



# Sintering of Ni-Mo-W steels and their properties

L.A. Dobrzański <sup>a,\*</sup>, J. Otręba <sup>a</sup>, M. Actis Grande <sup>b</sup>, M. Rosso <sup>b</sup>

<sup>a</sup> Division of Materials Processing Technology, Management and Computer Techniques in Materials Science, Institute of Engineering Materials and Biomaterials, Silesian University of Technology, ul. Konarskiego 18a, 44-100 Gliwice, Poland

<sup>b</sup> Politecnico di Torino – Alessandria Campus, Viale T. Michel 5- 15100 Alessandria, Italy

\* Corresponding author: E-mail address: leszek.dobrzanski@posl.pl

Received 16.05.2006; accepted in revised form 25.01.2007

## ABSTRACT

**Purpose:** The paper focuses on microstructural and mechanical characteristics of Ni-Mo and Ni-Mo-W sintered steels with increasing amount of tungsten, low and high addition of carbon.

**Design/methodology/approach:** Prepared mixes of powders have been compacted at 700 MPa and sintered in a vacuum furnace with argon backfilling at 1120°C for 60 minutes; after sintering rapid cooling has been applied with an average cooling rate of 2,5°C/s. Produced sets of samples have been studied by scanning electron microscopy (SEM) with EDS for phase distribution and mapping and light optical microscopy (LOM) for microstructure observations. Mechanical properties such as impact energy, microhardness and wear rates were evaluated depending on chemical composition and the effect of applied sintering process with rapid cooling. Wear resistance was investigated using both pin on disk and disk on disk tests.

**Findings:** The highest value of impact energy was achieved for set of steels with smaller amount of carbon. It was noticed that the presence of tungsten didn't differ much the results. The situation was opposite in the case of microhardness, where the best results were obtained for set with addition of 0.6%C and reached 452 HV0.1. The microstructure of all investigated alloys was mainly martensitic with minor presence of bainite in the set of steels containing low addition of carbon.

**Research limitations/implications:** According to the powders characteristic, the applied cooling rate seems to be a good compromise for mechanical properties and microstructures, nevertheless further tests should be carried out in order to examine different cooling rates and parameters of tempering process.

**Originality/value:** The effect of small additions of W and WC to low alloyed steels, especially in terms of hardenability and wear resistance, was investigated.

**Keywords:** Manufacturing and processing; Powder metallurgy; Sintering; Low alloyed steels; Sinter-hardening

## PROPERTIES

### 1. Introduction

The development of powder metallurgy increased in past years as an alternative technology characterized by low cost and no need of metal working processes such as machining, stamping and forging. Growth of powder metallurgy goes together with all

technological advancements through development of new alloys, processes to obtain powders, production of lubricants and near net shaped parts. Basically four different types of powder systems are available in ferrous powder metallurgy: iron powder simply admixed with alloying elements (such as copper, nickel and molybdenum), iron pre-alloyed (generally with Mo or Ni) via diffusion bonding, steel powders obtained via atomization and

finally hybrid systems as a combination of pre-alloyed, alloyed Fe and addition of copper and nickel [1-5].

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. 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].

Relative small impact on compressibility if compared to other alloying elements, together with good response in hardenability, made molybdenum 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 [7-12].

The role of powder metallurgy steel compacts prepared from Fe-Mo pre-alloyed powders is growing due to the possibility to obtain high density powder mixtures and homogenous or heterogeneous microstructures, which promote tensile and fatigue strength (the degree of heterogeneity varies with the carbon concentration).

High compressibility allows producing green compacts with green density reaching  $7.3 \text{ Mg/m}^3$ , promotes sintering density and improves pore morphology without the need of applying double pressing and double sintering processes. Presence of heterogeneous microstructures and high densification gives higher fatigue properties as an important advantage in many applications and allows Fe-Mo pre-alloyed powders, in part with addition of Ni through admixing or diffusion bonding, to be used for sinter-hardening [8, 13].

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. Cost reducing, technical and manufacturing economy, improved process efficiency, 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 and some important advantages can derive from its use [14-19].

