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On the tribological behavior of nanoalumina reinforced low alloy sintered steel

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ABSTRACT

Powder metallurgy (PM) technique offers progress of new material processing for applications requiring various combinations of properties. Demanding for applying ceramic materials in tribological concept is increasingly growing over last two decades. Unique characteristic of ceramic materials such as low density, high hardness, low thermal expansion, high corrosion and tribological resistance is the rudimentary reason. In this study, different weight percentage of alumina nanoparticles was added to low alloy powder steel (Astaloy 85Mo) as reinforcement agent. Microstructure and tribological behavior of the metal matrix composite has investigated at dry condition and room temperature for different loads by reciprocating tribometer. Sintered specimens possess homogenous microstructure with bainitic and partial ferrite feature in retained austenite matrix. Outcomes show improvement in wear resistance by increasing of alumina nanoparticles containing 3 wt.%, porosity level of 15.38% and micro hardness of 105.4 HV which demonstrates the best wear resistance properties. Tribological behavior of PM steel parts is so complex due to existing pores. Not only do surface pores deteriorate the wear resistant as inherent characteristic but also the properties could enhance at optimum porosity level. An important role of surface porosities which have crucial influence on decreasing wear rate is trapping wear debris causes severe wear. Mixed mode of abrasive, adhesive and oxidation mechanisms were distinguishing according to electron image analysis.

1. Introduction

Powder metallurgy (PM) methods, among different processes of manufacturing and forming of materials, have got prominent role in decreasing raw materials consuming and saving energy developing in many industry fields especially automotive due to technical and economic advantages. PM processes have versatility as well producing abundant range of materials, microstructures and considered properties [1,2,3]. Typical materials fabricated by PM process are exposed to high and complicated loads. Quality, applicability and life time of many components are limited by static (tensile, three point bend test) and dynamic stresses (fatigue and tribological behavior). Tribological attribute is a complex matter including deformation, material relocation and failure, impose great financial burden every year [4,5].

An obvious difference between sintered component and fully dense materials is existence of pores remained after the pressing and sintering of metal powders. Pores prime to reducing the wear resistance, as a mechanical strength representative, and also enhance it by acting as lubricant storage releasing during utilizing of the specimen. Furthermore, the porosity possess efficient role in process of removing wear debris from the sliding interfaces, as mentioned by some authors [6–8]. Consequently, tribological treatment of sintered material is so complex and depends on many factors mainly wear conditions.

Many techniques have been carried out to ameliorate wear resistance like surface heat treatment, sinter hardening, plasma nitriding, and carburizing [9–11]. A crucial method to fabricate wear resistant material is adding hard particles to a tough matrix in order to generate metal matrix composites (MMC). Iron-based composites containing some volume fraction of carbide, nitride, boride, and/or oxide particles are regularly MMCs for applications operating in wear condition [12]. Hard ceramics-reinforced MMCs correlate combination of properties and in situ fabrication are so eminent. Steel matrix composite reinforced by alumina nanoparticles is a suitable candidate ones [13].

Alumina ceramics have enjoyed excellent resistance to oxidation, corrosion and wear due to its structure. Although low sintering temperature comparing with other ceramics, made it more functional and economical but, the potential of alumina ceramics in many applications is limited by their relatively high brittleness and low toughness. Applying alumina nanoparticles as a reinforcement in the MMCs will dwindle constrains and supply variety of

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applications owing to combining suitable properties of both classes of materials [14].

In this experiment, not only the effect of adding diverse weight percentage (1–5 wt.%) of alumina nanoparticles on the tribological behavior of a low alloy sintered steel entitled as Astaloy 85Mo was investigated but also microstructure characterization is under consideration. The main aim of the study is balancing of mechanical properties generated by adding an optimum percentage of alumina nanoparticles instead of increasing density. This technique could decline the weight of sintered part used in automobile industry. Finally, relationship between microstructure and wear properties of the MMC will be studied.

