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Sintered Steel Composites Reinforced with Ceramic Nanoparticles: Fabrication, Characteristics and Wear Behavior

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ABSTRACT

Steel composites reinforced with alumina nanoparticles were prepared by powder metallurgy process. Samples containing two different amounts of carbon (0.35 and 0.55 wt.%) and 0-5 wt.% Al_2O_3 nanoparticles were fabricated by mixing, compacting and sintering of diffusion bonded Distaloy AE powder. The density, hardness and wear tests were performed on the prepared samples. Furthermore, the microstructures and worn surfaces were analyzed by optical and scanning electron microscope (SEM), respectively. The wear tests were carried out in forces of 30, 40 and 50 kN and 1000 m distance in dry condition and ambient temperature. The results showed that increasing the alumina nanoparticles would reduce the density (~ 10.8 and 9.6 % for 0.35 and 0.55 wt.% C, respectively) and would increase the hardness (~ 14.1 and 7.2 % for 0.35 and 0.55 wt.% C, respectively) of sintered samples. With increasing carbon content, the amount of lost material and the rate of wear decrease. The lost volume increases with increasing wear distance and applied force. Addition of reinforcement to the steel matrix, improves the wear resistance up to 3 wt.% alumina nanoparticles. This increase in the samples varies between 30% to 73%. More increasing of alumina (5 wt.%) decreases the wear resistant of samples. The wear mechanisms including oxidation in low forces that convert to adhesive and abrasive with increasing applied force. The wear curves indicate that as the wear distance increases, the lost volume increases, while the wear rate decreases.

1. Introduction

The economic factors, the production value, proper quality and gaining of market share are important issues that need to be addressed in order to part manufacturing in today's developing industry. Among the various methods, powder metallurgy (PM) is an efficient process for manufacturing the parts, given the aforementioned aspects [1]. Powder Metallurgy is a precision method for producing the metal products by compacting powders to a desirable shape and then sintering at an adequate temperature in controlled atmosphere. Furthermore, in contrast to the conventionally produced components, production costs were lowered through the elimination of finishing process, reduced material scrap and often environmental cleaner production process by powder metallurgy [2].

PM parts are often used in wear conditions, which is expanding in the automotive industry, the office machines components and other fields. In recent years, demand for the production of steel parts through the powder metallurgy process has been considered due to its economic aspects [3-5]. Parts manufactured by this method are often exposed to high mechanical loads and complex forces, the most important of which are motor components, power transfer gears, plastic injection extruder and some parts that are used in the aerospace industry [6, 7].

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Mechanical behavior of sintered steels is determined principally by tensile, hardness and fatigue tests [2]. However, the demand for the production of parts that are in contact with each other during the service is increasing. This issue stimulates researchers to improve the operation of powder metallurgy components under dynamic conditions. The durability and life time of many PM components are limited by the wear process. The wear phenomenon is one of the problems that the industry has been encountered for a long time and it has been a major section of the destruction in the industry [1]. For this reason, the study of wear and tribological behavior along with other mechanical properties such as yield strength and fracture toughness are important. Powder metallurgy steels are widely used to operate under sliding, rolling, abrasive and contact wear conditions and a deep understanding of their tribological behavior is very important [6]. The chemical composition, additives, porosity, heat treatment, surface treatment, lubrication and wear loading variables are among the most important factors influencing the wear of sintered steels [8, 9].

The wear behavior of full density steels has been thoroughly studied and relatively large amounts of data for various types of steels have been reported under different conditions and in various wear environments. However, less attention has been paid to the wear behavior of powder metallurgy materials. The presence of pores and special microstructure that are specific to porous sintered materials led to a more complex wear behavior of these components compared to full dens materials [4].

One of the ways for improving the wear behavior of powder metallurgy parts is the addition of reinforcements to metal matrixes and fabrication of composites. In fact, the important method in the manufacture of wear resistant parts is the addition of hard particles to the tough matrix in order to produce metal matrix composites [10]. Hard materials dispersed in the matrix have been shown to increase the strength, stiffness, wear resistance and mainly would reduce the density [11]. Due et al. [12, 13], showed that the wear resistance and hardness of low-carbon steel is improved by the formation of TiC-VC particles in the iron matrix. Steel-based composites containing a minor fraction of carbide, nitride, boride and oxide particles are common composites for wear applications [7]. Powder metallurgy provides a suitable solution for adding second-phase particles to the matrix and improving wear resistance. According to Torralba and Zapata [14, 15], the addition of alumina and carbides to powder metallurgy high speed steels would result in a significant improvement in the sinterability and wear behavior. Reinforcing particles have a significant effect on the improvement of wear resistance, which can limit the growth of cracks and increase the load bearing. One of the advantages of adding a reinforcing particle to a metal matrix is the creation of a fine microstructure in PM process which can improve the mechanical properties and would enhance the wear resistance. Garcia et al. [16], examined the wear behavior of a AISI M2 PM steel with various amounts of vanadium carbide (3, 6 and 10 wt.%) after sintering. The microstructure refinement and the formation of carbonitride compounds could be regarded as reasons of improving the wear resistance of such steels. Due to the possibility of carrying out heat treatment and various surface treatments on the steels, increasing the wear resistance often is done through these operations. However, powder metallurgy provides the opportunity to add hard ceramic particulate reinforcement like alumina to the steel matrix for enhancing the wear property. Alumina particles have excellent oxidation, corrosion and wear resistance. The utilization of alumina nanoparticles as reinforcement in metal matrix composites leads to a proper combination of reinforcement and matrix properties [17].