## 2. Experimental procedure

Different compositions have been tested in order to investigate the influence of various tungsten additions followed

by low (0.4%) and high (0.6%) carbon content. The chemical composition of powders used in this study and their designation are listed in Table 1.

Samples were obtained using a 2000 kN hydraulic press with applied pressure of 700MPa. Rectangular (10x55) and a disk shaped mould (40mm diameter) were used in order to prepare samples for Charpy, three-point bending and both pin on disk and disk on disk tests.

Before the sintering process, the debinding was performed at 550°C for 60 minutes in a fully nitrogen atmosphere. 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 1120°C for 1h and rapidly cooled with a rate 2.5°C/s. Figure 1 presents the temperature performance of sintering and cooling applied.

Water displacement method was used to evaluate the densities of sintered steels. Microstructure was investigated using LEICA MEF4A light microscope after polishing samples and metallographic etching with Nital 2%. Microhardness (HV0.1) test was conducted on each surface using Vickers hardness indenter.

Unnotched Charpy impact test was performed on all materials at room temperature. In order to determine wear resistance both pin on disk and disk on disk tests were introduced. Pin on disk test was carried out through a tribometer entirely developed in the Alessandria Campus of Politecnico di Torino.

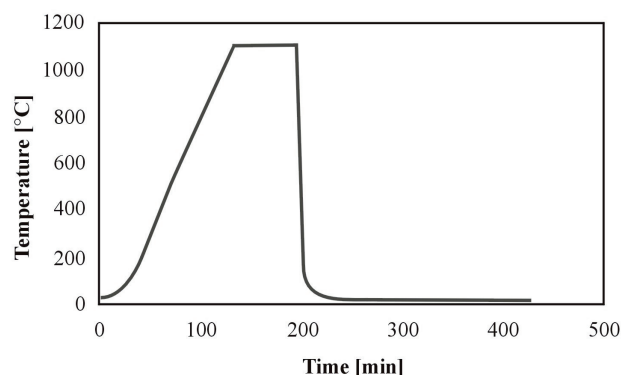


Fig. 1. Temperature performance during sintering and rapid cooling

Discs used in test were prepared from analyzed materials and the abrasive media - counterpin with 3 mm diameter made of WC-Co. The loads applied during experiment were 15 and 25N; the rotation speed of the disc was 0.26 m/s. Steels were tested on both sides and weighted several times up to 1000 meters of sliding distance.

Disk on disk test was done on tribometer with 500 N and 1000 N of load (for steels 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 selected speed was 0.2 m/s for driver and 0.18 m/s for driven disc.

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	Bal.
W0-BC	0B	2.00	1.50	-	0.40	Bal.
W1-AC	1A	2.00	1.50	0.10	0.60	Bal.
W2-BC	1B	2.00	1.50	0.10	0.40	Bal.
W2-AC	2A	2.00	1.50	0.20	0.60	Bal.
W2-BC	2B	2.00	1.50	0.20	0.40	Bal.
W3-AC	3A	2.00	1.50	0.30	0.60	Bal.
W3-BC	3B	2.00	1.50	0.30	0.40	Bal.

Table 2.  
Sintered density of studied compositions

	Steel							
	low carbon content				high carbon content			
	0B	1B	2B	3B	0A	1A	2A	3A
Density [Mg/m <sup>3</sup> ]	7.26	7.14	7.23	7.19	7.25	7.20	7.19	7.18

During the test, the weight change of samples was measured using weight with the sensibility of  $10^{-4}$ g after each 100 m reaching 500 m of the total sliding distance.

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

### 3. Description of results

Water displacement method used for evaluating the density after sintering effected with values of 7.25 and 7.26 Mg/m<sup>3</sup> for steels with no presence of tungsten, 0.6 and 0.4% C, respectively. Among specimens with different additions of tungsten, the highest density equal 7.23 Mg/m<sup>3</sup> obtained the material marked as 2B containing 0.2%W and low carbon content. The results of sintered density are presented in Table 2.

Metallographic observations of steels were carried out after their polishing and etching with nital 2%. It could be noticed that all tested materials after the applied process of sinter-hardening proved to have microstructure mainly composed of martensite.