2. Experimental details

2.1. Materials and fabrication

The partially diffusion pre-alloyed powders (with commercial name of Astaloy 85Mo) made by Höganäs Sweden, mixed with 1% micro-wax as lubricant, was chosen for matrix composite. This powder produced through water atomization process, comprising high compressibility and homogenous microstructure after sintering. Different weight percentage (1, 3 and 5 wt.%) of alumina nanoparticles were added as the reinforcement agents. Alumina-α is the hardest and densest form of aluminum oxide with density of 3.97 g/cm^3. It is the most economic ceramic reinforcement in comparison to others carbides (e.g. TiC and SiC) or oxides nanoparticles. Properties of used powders in this research are given in Table 1. Different percentages of alumina nanoparticles were mixed homogeneously with the base powder using tumbling mixer for 20 min. Consequently, samples were cold pressed at 450 MPa by single axial double action hydraulic machine (100 tons capacity) according to ASTM: E8/E8M-13a. Sintering process was carried out in 1080 °C for 30 min in a N_2–10%H_2 reducing atmosphere with the cooling rate of 0.5 °C/s. The ASTM: B962-13 was selected in order to measure densities and porosity volume percentage of sintered samples. Depending on alumina nanoparticle weight percentages, different volume proportions of porosity were obtained. To identify the effect of reinforcement amounts on composites hardness, Vickers hardness measurements were carried out according to ASTM: E384-11e1 on the punch surface of the samples. Hardness values have got divers amounts in PM parts due to existence of pores. Subsequently, hardness tests were done for five indentations and average amounts were reported.

2.2. Wear test

Dry wear testing was performed at ambient temperature in humidity of 30–35% by pin on flat method. The pin was made of AISI52100 steel with hardness of 64 HRC and diameter of 5 mm. Chemical compositions and some selected properties of the abrasive pin are given in Table 2. Normal loads of 25, 35 and 45 N and a constant sliding velocity correspond to frequency of 25 Hz were selected as wear test parameters. This frequency is approximately equivalent to the velocity of 0.15 m/s. The wear test was done on the punch surface of the samples for 1000 m. After each 200 m, samples were taken out of the sample holder then were cleaned and weighted. Afterward, they were remounted in the wear tester at the same location. To improve results accuracy, each experiment for the wear test was accomplished 5 times and the average of results was taken into consideration.

2.3. Light optic microscopy

Samples were mechanically polished and low rate and pressure was selected for grinding and polishing for reduction of eventual effect on porosity morphology. Metallographic observations were carried out using a light optic microscope (LOM) IMI-420 that was made on un-etched and etched micrographs in order to identify microstructure morphology. Metallographic samples used for identification of microstructure were etched with 3% Nital.

2.4. Electron microscopy

Before mixing powders, size and distribution of alumina nanoparticles were examined by transmission electron microscopy TEM

Table 1
Characteristics of powders used in this study.

Material	Particle size	Purity	Company made
(Al_2O_3-α) alumina – alpha	20–30 nm	+99%>	Degussa-Germany
(Astaloy 85 Mo) PM iron base	45 µm	Fe–0/85Mo–0/45C	Höganäs-Sweden

Table 2
Chemical composition and some selected properties of the pin used in the wear test.

Fe (wt.%)	C (wt.%)	Cr (wt.%)	Mn (wt.%)	P (wt.%)	Si (wt.%)	S (wt.%)	Elastic modulus (GPa)	Hardness (HRC)
Bal.	0.98–1.1	1.3–1.6	0.25–0.45	0.025	0.15–0.35	0.025	205	64

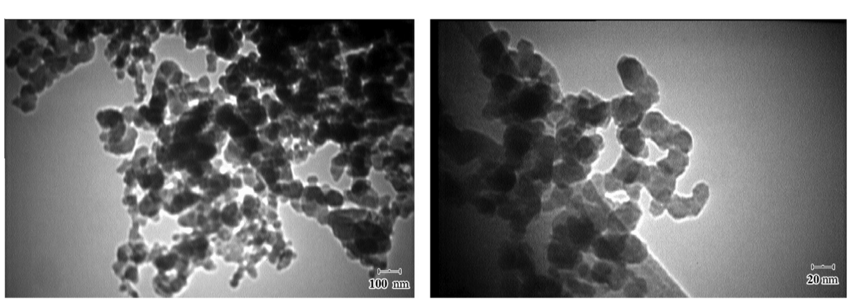

Fig. 1. TEM micrograph of alumina particles.