In the present study, steel-based composites with different amounts of second phase were produced via powder metallurgy process and the effect of nanoalumina particles on the density, hardness and wear behavior of produced composites were investigated.

2. Experimental Procedure

For the production of specimens, a low-alloy diffusion bonded steel powder named as Distaloy AE (Hoganas Co.) was used. The chemical composition is presented in Table 1. The production process consists of the co-annealing of a mixture of iron powder and Ni, Cu and Mo powders in a medium temperature and reducing atmosphere. During heating, the alloying elements partially diffuse into the iron-base powders and a metallurgical bonding between the iron powders and the alloying element particles would be formed.

Table 1. The chemical composition of Distaloy AE steel powder.

Alloying Element	Wt.%
Carbon	0.01<
Nickel	4.00
Copper	1.5
Molybdenum	0.5

0.8% zinc stearate was added to the powder as a lubricant. Also, in order to study the effect of carbon content on the microstructure and wear behavior, two groups of samples were produced with addition of 0.35 and 0.55 wt.% carbon as natural graphite (UF4). In order to investigate the effect of second phase on the wear behavior, 0, 1.5, 3 and 5% nano-alumina were added to each group. The homogenizing was performed by a 1 kg laboratory mixer for 25 minutes at a speed of 40 to 50 rpm. The lubricant was added at the final stage of mixing in order to eliminate the powder agglomeration. The average size of the alumina powder is 80 nm and its purity is more than 99% (US research nanomaterials Inc.). Fig. 1 shows the transmission electron microscope (TEM) image of nanoalumina powder.

Powders (Distaloy AE, carbon, nanoalumina and lubricant) after mixing were compacted in a die and finally flat test bars were fabricated according to ISO 3928 standard [18]. The samples were compacted with 600 MPa pressure by a hydraulic machine (60 tons capacity). The drawing of these standard samples is shown in Fig. 2.

The sintering of green samples was carried out in a mesh belt continuous furnace at 1120 ºC for 30 min in 90%N_2/10%H_2 atmosphere. The produced samples are shown in Fig. 3.

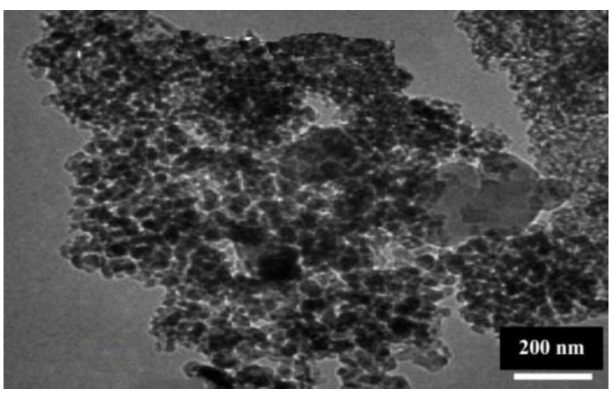
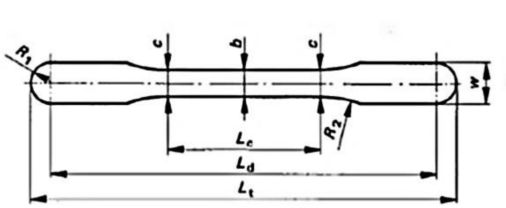


Fig. 1. TEM image of nanoalumina powder.



b	c	L_c	L_d	L_t	W	R_1	R_2
5.70 ± 0.02	b + 0.26	32	81.0 ± 0.5	89.7 ± 0.5	8.7 ± 0.2	4.35	25

Fig. 2. The drawing of flat samples according to ISO-3928 standard (All dimensions in millimeters) [18].

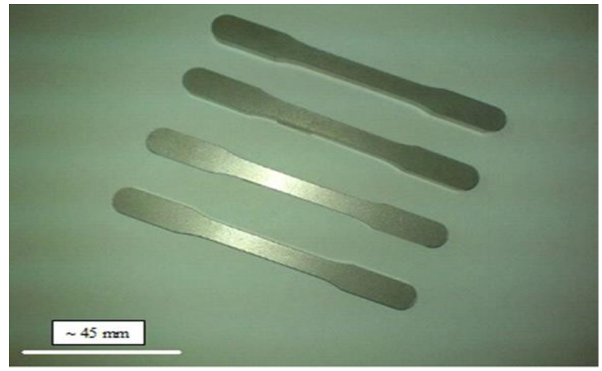


Fig. 3. Sintered flat standard samples.

