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Mechanical properties and microstructural characteristic of sinter-hardened steels

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ABSTRACT

Purpose: The main goal of this paper was the examination of the role of W addition on properties of two sinter-hardened alloys with two different carbon levels. Additionally the influence of sinter-hardening process has been evaluated. Microstructural characteristic and mechanical properties of Ni-Mo steels with increasing amount of tungsten (from 0 to 0.3% wt.) were taken under consideration.

Design/methodology/approach: Powder mixes have been compacted at 700MPa and sintered in a vacuum furnace with argon backfilling at 1120°C for 60 minutes. Rapid cooling has been applied with an average cooling rate of 2.5°C/s. Obtained steels were analyzed by scanning microscopy with energy dispersive spectroscopy (EDS) for phase distribution and mapping and light optical microscopy for observations of the microstructure. Charpy impact test, three-point bending, microhardness, pin-on-disk and disk-on-disk tests were used.

Findings: The outcome of implemented vacuum sintering with rapid cooling as well as chemical composition were studied in terms of mechanical properties, focusing in particular on impact energy, hardness and wear resistance. The results achieved after the investigation of Ni-Mo and Ni-Mo-W sinter-hardened steels with low and high carbon content proved that applied process of sintering under vacuum and rapid cooling brought expected outcome.

Research limitations/implications: The characteristics of powders and the applied cooling rate were found be a good compromise for mechanical properties and microstructures, though further researches should be carried out in order to examine different cooling rates and parameters of tempering process.

Originality/value: The effect of W and/or WC additions to highly alloyed steels is well known. In the work the effect of small additions of W and WC to low alloyed steels, especially in terms of hardenability and wear resistance, was investigated.

Keywords: Powder metallurgy; Sintering; Low alloyed steels; Sinter-hardening

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MATERIALS MANUFACTURING AND PROCESSING

1. Introduction

Powder metallurgy development has rapidly increased in past years as an alternative technology profitable because of low cost and no need of metal working processes such as machining, stamping and forging. It has continued to displace competing cast or wrought technologies in automotive applications. One of the main advantages of the technology is the easiness of mixing different metal powders and composing new materials with unique physical and mechanical properties that cannot be obtained by standard melting-casting processes. Growth of powder metallurgy processes using sintering is followed by all technological advancements throughout development of material systems with higher and more reliable performance than previously available, new processes of obtaining powders, lubricants and near net-shaped products that are widely used in the production of automobile parts [8,11,14,17].

Introduction of PM steels with a success into new applications requires meeting the needs and expectations of end users for improved performance and superior efficiency than current systems. Even though every application has individual necessities, several common features can be listed and are shown in Figure 1 [3].

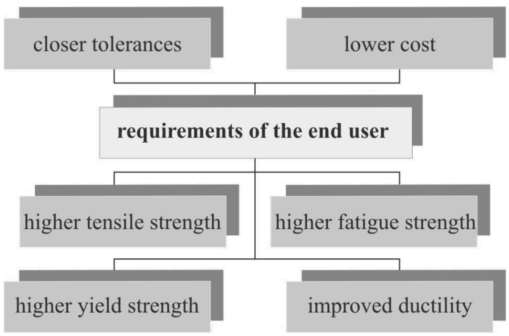

Fig. 1. Market requirements for PM steels (Causton, 1996)

Sinter-hardening is an increasingly popular technique, which requires to cool parts from the sintering temperature at a rate sufficient to transform a significant portion of the material matrix into martensite. Application of that process allows avoiding secondary heat treatment, contamination of pores in the sintered steels with quench oil and helps in subsequent surface treatment improving the environment of working place. Ultimate tensile strength and hardness of sinter-hardened parts are equivalent and even superior to those conventionally heat treated PM steels obtained by double pressing and sintering. This fact was confirmed in number of publications. Cost reducing, technical and manufacturing economy, improved efficiency of process, densities and mechanical properties, make PM products more appealing, especially in case of applications where high wear resistance is required. Sinter-hardening permits the production of powder metallurgy components having high apparent hardness and high strength and is applied for components difficult to be quenched because of their shape or dimensions [1,2,4,5,7,13,15].