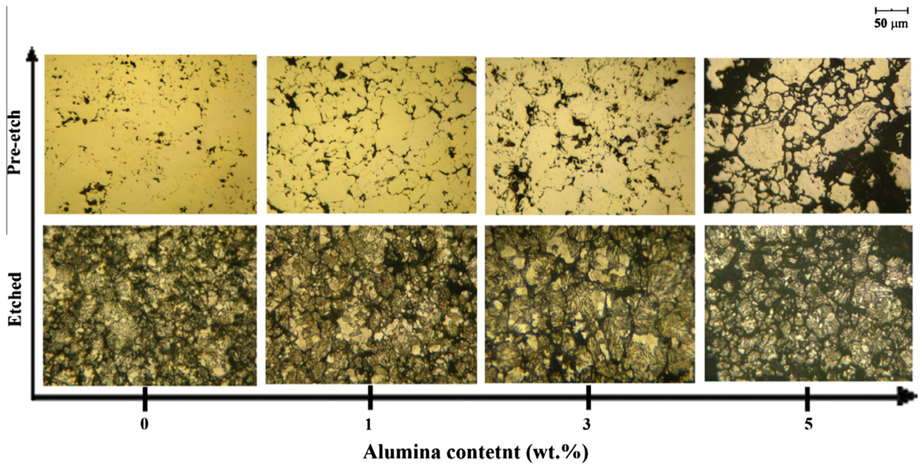

Fig. 2. Typical microstructure of pre-etch and etched specimen versus different alumina content.

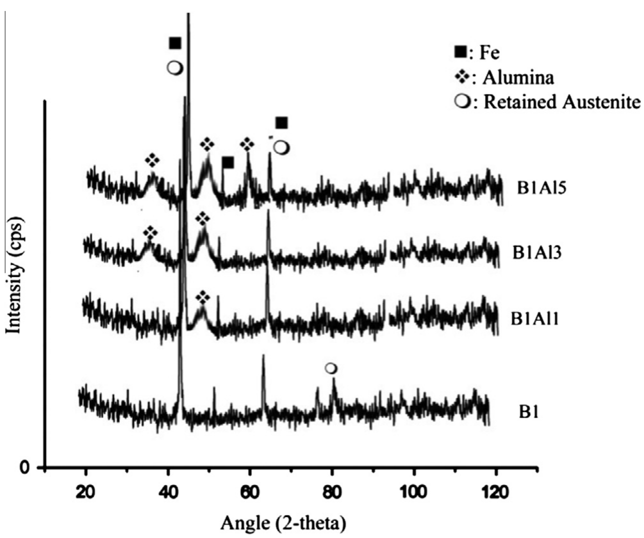

Fig. 3. XRD patterns of sintered metal composite.

(LEO/ZIESS, Germany) operated at the voltage of 80 kV. To understand wear mechanisms, worn surface were analyzed by SEM (JSM-6360, JEOL, Japan) equipped with an energy-dispersive X-ray spectrometer (EDS) for element distributions for distinguishing of dominant wear mechanism.

2.5. XRD analysis

The structural constituents were identified by X-ray diffraction (XRD, Shimadzu (x2), Japan) operated at the voltage of 40 kV, current of 40 mA and scanning speed of 3°/min for phase identity using Cu Kα radiation.

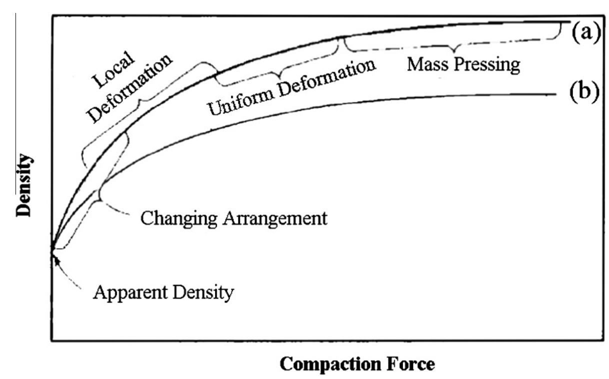

Fig. 4. Schematic of changing density in metallic powders compacting process and main steps, (a) absence of nanoparticles and (b) presence of nanoparticles.