Density measurement was performed according to ASTM-B328 [19] and Archimedes immersion technique. The apparent hardness was measured by the Vickers method and under 5 kg force. For this purpose, two parts were selected from each group and the hardness test was repeated five times. Then, the mean value was reported as hardness. These tests were performed according to the MPIF-43 standard [20]. Also, for determination of metallurgical phases, microhardness Vickers test was done according to ASTM-E384 standard [21] and under 0.5 Kg force.

Dry wear test was done at ambient temperature in humidity of 30–35% by linearly reciprocating ball-on-Flat method. The pin is made of hardened steel. The vertical load, speed and maximum distance were selected as 30 to 50 KN, 0.15 m/s and 1000m, respectively.

The weighing process of the samples was repeated to calculate the lost weight after every 250 m of distance. It is worth noting that the first 250 m of wear distance was considered as sealing region. Wear test data are generally presented as curves known as wear characteristics (lost volume vs. wear distance). Two different regions could be detected in these curves. The first area is named sealing region in which the wear is unstable. The second area is named as uniform and stable wear zone. In fact, in the unstable wear zone (sealing region), two contacting surfaces are starting to be compatible. The extent of the unstable wear region depends on the applied force. When applied force reduces, this area becomes more restricted. In the sealing region, work hardening and phase transformation can be occurred and the wear rate varies. After the sealing zone, the wear rate does not change over time, and therefore, the area is said stable wearing area. In order to determine the wear rate, the following equation was used [22].

$$K = \frac{\Delta V}{F_N . S} = \frac{\Delta m}{\rho . F_N . S} \tag{1}$$

where K, ΔV, F_N and S are wear rate, worn volume, applied force and sliding distance, respectively. ΔV is calculated through the ratio of weight loss (Δm) to density (ρ).

Metallographic observations were performed on the prepared samples after polishing and etching using a light optical microscope. Because of the possibility of pores deformation during sample preparation, these operations were performed at the minimum possible speed. In order to investigate the wear mechanisms, the worn surfaces were evaluated by scanning electron microscope (SEM).

3. Results and Discussion

The microstructure of powder metallurgy sintered steels generally consists of pores and other metallurgical phases, such as ferrite, perlite, austenite, bainite and martensitic, depending on the chemical composition and cooling rate. The general microstructure of fabricated PM steel is shown in Fig. 4.

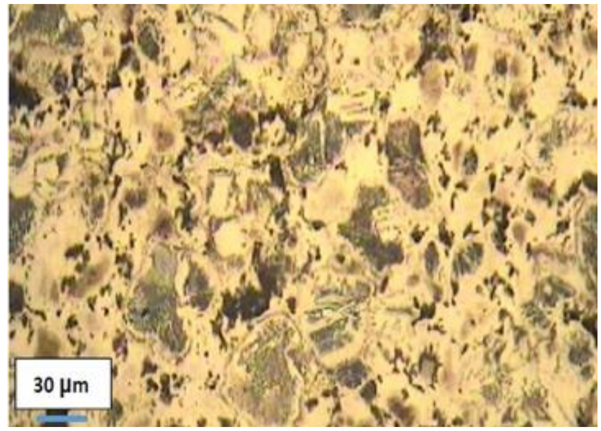


Fig. 4. The microstructure of Distaloy AE PM steel.

The results of the phase analysis and the microhardness tests indicate that the microstructure includes porosity, base powder particles consisting of perlite and ferrite phases, martensite network around the base powder particles, nickel rich regions and points of bainite.

Carbon is one of the most important alloying elements in all types of steels as well as powder metallurgy sintered steels. The change in the microstructure by the presence of a higher carbon content, and consequently the change in mechanical properties, is a major result of the presence of carbon in these steels. In powder metallurgy samples, increasing carbon content can create more strength microstructure. The results of microstructural and microhardness investigations in this study indicate that with increasing carbon percentage from 0.35% to 0.55%, the amount of ferrite in base powder particles decreases and the perlite increases. This issue has been explored in details in another article [23]. Also, by increasing the carbon content, the martensitic network hardness was also enhanced. Comparison of the microhardness of base powder and martensite networks in these two groups of steels shows that with increasing the carbon content, the hardness of base powder particles increases about 30% (from 195 to 251 Vickers) and the hardness of the martensite network enhances 18% (from 458 to 540 Vickers). Therefore, it is concluded that due to the role of carbon on the microstructure, the macrohardness and mechanical properties, including wear behavior could be changed.