Microstructure of composition without addition of tungsten and with low carbon content (0B) consisted of low carbon martensite, some retained austenite and lower bainite. Similar microstructures were observed for steel with high carbon concentration, where apart from martensite and retained austenite also finer martensite was detected. Steel containing same quantity of W, but higher carbon content consists of martensite, lower bainite and retained austenite in smaller amount when comparing to steel 0A without added tungsten. Microstructures obtained for steels with 0.1%W containing respectively low and high carbon content are shown in Figures 2 and 4, those for compositions with addition of 0.3%W are presented in Figures 3 and 5.

During examinations on the scanning electron microscope the EDS analysis were achieved. Chemical composition analysis of individual structural components in studied materials shows the Ni-rich areas (bright regions) as well as areas where the presence of Mo is higher (darker regions). Diffusion of tungsten to the material matrix created W-rich regions. Figures 6 to 9 show the microstructure after SEM/EDS analysis. On these figures not resolved tungsten powder can be seen. Table 3 shows the concentrations of major alloying elements determined by EDS analysis in investigated sintered alloys.

Higher values of impact energy were achieved for materials admixed 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<sup>2</sup>. Material 0B containing 0.4%C without addition of tungsten shows the best result of impact energy and is equal 27.2 J/cm<sup>2</sup>. Similarity can be observed for steels 1B (0.1%W) and 3B (0.3%W) where the impact energy is 24.8 J/cm<sup>2</sup>.

The measurements of microhardness performed on studied materials were led on each surface, starting from 20  $\mu$ m from a border and continuing each 30  $\mu$ m up to the centre. The highest value HV0.1 was achieved for steel containing 0.2%W with high carbon addition (2A). There are slight differences between results of measurement for set of steels marked with B, where the average was 280 HV0.1.

Three-point bending test resulted with a highest value 1713 MPa of maximum force for the steel designated as 0A, containing no addition of tungsten and high amount of carbon. Addition of tungsten decreased  $F_{max}$  and for steels 1A, 2A and 3A reached the average value of 1630 MPa. A definite decrease in strength was observed when reducing carbon content to 0.4%. Within steels with lower concentration of carbon the highest value of  $F_{max}$  was noted for steel 1B composed with addition of 0.1%W. Figures 10 and 11 present the force versus deformation for steels 0A and 1B, respectively.





