has little effect on the density. Avarage hardness values of specimens are obtained as 246, 253,5 and 311,5 $HV_{0.1}$, respectively, for alloys with 0, 2, and 4 % nickel alloys.

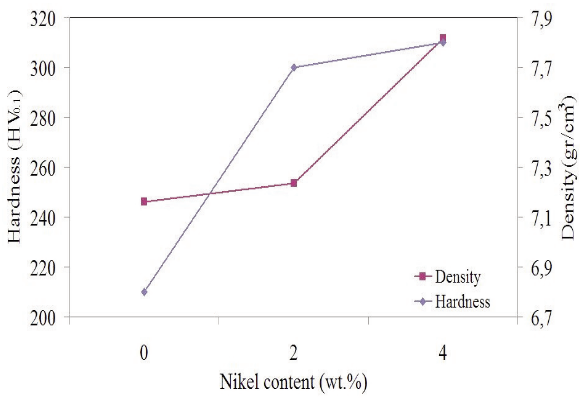

Figure 4. Effect of nickel content on hardness and density.

After the density measurements, bending tests were carried out. The Weibull statistical model is applied to the results of the 3 point bending tests. The Weibull statistical calculation is generally used in the powder metalurgical specimens with high porosity to obtain defect sensitivity. In this method, probability of 50 % fracture is used as the toughness value. The slope (m) of the diagram shows the sensivity to the surface and microstructural defects.

$$P = 1 - \frac{1}{e^{\frac{V}{V_0}\left(\frac{\sigma}{\sigma_0}\right)^m}} \quad (1)$$

$$P = \frac{n}{N+1} \quad (2)$$

In these equations P: probability of fracture, V: Volume, V_0 : Unit volume with only one defect, σ: stress, σ_0: stress per unit volume, m: Weibull constant (slope of the line), n: number of broken samples, N: number of total samples [21].

The bending test results and Weibull statistics showed that the bending strength for 50 % fracture probability are 947 MPa, 1166 MPa and 1156 MPa for 0, 2 and 4 % nickel containing specimens, respectively. Bending strength of the specimens increases with adding 2 % nickel, and stays nearly constant for increasing the nickel content to 4 %. However fracture probability of the 2 % nickel containing specimen has minimum defect sensitivity. The slopes of the lines for 0 and 4 % nickel containing specimens are higher than the 2 % nickel containing specimen. To show the differencies between the slope of the lines of each alloy with different nickel contents, fracture probability diagrams were plotted in Figure 5.

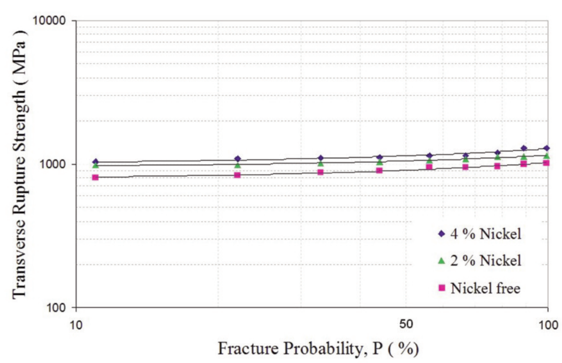

Figure 5. Fracture probability plots, a) nickel-free , b) 2 % nickel and c) 4 % nickel

3.3 Wear Tests

Porosity level and microstructure of the materials effected the wear test results. The 2 % nickel containing specimen, with lowest defect sensitivity, has higher wear resistance. The test results with 20 N and 40 N loads showed that the nickel-free specimen with the highest porosity has the highest weight loss ratio. The effect of nickel content on the mass loss of the specimens during dry sliding wear is illustrated in Figure 6. With a decrease in porosity, wear weight loss decreases gradually (2 % Nickel,) and weight loss increased with increased retained austenite content (4 % Nickel).

During the wear tests, the friction coefficients are recorded simultaneously. The values of the friction coefficients show a similar relation with the weight loss data. Low hardness and high porosity of the nickel-free specimen leads to a higher friction coefficient value for this specimen. With increasing nickel content in the specimens, the hardness increases and the friction coefficient of the specimens decreases (Figure 7).