During compaction of samples containing nanoparticles as reinforcement, it was determined that by increasing the amount of nanoparticles, the density decreases. This decrease is due to the small size (high surface), as well as the high hardness of the alumina nanoparticles, which prevents more compressibility of the base powder. By increasing the amount of nanoparticles, the steps of changing the powder arrangement and particle local deformation, during the pressing, will be more limited. In other words, the presence of alumina particles at different stages of powder consolidation limited the powders rearrangement and localized deformation. The placement of alumina nanoparticles in the empty spaces of the powders changes the sample compaction and, by reducing the initial stages of powder particle deformation, reduces the density in the parts.

Furthermore, the general feature of the porosity in the microstructure was altered by increasing the alumina nanoparticles. In general, increasing the porosity causes the state of the porosity changes from a single, regular state to inter-connected and irregular. This leads to an increased local strain, a reduction in the strength and ductility of powder metallurgy components [24]. In the samples with low porosity and small pores, trapping of debris is not easy. This phenomenon is one of the main reasons for improving the wear properties of porous samples (samples made by powder metallurgy) [9, 19, 22, 25].

Density is the main characteristic of the powder metallurgy materials and porosity is considered as the most important characteristic of sintered samples microstructure, which has a significant effect on all of properties, and in particular on mechanical behavior. The presence of pores as a discontinuity in the parts, could cause stress concentration and affect the behavior of the material against stress. As a general issue, the mechanical properties of the sintered steels are reduced by pores [26]. Since in the present research, the conditions such as press pressure, chemical composition, temperature and time of sintering and the particle size of the powder particles are the same for all specimens, the difference in the density and morphology of the pores can be caused by the presence of secondary nanoscale phase. The density, percent of porosity and hardness are shown in Table 1.

The changes of density show that the density decreases with the increase of secondary phase particles. The reason can be related to two issues. First, alumina particles have lower density (3.95 g/cm^3) compared to steel (7.8 g/cm^3) and according to the mixing rule, the composite density decreases with increasing mass fraction of a lower density component (alumina). Second, the presence of hard particles (nano-alumina) in the powder mixture reduces compressibility, and therefore, with increasing the fraction of nanoparticles, samples with lower density are produced [27]. As shown in the previous section, also the shape of pores changes from approximately single and closed to inter-connected state with the increase of second particles.

As shown in Table 1, the hardness value in constant carbon content increases when nano-alumina particles increase up to 3%. It is expected that the addition of alumina due to the high hardness of these particles, in accordance with the mixing rule in the composites, and the role of secondary phase as grain growth inhibitor can increase the hardness of samples [17].

Although, the role of these hard particles is important in changing the compressibility of the powder mixture, the results indicate a reduction in the powder condensation due to the presence of nano-aluminum. By increasing the weight fraction of the second phase particles, the density decreases. Despite the density reduction, hardness increases with increasing of nanoparticles up to 3%. This has already been observed for alumina particles reinforced aluminum powder [27]. The composite hardness is achievable from the following equation [28]:

$$\mathrm{H_c} = \rho_{fc}\left(H_m + \frac{\emptyset_p^{0.5}(H_p - H_m)}{d^{0.25}}\right) \quad (2)$$

where ρ_{fC}, H_m, Φ_p, H_P and d are respectively the relative density of the composite, the matrix hardness, the volume fraction of the reinforcement, the hardness of reinforcement and the particle diameter. According to the Eq. 2, adding nano-particles with high hardness (higher values of H_P), will increase the composite hardness.

In other words, it can be noted that according to the conditions of the production, especially the conditions of compression, the hardness of the manufactured parts is more influenced by the presence of secondary phase and less affected by the density.

There is also no increase in hardness of samples containing 5% wt. Al_2O_3. In these samples, the hardness decreases. The reason could be attributed to the non-uniform distribution and agglomeration of nanoparticles with increasing of nanoparticles weight fraction. Reinforcement agglomeration has been observed and reported as a result of an increase in the amount of the second phase from a certain value [17]. In this case, the reinforcing particles are not distributed uniformly and homogeneously, and direct contact between them without creating a strong bond leads to the loss of mechanical properties, such as hardness [17, 29].

The lost volume curves against wear distance for tested parts contain various amounts of nanoparticles and 0.35 and 0.55 wt.% carbon in different normal loads are shown in Figs. 5 and 6. As it can be seen, the increase in wear distance is associated with an increase in lost volume for all parts. In addition, as the amount of normal force increases, the lost volume increases. Increasing the amount of applied force leads to a change in the wearing mechanisms and, as a result, increases the lost volume of the wear [6, 25, 30].

The SEM images of worn surfaces are shown in Figs. 7 to 9. In low normal forces, the mechanism governing the wear process involves the oxidation wear (Fig. 7). The formation of oxide on contact surfaces will protect surfaces and avoid direct contact between the two surfaces. The oxide layer acts as a cover and reduces wear.