3. Results and discussions

3.1. Microstructural characterization

Before mixing powders, TEM analysis has carried out to define particle size and dispersion of alumina nanoparticles having approximately 20–30 nm in size and the particles are agglomerated as follows by Fig. 1. Bondings among alumina nanoparticles prohibit slipping on each other and lead to decrease density when utilize as reinforcing agent. Fig. 2 shows the typical homogenous microstructure of sintered samples. Important characteristic of molybdenum is shifting eutectoid transformation point to higher temperatures and lowers the eutectoid carbon content. The major influence of this alloying element is actually on the eutectoid composition rather than eutectoid temperature [20]. Pre-alloyed sintered steel (Astaloy 85Mo) has got homogenous microstructure. This microstructure contains bainitic and some trace of ferrite in retained austenite matrix with different morphologies. Difference phases are characterized by XRD analysis demonstrates in Fig. 3.

Table 3
Characteristics of specimens.

Code	Composition	Sintering density (g/cm^3) ± 0.05	Porosity (%) ± 0.05	Hardness (HV5)
B_1	Astaloy 85 Mo + 0 wt.% alumina	7.1	8.97	122.86
B_1Al_1	Astaloy 85 Mo + 1 wt.% alumina	6.8	12.82	116.93
B_1Al_3	Astaloy 85 Mo + 3 wt.% alumina	6.6	15.38	105.4
B_1Al_5	Astaloy 85 Mo + 5 wt.% alumina	6.4	17.94	99.83

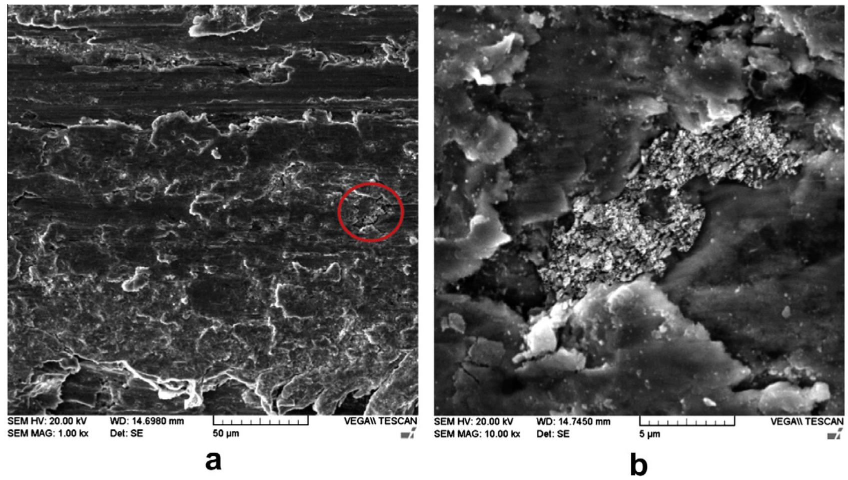

Fig. 5. (a) Large porosity in worn surface of the B1Al1 and (b) high magnification of circle indication showing debris filled the porosity.