Fe-Mo pre-alloyed powders, in part with addition of Ni through admixing or diffusion bonding, can be successfully used at the conventional sintering temperatures and in sinter-hardening process. They are characterized by high density, homogenous or heterogeneous microstructures (the degree of heterogeneity varies with the carbon concentration), which promote tensile and fatigue strength. High compressibility of Ni-Mo powders allows producing powder metallurgy steel compacts with green density reaching 7.3Mg/m^3, promotes sintering density and improves pore morphology without the need of applying double pressing and double sintering processes. In addition, Ni-Mo sintered alloys are characterized by relatively low value of dimensional change [6,13].

In pre-alloyed powders the selection of type and amount of alloying elements additions has to be considered and focused, in particular, on compressibility, sinterability of green compacts and on the transformation of austenite in order to achieve the desired microstructure. This concept shows that PM steels can be obtained from premixes including high compressibility matrix admixed with alloying elements characterized by ability of fast diffusion and promoting sinterability. Pre-alloys shouldn't reduce compressibility, but ought to contribute to hardenability in the presence of carbon [6,9,10,16].

In addition, the way chosen for alloying, distribution of powders and green density clearly affect porosity of sintered parts. All processing variables, sintering temperature, time of the process and post sintering cooling rate have to be well defined with the aim of modeling final properties required in specific field application [6,9,10].

Molybdenum, due to its relative small impact on compressibility if compared to other alloying elements, good response in hardenability, is one of the main pre-alloyed elements used in powder metallurgy industry. It has been proved that the addition of nickel to iron-molybdenum powders increases sintered density, hardenability and reduces sintering activation energy. Ni-rich areas provide local ductility and influence positively hardness and strength of sintered materials. Both elements, Ni and Mo form highly stable oxides determining the sintering atmosphere to be utilized and allowing for development of bainitic-martensitic microstructure when standard conditions of cooling are used. Products prepared from Fe-Mo pre-alloyed powders with presence of heterogeneous microstructure, high densification and better fatigue properties show many advantages in number of applications [6,9,10,16].

The effect of W and/or WC additions to highly alloyed steels is well known, especially in the case of hardenability and wear resistance [12,18], but there is no recollection of literature of W additions in small amounts into low alloyed products. The goal of this work was also to investigate the role of tungsten on the properties of two different carbon levels pre-alloyed steel powders.

2. Experimental procedure

Different compositions have been tested in order to investigate the influence of various tungsten additions into low (0.4%) and high (0.6%) carbon content of pre-alloyed steel powders. The chemical composition of powders used in this study and their designation are listed in Table 1.

Pre-alloyed steel powders, with addition of 0.7% lubricant, were pressed in a 2000kN hydraulic press applying pressure of 700MPa, using rectangular (10x55) and a disk shaped mould (40mm diameter) in order to prepare samples for Charpy, three-point bending and both pin-on-disk and disk-on-disk tests. De-waxing process at 550ºC for 60 minutes in a fully nitrogen atmosphere was performed before the sintering. Sintering was carried out in vacuum furnace with argon backfilling. The furnace was equipped with a cooling zone to provide accelerated cooling from the sintering temperature. Green compacts were sintered at the temperature 1120ºC for 1 hour and rapidly cooled with a rate 2.5ºC/s. The performance of the temperature during sintering and rapid cooling is shown in Figure 2.

Table 1.
Chemical composition of studied powder mixes

Grade powders	Composition designation	Elements concentration (wt.%)				
		Ni	Mo	W	C	Fe
W0-AC	0A	2.00	1.50	-	0.60	95.9
W0-BC	0B	2.00	1.50	-	0.40	96.1
W1-AC	1A	2.00	1.50	0.10	0.60	95.8
W1-BC	1B	2.00	1.50	0.10	0.40	96.0
W2-AC	2A	2.00	1.50	0.20	0.60	95.7
W2-BC	2B	2.00	1.50	0.20	0.40	95.9
W3-AC	3A	2.00	1.50	0.30	0.60	95.6
W3-BC	3B	2.00	1.50	0.30	0.40	95.8