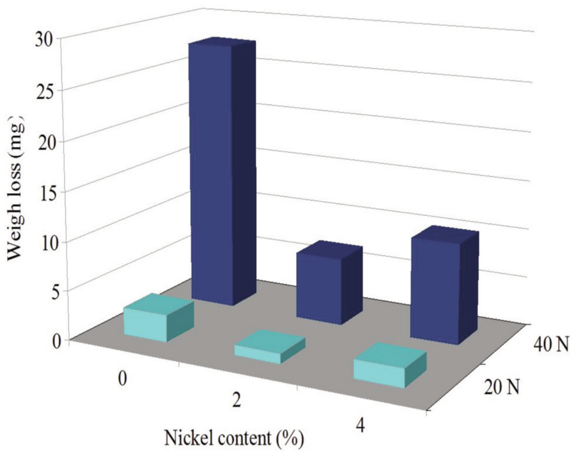

Figure 6. Effect of nickel content on weight loss during dry sliding

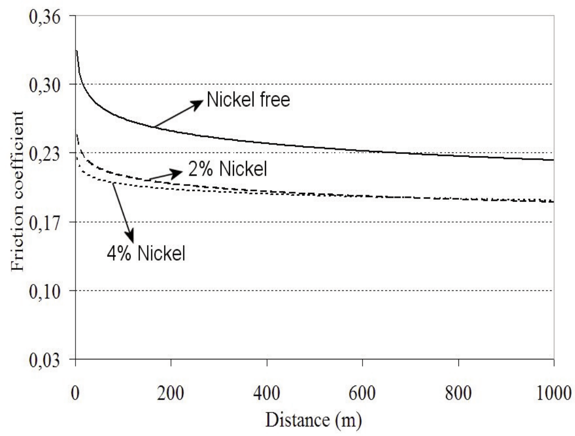

Figure 7. Effect of nickel content on friction coefficients

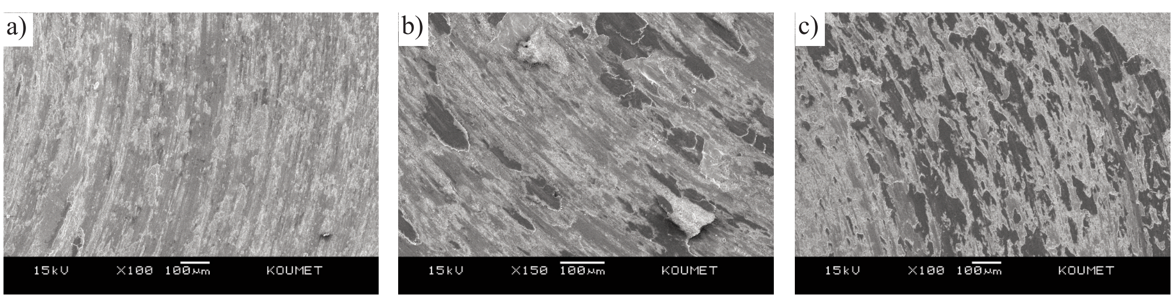

Figure 8. SEM micrographs of the worn surfaces of the specimens (a) nickel-free, (b) 2 % nickel and (c) 4 % nickel.

3.4 Worn Surface Investigations

To understand the wear mechanisms, the worn surface of the samples corresponding to the applied load of 40 N were investigated by scanning electron microscopy and shown in Figure 8. The SEM images show that during wear test some plastic deformation occurs in the steel ball-specimen interface. In the nickel-free specimen, the wear tracks are very smooth while with increasing nickel the wear tracks exhibit more roughness. In the nickel-free specimens, the dominant wear mechanism is abrasive wear, and with increasing nickel content the dominant mechanism becomes adhesive wear. In specimens with nickel content, the interaction between the steel ball and specimen increases and the wear mechanism becomes more adhesive and leaves rough wear tracks.