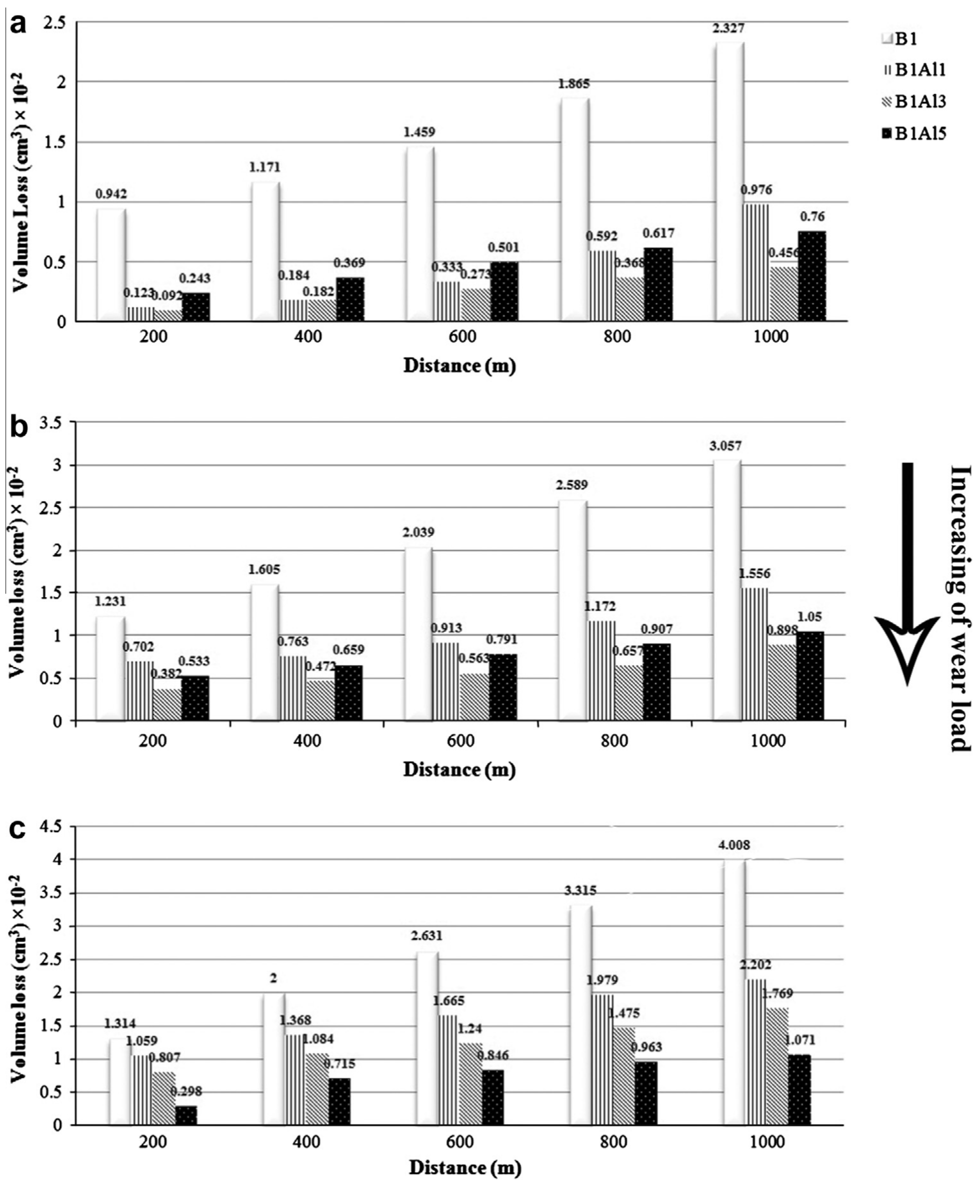

Fig. 6. Variation of volume loss with sliding distance in load of (a) 25 N, (b) 35 N and (c) 45 N.

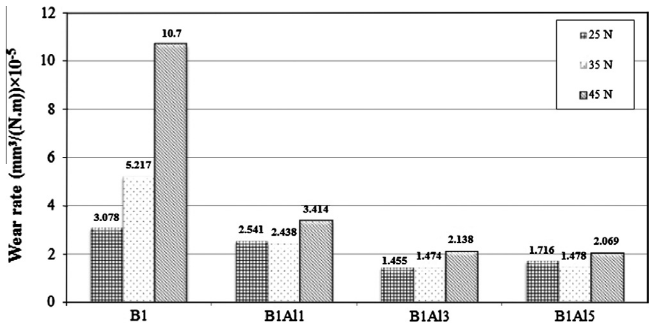

Fig. 7. Variation of wear rate of specimens with varying loads and alumina content at a constant sliding.