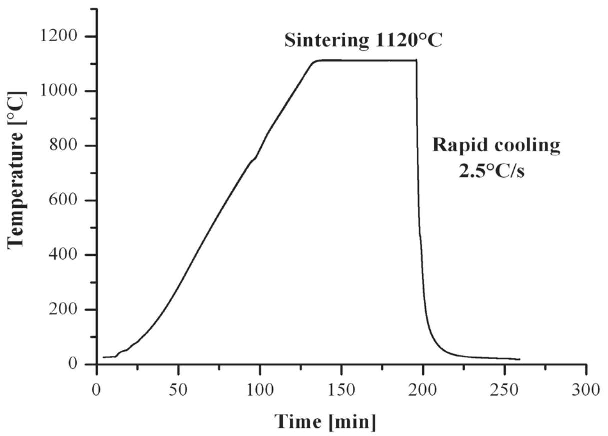

Fig. 2. Thermal cycle of sintering and rapid cooling of studied compositions

In order to evaluate the densities of studied Ni-Mo-(W) sinter-hardened steels, water displacement method was used. Microstructure was investigated using LEICA MEF4A light microscope after polishing samples and metallographic etching with Nital 2%.

Microhardness in the scale HV0.1 performed on studied materials was measured in every direction, starting from 20μm from a border and continuing each 30μm up to the centre on Vickers hardness intender.

Room temperature unnotched Charpy impact test was performed on all materials. In order to determine wear resistance both pin on disk and disk on disk tests were introduced.

Zwick Z100 machine was used to perform three-point bending test. Initial load applied was equal 10N and the speed 1.5mm/min. The mode of the probe was set for continuous controlling the crossbeam's position.

Pin-on-disk test was carried out through a tribometer with discs prepared from analyzed materials and the abrasive media – counterpin with 3 mm diameter made of WC-Co. The loads applied during experiment were 15N and 25N; the rotation speed of the disc was 0.26 m/s. Samples were tested on both sides and weighted several times up to 1000 meters of total sliding distance.

Disk on disk test was done on tribometer with 500N and 1000N of load (for samples with low and high concentration of carbon, respectively). Two discs having 40 mm diameter and 10 mm thickness, one made from the studied material the other from WC-Co, ran against each other, with the 10% difference between their rotation speeds. The speed selected for driver was 0.2 m/s and 0.18 m/s for driven disc. During the test, the weight change of samples was measured using weight with the sensibility of 10^{-4}g after each 100m, reaching 500m of the total sliding distance.

3. Results and discussion

The sintering and green density for steels obtained from powders is presented in Figure 3.