Table 1- Density, porosity and hardness of sintered samples contain various amounts of reinforcement.

Sample	Density (g/cm^3) ± 0.05	Porosity (%) ± 0.05	Hardness (HV5)
0.35C-0Al_2O_3	7.4	5.12	184
0.35C-1.5 Al_2O_3	7.1	8.97	195
0.35C-3Al_2O_3	6.8	12.82	210
0.35C-5Al_2O_3	6.6	15.38	190
0.55C-0Al_2O_3	7.3	6.41	208
0.55C-1.5Al_2O_3	7.1	8.97	216
0.55C-3Al_2O_3	6.9	11.54	223
0.55C-5Al_2O_3	6.6	15.38	197

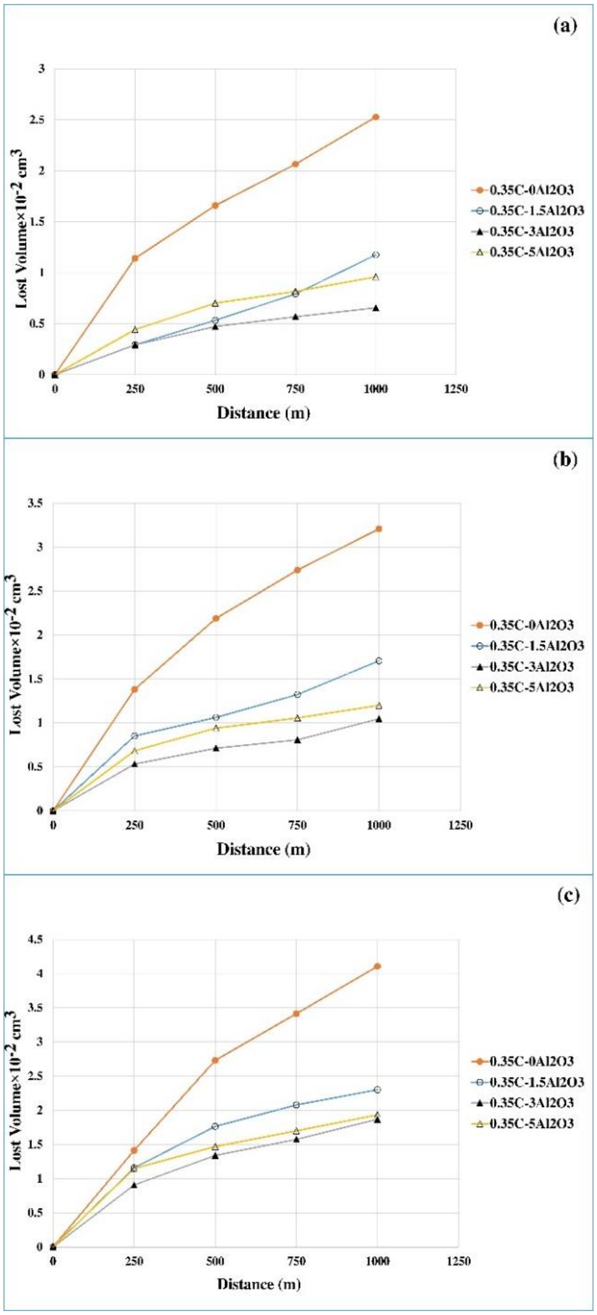


Fig. 5. Volume lost against wear distance for samples contain 0.35 wt.%C and various nanoparticles for a) 30 KN, b) 40 KN and c) 50KN.

By increasing the amount of force to the 40 kN, adhesive and abrasive wear (Fig. 8) are the dominant wear mechanisms. In the 50 kN force, the dominant mechanism is abrasive (Fig. 9). In fact, the increase in force leads to a breakdown of the oxide layer and severe plastic deformation of pores also occurs during wear. It should be noted that the depth of the grooves also increases with the increase in force.

In a constant force, with increasing the amount of carbon, the lost volume decreases. In steels, hardness increases while carbon increases. Increasing carbon can improve the wear behavior and reduce the material mass lost, under the wear process. The reason for this is the increase in the amount of perlite in the base powder particles relative to the ferrite due to the increase in carbon content. Since the perlite phase has a better wear resistance than ferrite [4], the improvement of wear behavior in samples with more carbon content is observed in this study.