According to the pattern, peaks of alumina nanoparticle have detected by increasing of its content. Also, the sign of retained austenite shows itself in all sintered sample confirm by XRD. This microstructure is agreed to Moghaddam et al. [21]. As expected, hardness declined as density decreased (Table 3). The hardness of PM samples is affected by structural features like microstructure and porosity decreasing effective cross-section. Actually, porosity level is the chief factor to determine PM sample mechanical properties as reported by some authors [20,22,23]. Increasing nanoparticles in specimens lowers density and consequently hardness value.

3.2. Porosity

Table 3 illustrates porosity levels versus adding different amount of alumina nanoparticles. Fig. 2 (pre-etched) demonstrates distribution of surface pores of specimens. As it is shown, percentage of open pores increases with adding some amount of alumina nanoparticles. Density and porosity level of PM samples hinges on compact pressure, powders chemical composition, sintering time and temperature as well as particle size [15]. Generally, more irregular pores, with a higher degree of interconnectivity and pore clustering is the result of an increase in porosity level. Increasing of former and later phenomenon lead to rising in strain localization, dropping the strength and ductility of the PM samples [16]. Total pore size was smaller and the pore shape was more circular with decreasing porosity as obvious in Fig. 2. In another words, increasing density resulted in an increase in the circularity of the pores. In specimens with low porosity level and small pore size, trapping debris by the pores changed to be done difficulty. At the highest porosity, samples with density of 6.4 g/cm^3 (B1Al5), the pore shape distribution was very wide, with a slightly greater fraction of irregular pores than circular ones. At the lowest porosity, samples with 7.1 g/cm^3 density (B1), the shape distribution was twisted toward more circular pores.

The nature of hard and fine alumina nanoparticles is responsible for density modification. Fig. 4(a and b) illustrates density versus compact pressure for metallic powders in absence and in presence of nanoparticles. These particles will shorten first steps of deformation powders in compacting process. In fact, nanoparticles agglomerations perched in empty places among powders during compacting and altered compressibility. Consequently, the density of the MMC specimens will decrease. Presence of pores could be effective due to lowering wear rate at dry condition acting as a sink which gather wear debris from surface and improved wear resistance by reduction in abrasive wear [4,17]. Gathering wear debris from surface leads to a drop in wear rate due to reduction of outlined features [18]:

(1) Contact pressure comparing with another PM sample with open pores that wear debris was not trapped in.
(2) Possibility of forming and agglomerating of greater debris during wear.
(3) Plastic deformation near pores leading to generate metallic debris.

It should be noticed that mechanism of trapping wear debris by pores incredibly depends on its level. Thus this mechanism will neutralize when pores become too large or pore clusters occurring. Also, this mechanism will be diminished in absence of pores [19]. Fig. 5 shows a large porosity in worn surface of the sample coded with B1Al1 trapped metallic and oxide debris. White-dot shapes distributed in Fig. 5(b) represent alumina reinforcement in sintered sample.

3.3. Tribological behavior

Variation of volume loss with sliding distances of 200, 400, 600 and 1000 m for samples with the loads of 25, 35 and 45 N, are shown in Fig. 6. Growing volume loss is outcome of increasing in applied load. These changes in volume loss are close at 25 N and 35 N but it rises dramatically at 45 N. The effects of the applied load at a constant sliding velocity of 0.15 m/s on the wear rate of specimens were plotted in Fig. 7. Wear rate was calculated by Archard equation [24]. SEM micrographs of worn surfaces at different loads and alumina content are illustrated in Fig. 8. Although wear in PM steel samples is more complicated than full dense steel, but it can be seen in rough steels that with increasing of applied