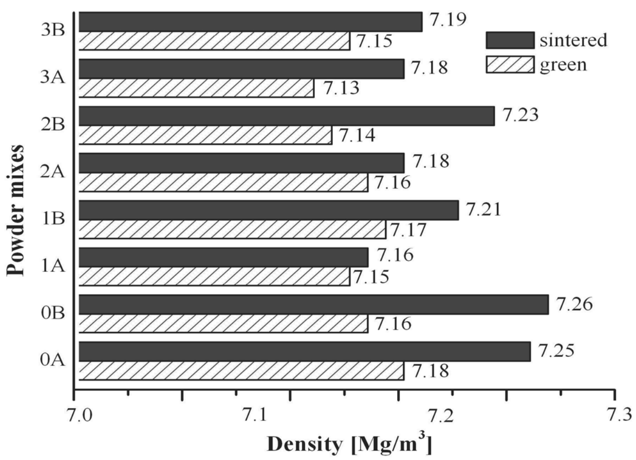

Fig. 3. Green and sintered density of studied compositions

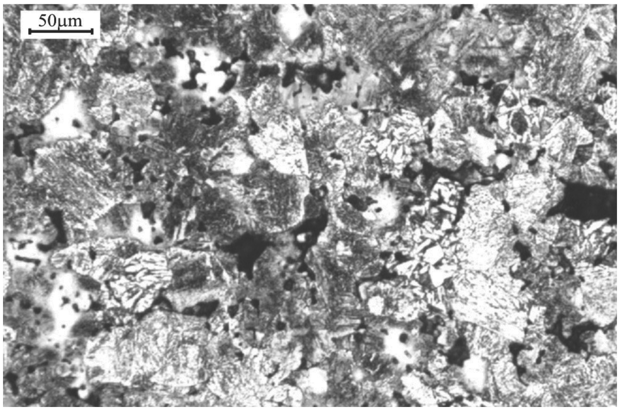

Fig. 4. Microstructure of composition 0B

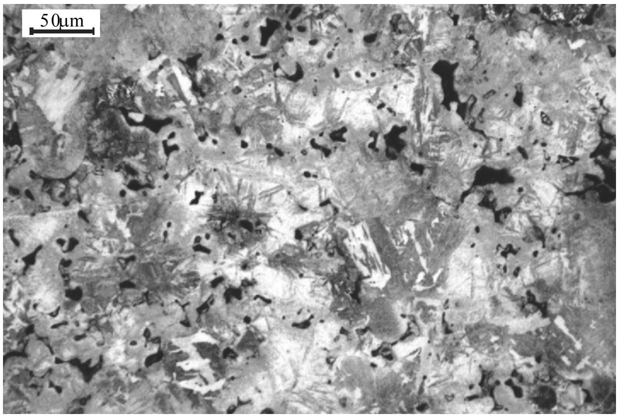

Fig. 5. Microstructure of composition 2A

The sintering density for steels obtained from powders without addition of tungsten and 0.4%C was 7.25Mg/m^3, while with 0.6%C – 7.26Mg/m^3.

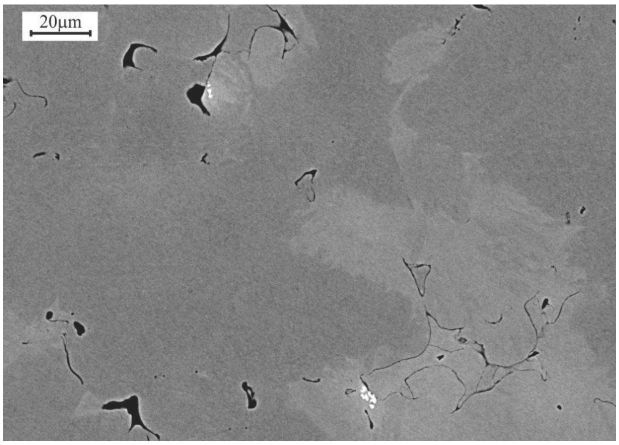

Fig. 6. Distribution of tungsten in mix 1A

Table 2.
Microhardness HV0.1 of Ni-Mo-(W) sinter-hardened alloys

0.4%C	0B	1B	2B	3B
	277.97	279.77	272.06	288.39
0.6%C	0A	1A	2A	3A
	377.6	390.83	452.25	437.04

Among steels with different amounts of tungsten the highest sintered density equal 7.23Mg/m^3 was achieved for steel marked as 2B, containing 0.4% of carbon and 0.2% of tungsten.

Metallographic observations of samples were carried out after their polishing and etching with nital 2%. All tested materials after the applied process of sintering for 1 hour in the temperature of 1120ºC and rapid cooling with the average cooling rate of 2.5ºC, proved to have microstructure mainly composed of martensite.

Microstructure of composition without addition of tungsten and with low carbon content (0B) shown in Figure 4, consisted of low carbon martensite, some retained austenite and lower bainite.