Fig. 2. Microstructure of composition 1B

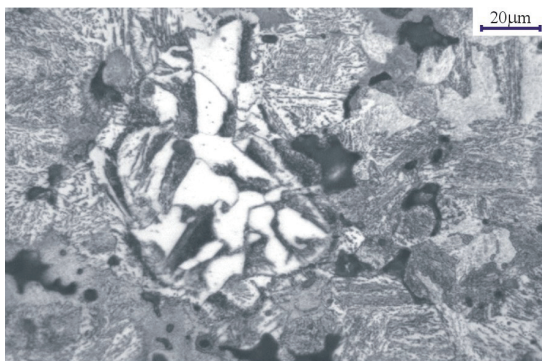


Fig. 3. Microstructure of composition 3B

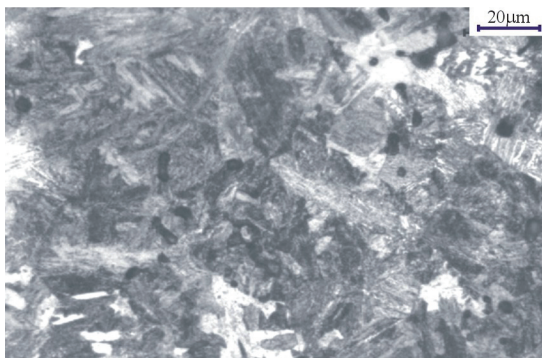


Fig. 4. Microstructure of composition 1A

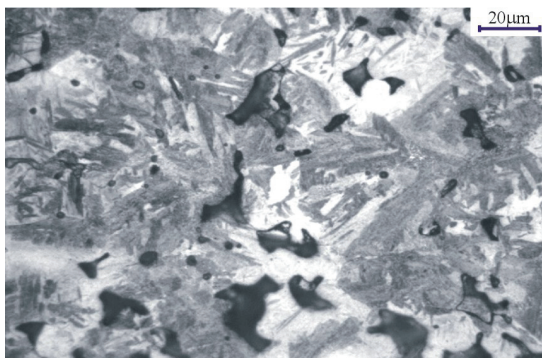


Fig. 5. Microstructure of composition 3A

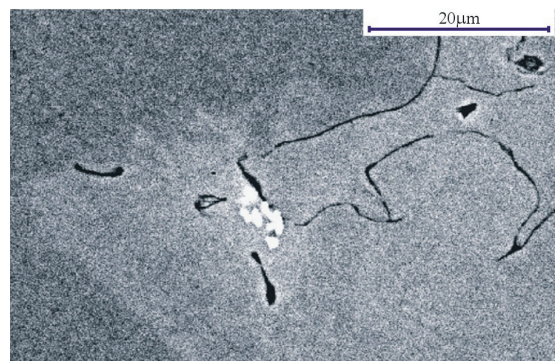


Fig. 6. SEM/EDS analysis carried out on composition 1B

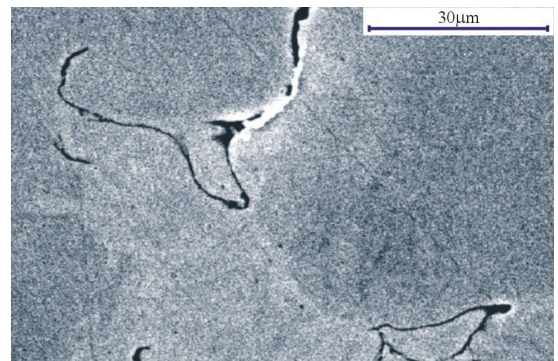


Fig. 7. SEM/EDS analysis carried out on composition 3B

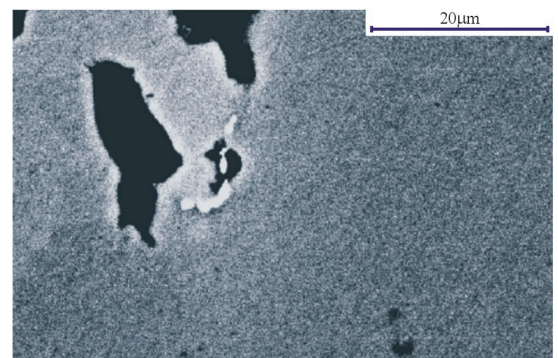


Fig. 8. SEM/EDS analysis carried out on composition 1A

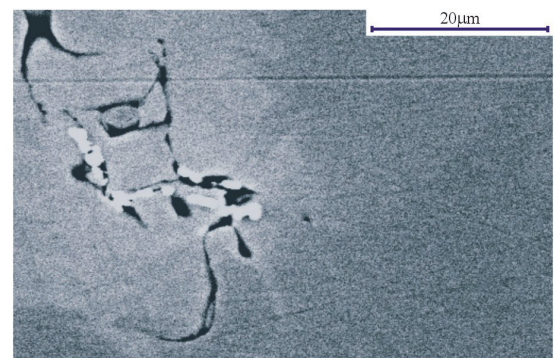


Fig. 9. SEM/EDS analysis carried out on composition 3A

Table 3.

Chemical composition of sintered steels, determined by EDS analysis on defined grains

Composition 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.

Mechanical properties of studied set of compositions

	Composition							
	low carbon content				high carbon content			
	0B	1B	2B	3B	0A	1A	2A	3A
IE [J/cm <sup>2</sup> ]	27.2	24.8	25.7	24.8	21.9	21.2	21.8	22.0
HV0.1	277.97	279.77	272.06	288.39	377.6	390.83	452.25	437.04
F <sub>max</sub> [MPa]	1290	1315	1295	1287	1714	1635	1625	1630
ε F <sub>max</sub> [%]	2.23	2.22	2.27	2.20	1.96	1.95	1.95	1.74