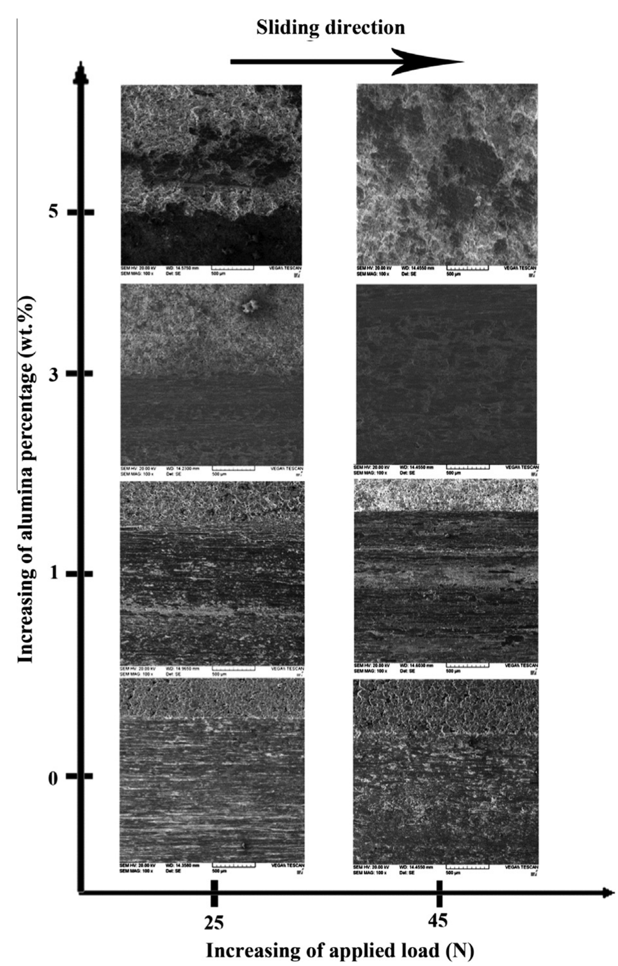

Fig. 8. SEM micrograph of worn surface of the specimens at different loads versus diverse alumina content.

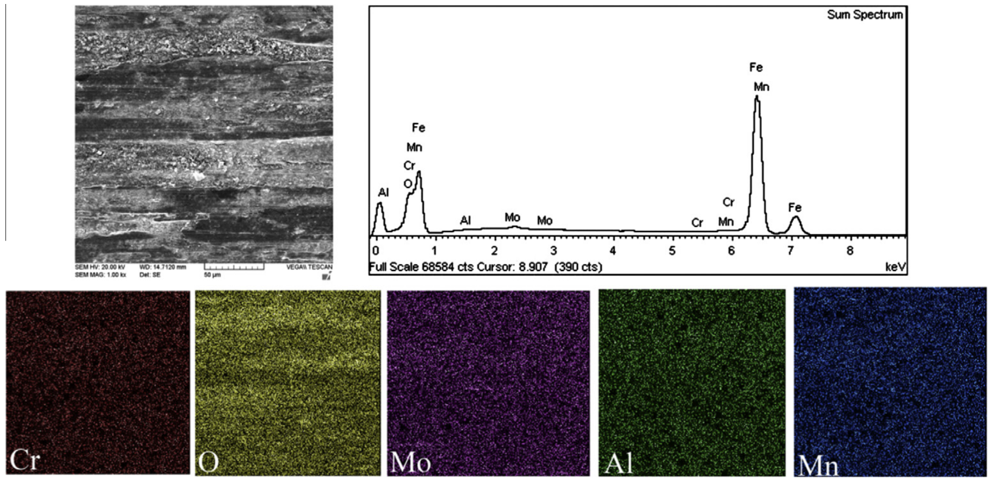

Fig. 9. Worn surface, EDS analyze of the region and elemental distribution of B1 at 25 N: versus Cr, O, Mo, Al and Mn elemental distribution.