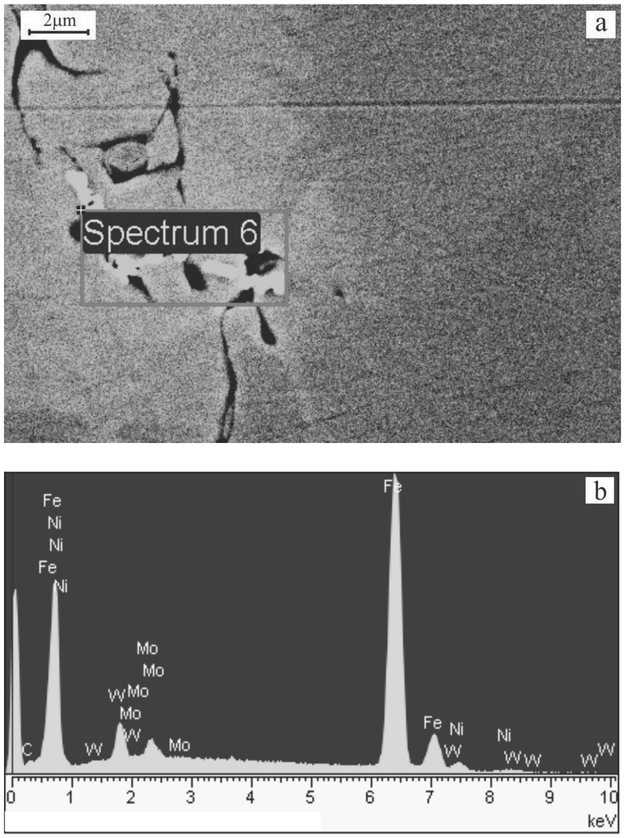

Fig. 7.a) SEM image of sintered steel designed as 3A (marked spectrum refers to agglomerates of tungsten),b) EDS analysis carried out on selected region

Table 3.
Chemical composition of investigated Ni-Mo-(W) sinter-hardened steels, determined by EDS analysis on defined grains

Compositio n designation	Elements concentration [%]									
	Fe		Ni		Mo		C		W	
	wt.	at.	wt.	at.	wt.	at.	wt.	at.	wt.	at.
0B	95.48	93.99	1.43	1.34	2.37	1.36	0.72	3.31	-	-
0A	95.74	95.95	1.23	1.17	2.76	1.61	0.27	1.26	-	-
1B	86.53	89.07	4.65	4.55	2.69	1.61	4.28	4.18	1.85	0.58
1A	88.71	91.50	1.73	1.70	2.21	1.33	4.77	4.66	2.57	0.81
2B	92.56	97.39	2.35	2.35	3.06	1.88	0.43	2.03	2.53	0.81
2A	89.38	93.05	4.00	3.96	3.10	1.88	0.56	2.70	3.53	1.12
3B	88.86	91.50	1.88	1.84	2.85	1.71	4.44	4.33	3.96	1.21
3A	90.28	93.72	2.58	2.54	2.88	1.74	0.14	0.69	4.12	1.30

Table 4.
Results of EDS analysis carried out on selected area of material 3A (spectrum 6)

Element	Chemical composition, %	
	Weight	Atomic
C	1.24	6.11
Fe	79.53	84.09
Ni	3.41	3.42
Mo	4.39	2.71
W	11.43	3.67
Total	100.00	100.00

The average microhardness of that steel was approximately equal 278HV0.1. Respectively higher microhardness (377HV0.1) was noted when increasing the amount of carbon to 0.6% (0A). Microstructure of that composition apart from martensite and retained austenite contained also finer martensite. The microstructure of materials containing various additions of tungsten and 0.6%C consisted of martensite, lower bainite and smaller amount of retained austenite, especially when comparing to material 0A without added tungsten.

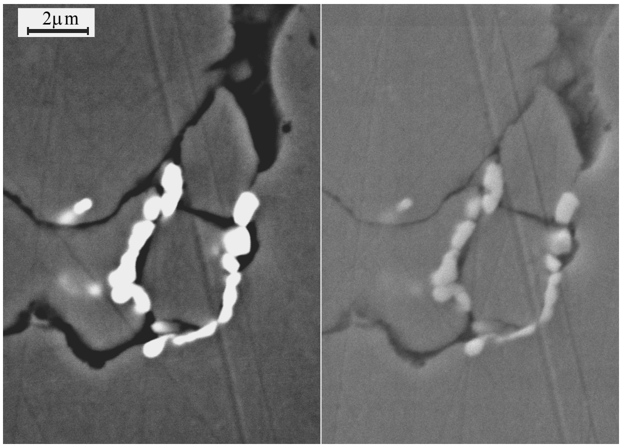

Fig. 8. Detail of the distribution of W in mix 3B

Among these steels, the highest value of microhardness was achieved for material composed with addition of 0.2% of tungsten and reached 452HV0.1. The microstructure of mix designed as 2A is presented in Figure 5. The results of microhardness test in the scale HV0.1 for studied sinter-hardened alloys are shown in Table 2. Their analysis revealed that addition of tungsten in studied Ni-Mo-W sinter-hardened steels as well as higher carbon content caused significant growth of hardness.