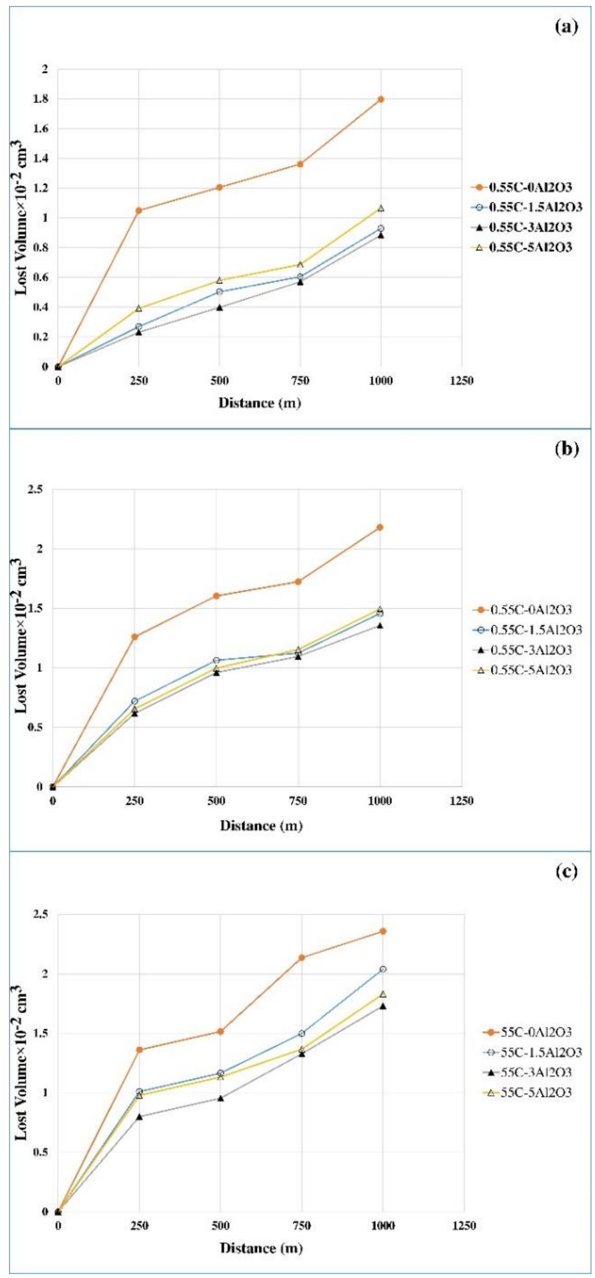


Fig. 6. Volume lost against wear distance for samples contain 0.55 wt.%C and various nanoparticles for a)30 KN, b)40 KN and c)50KN

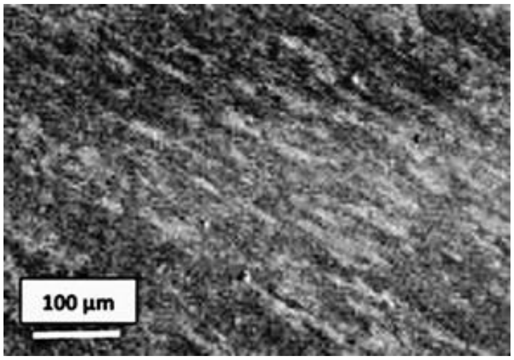


Fig. 7. Worn surface of sample contain 0.35 wt under 30 KN force and after 750 m.

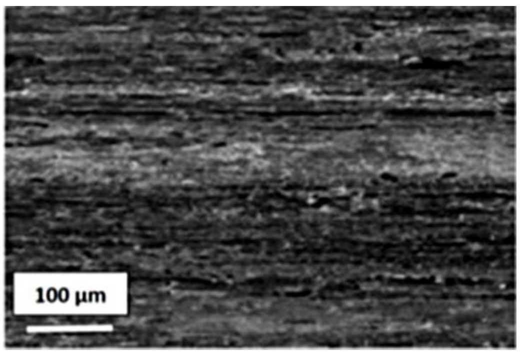


Fig. 8. Worn surface of sample contain 0.35 wt.% C under 40 KN force and after 750 m.

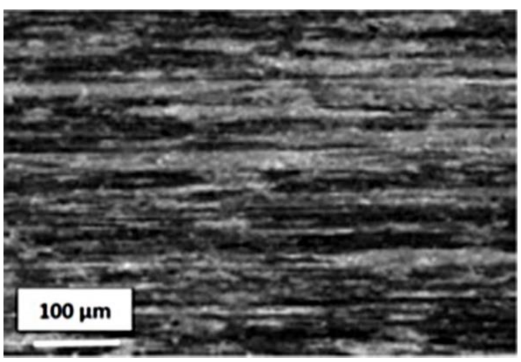


Fig. 9. Worn surface of sample contain 0.35 wt.% C under 50 KN force and after 750 m.