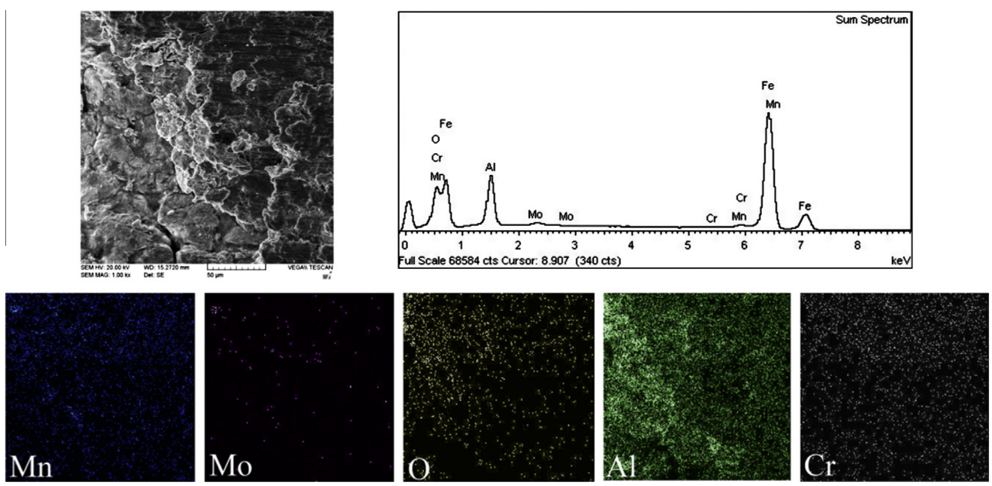

Fig. 10. Worn surface, EDS analyze of the region and elemental distribution of B1Al5 at 45 N: versus Mn, Mo, O, Al, and Cr elemental distribution.

load at any sliding velocity, wear rate will be increased [24]. Applied load is important parameters of wear process possessing crucial effect on tribological behavior. Changing in applied load can alter wear mechanism and lead to change wear resistance. Consequently, increasing in applied load often cause severe volume loss [5,19].

Furthermore, presence of alumina nanoparticles significantly ameliorated wear resistance by decreasing volume loss. Although wear resistance of specimens increased with adding alumina nanoparticles, but hardness values decreased gradually due to role of surface porosity as mentioned earlier. Surface porosity plays the role of sink in order to gather oxide and metallic particles from surface, processing hard behavior and could cause abrasion. Improved wear resistance in samples with increasing reinforcing agent is due to overlapping two features, increasing porosity level and hard alumina nanoparticles. Comparison among samples clearly states that hardness value decreased 19% with adding alumina nanoparticle up to 5 weight percentage. However, volume loss decreases 67% in 25 N, 65% in 35 N and 73% in 45 N. In another words, amount of improving wear resistance was really substantial. It meaningfully shows the positive effect of two features activated together. The oxidation wear mechanism is the most frequent ones in dry sliding wear of ferrous materials [10]. Extraordinary local temperatures released during dry sliding permit oxide film development. It can be seen in different MAP analyzes and EDS results in Figs. 9 and 10. Dark regions show oxidation layers forming at high local temperatures leading to delamination by cracks formation. After reaching a critical size, it breaks up and flake-like debris is generated. It seems that abrasive, adhesive and oxidation wear were the dominant mechanisms in worn samples. Although the B1Al5 specimen has the best wear rate and minimum amount of volume loss in the load of 45 N, but amount of Cr and Mn, as the elemental component in the pin, on its surface showed that predominant wear mechanism was adhesive (Fig. 10). Low volume loss should cause of adhesion of the pin elements on the surface of the B1Al5. High porosity level (almost 18%) and intense plastic deformation (Fig. 8) are evidences for this justification. Considering this point, the B1Al3 showed the best wear resistance among all samples. Outcomes from the EDS analysis on the worn surface describe abrasive, adhesive and oxidation wear mechanisms which one or two of them are interfered in wear morphology.

4. Conclusions

In this research, tribological behavior (at dry condition) of the pre-alloyed powder steel (Astaloy 85Mo) reinforced with alumina nanoparticles was investigated at conventional sintering process and below conclusions could be drawn:

(1) A major complexity in the PM–MMC samples was that pores could make a drop in mechanical behavior but optimum amount of them could play as a positive factor in tribological properties. Pores performance as a sink gathering wear debris from surface and improved wear resistance by reduction in abrasive wear.
(2) Increasing alumina nanoparticles, hardness values gradually decreased due to dwindling in density level.
(3) Increasing applied load could change wear mechanism and definitely the wear rate. Oxidation, adhesive and abrasive as dominant mechanisms are changing with increasing applied load. In order to avoid high volume loss, applied load should be lower down.
(4) Adding alumina nanoparticles as reinforcing agent has significantly increased wear resistance with lowering volume loss and wear rate. Specimens with 3 wt.% alumina nanoparticle (B1Al3) with porosity level of 15.38% owning hardness of 105.4 HV5, assigned as the best wear resistant material.

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