Chemical composition analysis of individual structural components in studied materials, obtained during energy dispersive spectroscopy examinations on the scanning electron microscope, shows the nickel rich areas seen as bright regions as well as areas with higher concentration of Mo. These areas are visible as darker regions. Table 3 presents chemical composition of sintered steels determined by EDS whilst Figure 6 presents the distribution of tungsten, Ni and Mo-rich areas in the sinter-hardened steel, obtained from high carbon powder with addition of 0.1%W.

Distribution of W into the matrix has been evaluated with scanning electron microscopy analysis with EDS for phase distribution. Figure 7a demonstrates SEM image of high carbon sintered steel with addition of 0.3%W, with marked spectrum for which EDS analysis has been carried out (Figure 7b). The studies of image indicate that tungsten was observed preferentially and has been accumulated in the proximity of pores. The dimension of tungsten agglomerates can be presented as a function of the amount of added tungsten. The energy dispersive spectroscopy analysis results for selected spectrum of composition 3A are set together in Table 4. The distribution of tungsten into material matrix was evaluated also by applying Back Scattered Electrons. Figure 8 refers to sintered steel with higher content of W – 0.3% and lower amount of carbon, designed as 3B.

The values of impact energy for investigated steels were in the range 21J to 27.2J. The highest impact energy was noted for steels produced with 0.4%C (set of compositions B). The tungsten addition shows minor influence on impact energy; for steels containing high carbon content and reaches the average of 21.7 J/cm^2.

Material 0B containing 0.4%C without addition of tungsten shows the best result of impact energy equal 27.2 J/cm^2. Equivalent values of impact energy (24.8J/cm^2) were noted for low carbon mixes - composition 1B (0.1%W) and the one containing 0.3%W marked as 3B. Plot in Figure 9 refers to values of impact energy for studied Ni-Mo-(W) sinter-hardened steels.

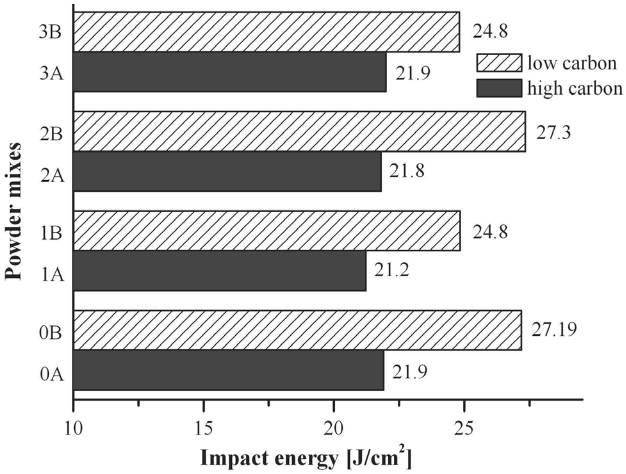

Fig. 9. Comparison of impact energy for investigated Ni-Mo-(W) sintered steels

Performed fractography analysis demonstrates that fracture surfaces of all steels are a mixed type of ductile and brittle fracture. Fracture surfaces are composed of wide and deep dimples in case of composition 0B (Figure 10)., ductile dimples are smaller and shallow when analyzing the material containing addition of 0.3%W and high carbon marked as 3A (Figure 11). In all studied steels the brittle cleavages of martensitic microstructure and tungsten agglomerates were revealed.

According to three-point bending test it was shown that the highest value σ_{max}=1713MPa of maximum stress was achieved for steel designated as 0A, containing no addition of tungsten and high amount of carbon. Tungsten addition decreased σ_{max} and for steels 1A, 2A and 3A reached the average value of 1630MPa. A definite decrease in strength was observed when reducing carbon content to 0.4%. Within materials with lower concentration of carbon the highest value of σ_{max} was noted for steel 1B composed with addition of 0.1%W.