The most important reason for the improvement of wear resistance in steels containing higher carbon is related to the increase of perlite in the microstructure. However, this change depends on the amount of carbon (hyper-eutectoid or hypo-eutectoid). Wear resistance for hyper-eutectoid steels (carbon more than about 0.7%) decreases with increasing the carbon. Probably, because the increase in the carbon in these steels leads to the formation of continuous cementite networks. Conversely, it has been shown that perlite microstructure in a hypo-eutectoid steel with carbon content similar to the present study, can improve the wear resistance [31]. The perlite phase can greatly prevent nucleation and growth of micro-cracks. In hyper-eutectoid steels, higher carbon causes the thickening of the boundary of cementite in perlite. The cementite phase does not tolerate plastic deformation during wear, and further cracking of this region reduces wear resistance. As a result, increasing the wear resistance with a higher carbon content in the hypo-eutectoid steels is more significant than that of the hyper-eutectoid [31]. It should be noted that increasing of hardness in powder metallurgy steels always does not increase the wear resistance, for example in heat treated PM steels. The reason is to create harder phases, such as martensite, which have less resistance to wear and, under certain conditions, increase the cracking and destruction of wearing surfaces [32, 33].

It is also clear from the plotted diagrams (lost volume vs. distance) that the wear resistance improves with increasing the alumina nanoparticles, but this increase continues to 3 wt.% of nanoparticles and then, by adding a greater amount of nanoparticles (5 wt.%), degradation of wear resistance could be seen. This improvement is associated with increasing the hardness of the samples due to the addition of nanoparticles. According to the results, it is determined that the optimum amount of alumina nanoparticles in the present study is 3 wt.% and the more nanoparticles create agglomerates, decrease hardness and wear resistance. Increasing the probability of agglomeration with the increase of the secondary phase amount has also been reported [17, 27]. With the increase in the amount of alumina nanoparticles before reaching the amount that leads to agglomeration, the amount of porosity increases, that these pores are suitable places for the trapping of wear debris that can improve the wear resistance [25, 32]. In samples with higher alumina nanoparticles (5 wt.%), the presence of the second phase reduced the compressibility of the powder. In these conditions, it has been shown that the pores are not suitable for trapping of the wearing debris, and the presence of these pores can accelerate the cracking and loss of the material [25].

The variation of wear rate against force are shown in Figs. 10 and 11 for samples containing 0.35 and 0.55 wt.% carbon and various amounts of nanoparticles. Investigating the wear rate for various samples in different forces and distances shows that with increasing distance and so increasing the amounts of material lost, the wear rate decreases. The reason is the better alignment and compatibility of the wear surfaces at the contact points over longer distances. Therefore, the initial higher wear rate decreases in long distances.

During increasing the applied load, the role of pores in creating larger deformation, cracking and thus increasing the wear rate is greater than their role in trapping of wear debris. Therefore, as it can be seen, the lost volume and the wear rate would increase with applying higher forces in a constant distance.

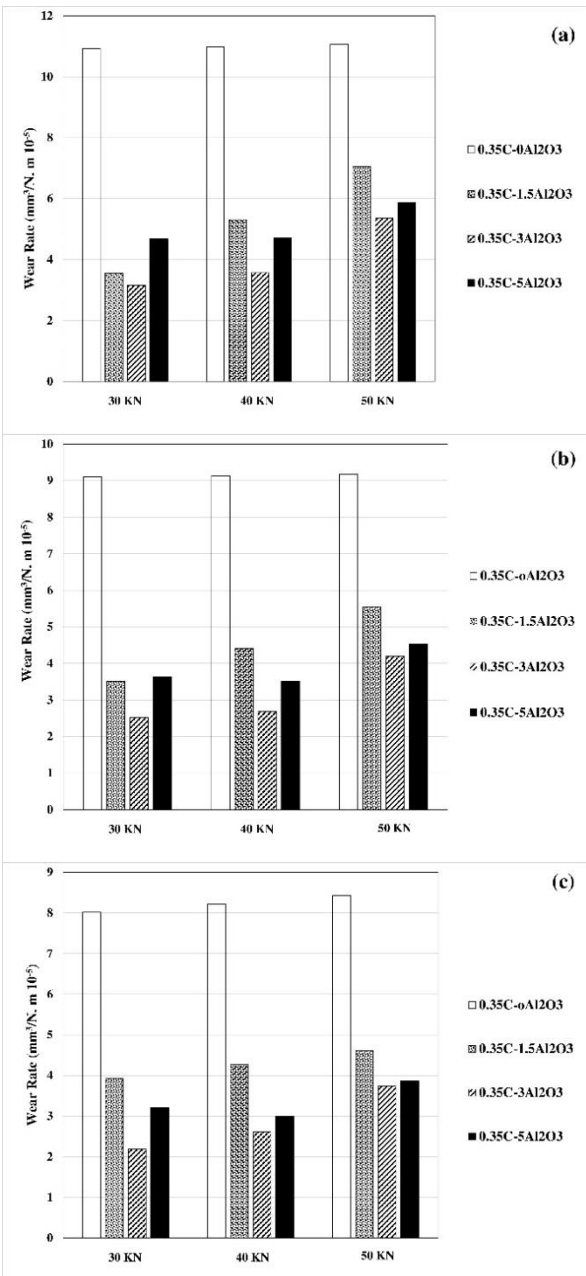


Fig. 10. Wear rate variations for Distaloy AE PM steels contain 0.35 wt.% carbon and 0, 1.5, 3 and 5 wt.% nanoparticles in a) 500 m, b) 750 m and c) 1000 m wear distance.