4. Conclusions

The effect of nickel on microstructure and dry sliding wear properties of pre-alloyed molybdenum P/M steel had been studied in this work and the following conclusions were evaluated. The results of this study confirm that nickel content has effectively improved the wear and toughness properties of the powder metallurgical material. The microstructures of all the samples consist of martensite, bainite and retained austenite. Martensitic structure and retained austenite content increased with increasing nickel amount. The wear resistance of the material is increased with increasing the content of nickel. The wear mechanism of the material with low nickel content was abrasive. With addition of 4 % nickel the retained austenite in the structure increases and adhesion appeared to be the most important wear mechanism with intense plastic deformation. The wear experiments showed that 2 wt.% nickel content has lowest weight loss. An optimum nickel content (2 wt.-%) has been defined where the lowest adhesive wear rate and highest bending strenght is obtained.

References

[1] X. Dai-hong,Y. Tie-chui,O. Xiao-qin, H. Yue-hui, Trans. Nonferrous Met. Soc. China, 21 (6) (2011) 1269.

[2] N. Talijan, V. Cosovic, J. Stajic-Trosic, A. Grujic, D. Zivkovic, E. Romhanji, J. Min. Metall. Sect B-Metall., 43 (2) B (2007) 171.

[3] I. Bailon-Poujol, J. Bailon, G. L'Esperance, Powder Technol.,210 (2011) 267.

[4] A. Molinari, E. Santuliana, I. Cristofolini, A. Rao, S. Libardi, P. Marconi, Mat. Sci. Eng. A-Struct., 528 (2011) 2904.

[5] T. Ramprabhu, S. Sundar Sriram, K. Boopathy, K. S. Narasimhan, U. Ramamurty, Metal Powder Report, 66 (2011) 28.

[6] D. Shanmugasundaram, R. Chandramouli, Mater. Design., 30 (2009) 3444.

[7] O. Y. Kalashnikova, L. P. Sorkin, Met. Sci. Heat Treat., 49 (2007) 288.

[8] R. Yılmaz, A. Gökçe, H. Kapdıbaş, Adv. Mat. Res., 23 (2007) 71.

[9] N. Candela, F. Velasco, M. A. Martinez, J. M. Torralba, J. Mater. Process Tech., 168 (2005) 505.

[10] G. Sun, Y. Zhang, C. Li, K. Luo, X. Tao, P. Li, Mater. Design., 31 (2010) 2737.

[11] M. rosso, L. a. Dobrzanski, J. Otraba, M. A. Grande, JAMME, 35 (2009) 117.

[12] E. Güler, M. Güler, J. Min. Metall. Sect B-Metall., in press, (2012).

[13] S. Karagöz, R. Yamanoglu, A. Yilmaz, H. Atapek, Materialprufung, 52 (2010) 2010.

[14] L. A. Dobrzanski, J. Otreba, Z. Brytan, M. Rosso, JAMME, 24, 187-190, 2007.

[15] Hatami, S., Malakizadi, A., Nyborg, L., Wallin, D., J. Mater. Process. Tech., 210 (2010) 1180.

[16] Lindsley, B., Int. J. Powder Metall., 44 (2006) 21.

[17] J. M. Torralba, F. Velasco, J. M. Ruiz-Roman, L. E. G. Cambronero, J. M. Ruiz-Prieto, J. Mater. Sci. Lett., 15 (1996) 2105.

[18] J. Espinoza-Cuadra, G. Garcia-Garcia, H. Mancha-Molinar, Mater. Design, 28 (2007) 1038.

[19] C. Ajus, S. S. M. Tavares, M. R. Silva., R. R. A. Corte, Revista Materia, 14 (2009) 993.

[20] B. D. Cullity, C. D. Graham, Introduction to Magnetic Materials, Seconda Edition, John Wiley & Sons, New Jersey, 2009.

[21] S. Karagöz, M. Zeren, Proceedings of the Symposium of Research, (1997) 145.