During the test also deformation was noted. It differed for each set of steels, for high and low carbon respectively, as follows. For materials with 0.6% of carbon (A) average ε was equal 1.9% (the lowest value obtained for material marked as 3A), for materials with 0.4%C (B) deformation reached the average of 2.23%. The analysis of three-point bending test allow to point out that addition of tungsten leads to decrease of deformation. Greater influence on deformation and maximum stress has addition of carbon. High carbon concentration enhances maximum stress and decreases deformation. Opposite behavior was noticed for investigated low carbon Ni-Mo-(W) sinter-hardened steels. Maximum stress and deformation as a function of different amounts of tungsten, low and high carbon content are shown in Figure 12.

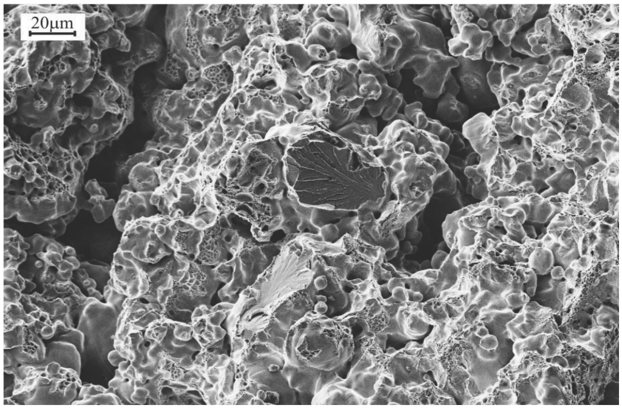

Fig. 10. SEM image of fracture surface of low carbon Ni-Mo steel without addition of tungsten (0B)

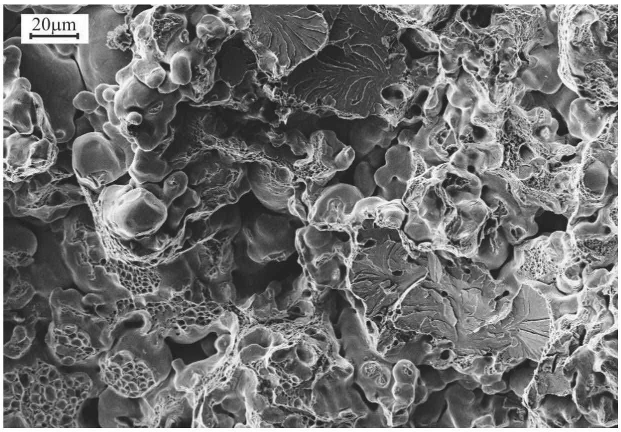

Fig. 11. SEM image of fracture surface of high carbon Ni-Mo steel with addition of 0.3%W (3A)

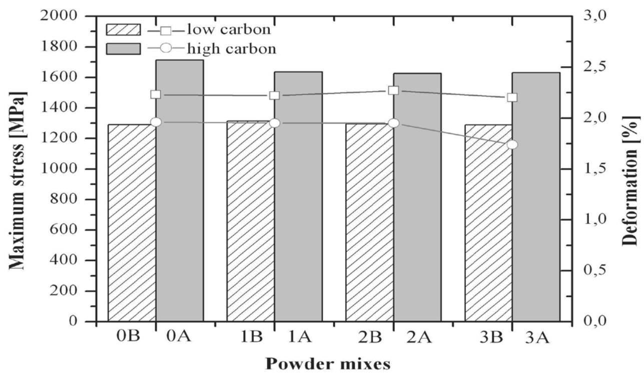

Fig. 12. Maximum stress and deformation as a function of different amounts of tungsten, low and high carbon content

The subsequent step of investigation was the wear resistance evaluation for sinter-hardened Ni-Mo-(W) steels. Results of performed pin-on-disk wear test presented the highest relative mass loss for steels without addition of tungsten and low carbon content, while the most resistant to abrasion was composition 2A where mass loss noted was only 0.0001%. The analysis of pin-on-disk results indicate that addition of tungsten leads to significant improvement of wear resistance. High resistance to abrasion of investigated materials, especially those with high carbon concentration, forced the change of applied load to 25N while initially applied was equal 15N. Figure 13 presents the wear resistance as function of the different amounts of tungsten for investigated compositions tested with pin-on-disk.