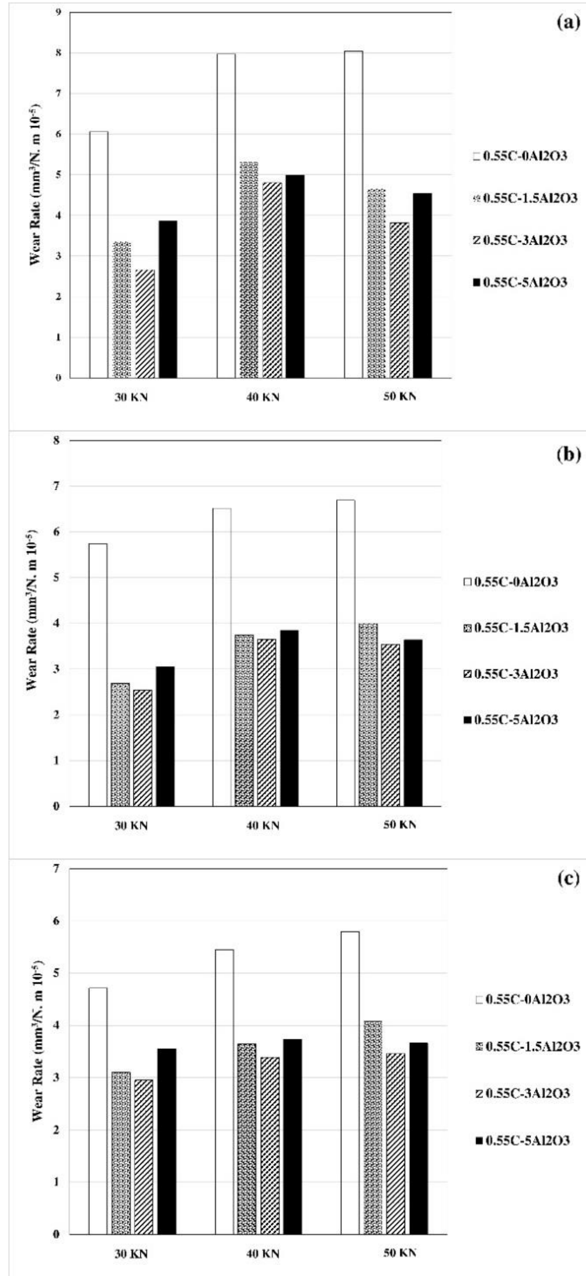


Fig. 11. Wear rate variations for Distaloy AE PM steels contain 0.55 wt.% carbon and 0, 1.5, 3 and 5 wt.% nanoparticles in a) 500 m, b) 750 m and c) 1000 m wear distance.

In general, wear rate of samples with 0.55 wt.% carbon due to the higher hardness is lower than those with a 0.35 wt.% carbon, which shows a positive role of carbon in improving the wear resistance of samples which was mentioned before. According to the results, the wear rate is reduced by adding up to 3% wt.% of the nanoparticles; however, by increasing the nanoparticle up to 5%, an increase in the wear rate was observed. This issue, as stated before, indicates the positive role of nanoparticles to an optimal amount before agglomeration to improve the wear resistance. Therefore, for samples without reinforcement, the wear rate is high, then, with increasing the nanoparticles, the wear rate decreases and then increases again.

4. Conclusions

In the present study, fabrication, characterization and wear resistance of a powder metallurgy steel nanocomposite containing copper, nickel and molybdenum alloy elements with 0.35 and 0.55 wt.% carbon and in the presence of 0-5 wt.% alumina nanoparticles as a reinforcement phase were studied and evaluated. The most important results are as follow:

- The results of the density measuring for samples containing nanoparticles indicate that the density decreases with increasing the amount of second phase.

- By increasing the wear distance and the applied force, the wear lost volume increases.

- SEM investigations indicate that the various mechanisms occur during the wear process. In low forces (30 kN), the mechanism is the oxidation wear. As the force increases, gradually the adhesive wear together with the abrasive wear forms the dominant mechanisms, which is associated with significant plastic deformation.

- When carbon content increases, the amount of lost volume decreases. Increasing the carbon leads to the formation of more perlite phase in the microstructure and thus improves the wear resistance.

- The results of the wear and hardness tests indicate that these properties are improved by increasing the nanoparticles, but this trend continues to 3 wt.%, and then, by adding a greater amount of nanoparticles (5 wt.%), degradation of hardness value and wear resistance is observed due to agglomeration.

- It is shown that with increase of distance, although lost volume increases, the wear rate decreases.

- Due to the positive role of carbon in improving the wear resistance, the rate of wear in samples with 0.55 wt.% carbon is lower than those with a 0.35 wt.%.

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