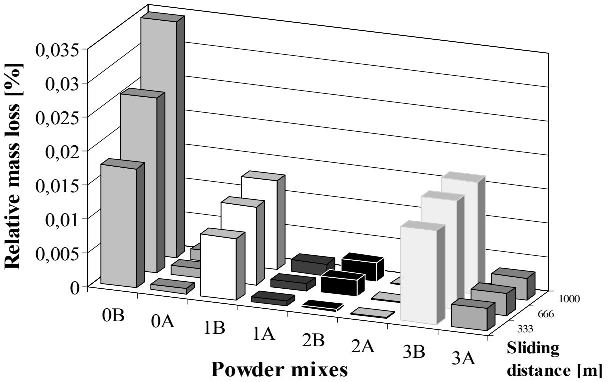

Fig. 13. Wear resistance as a function of different amounts of W for investigated compositions tested with pin-on-disk

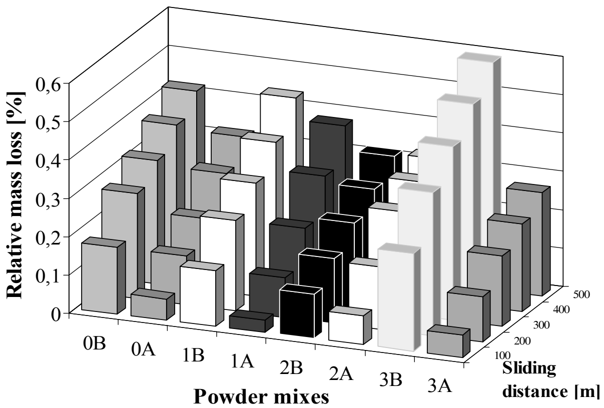

Fig. 14. Wear resistance as a function of different amounts of W for investigated compositions tested with disk-on-disk

Analysis of disk on disk test confirmed that the materials with addition of 0.3%W and 0.4%C are less resistant to abrasion when comparing to the others containing lower tungsten and for steel 3B the relative weight loss after 500m of sliding distance and load 50N was equal 0.59%. In case of steels composed with higher carbon content the load applied had to be changed to 100N. This change was caused with same reasons as mentioned in case of pin-on-disk. Figure 14 shows the results deriving from the disk-on-disk test expressed as a percentage relative mass loss in a function of sliding distance.

4. Conclusions

Investigation of Ni-Mo-(W) sinter-hardened steel alloys proved that the application of an average cooling rate of 2.5ºC/s determined the formation of mainly martensitic microstructures with small amounts of lower bainite and retained austenite, characterized by relatively high hardness values.

Addition of W in analyzed steels causes decrease of sintered density. This effect is most noticeable in the case of compositions with high carbon addition. The studies on scanning microscope specify that tungsten was distributed preferentially, observed as accumulations in the proximity of pores. In the same time, addition of tungsten affects mechanical properties, decreasing maximum stress and deformation which may be a fault of poor tungsten distribution in the material matrix.

The research proved that by adding tungsten into Ni-Mo pre-alloy leads to essential improvement of wear resistance. For the steels with high carbon and addition of 0.2%W shows slight relative mass loss and is most resistant to abrasion, which was confirmed by pin-on-disk and disk-on-disk tests.

Performed fractography analysis demonstrated the presence of ductile dimple facets and brittle cleavages of martensitic microstructure and tungsten agglomerates. Addition of tungsten into Ni-Mo steels seem not to influence the impact energy, however, further studies of its influence on mechanical properties ought to be conducted.

According to the characteristics of studied steels, the applied cooling rate seems to be a good compromise for mechanical properties and microstructures. Studied materials show good resistance to abrasion which could place them among materials in the specific field of application, especially where that feature is desired and determines their use.

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