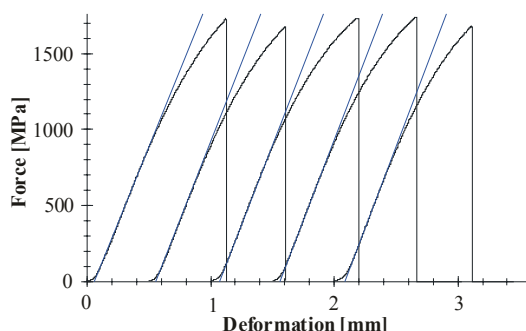


Fig. 10. The force versus deformation for steel 0A

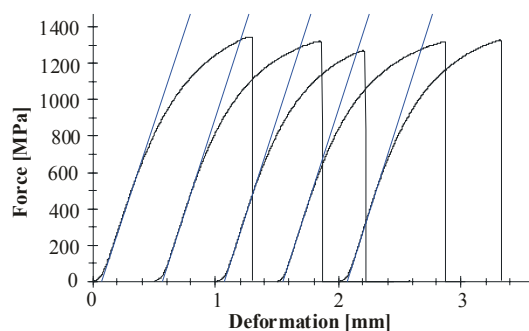


Fig. 11. The force versus deformation for steel 1B

The deformation was different for each set of steels. For compositions A average  $\epsilon$  noted was equal 1.9 % (the lowest value obtained for steel marked as 3A), for compositions B deformation reached the average of 2.23 %.

The next step of research was the wear resistance comparison for all investigated materials. Results of performed pin on disk

wear test presented the highest relative mass loss for steel 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 starting load applied during test was 15N and then changed to 25N due to high resistance of investigated materials. Figure 12 presents the correlation between sliding distance and relative mass loss for compositions B while Figure 13 for steels from a set A. Mechanical properties of investigated sintered alloys with low and high carbon content, respectively, are grouped in Table 4.

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 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 with high 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.

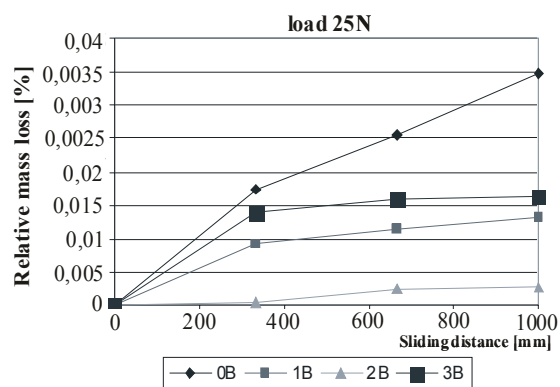


Fig. 12. Relative mass loss in function of sliding distance for B set



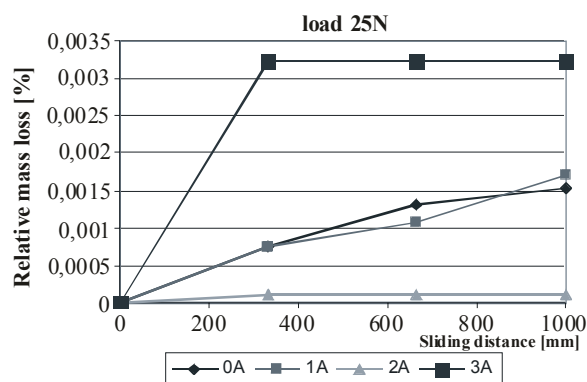


Fig. 13. Relative mass loss in function of sliding distance for A set of steels

## 4. Conclusions

The results achieved after the investigation of Ni-Mo and Ni-Mo-W sintered steels, with low and high carbon content and different amounts of tungsten, seem to be very promising. Applied process of sintering under vacuum and rapid cooling from the sintering temperature brought expected outcome.

Microstructure observations proved presence of mainly martensitic structure in all cases with minor occurrence of bainite in the low carbon steels. A marked difference in the results of mechanical properties was noted when increasing amount of carbon (compositions from a set A). No sensible differences were present among steels with same amount of carbon, but varied quantities of tungsten. The distribution of W into the matrix is nevertheless to be improved for a better increase of properties, especially in terms of wear resistance.

Investigated materials show good resistance to abrasion which could place them among materials in the specific field of application, where that feature is desired.

## Acknowledgements

Investigations were partially realized within the framework of the Socrates Erasmus programme, in Politecnico di Torino, Campus Alessandria, Italy.

## Additional information

The presentation connected with the subject matter of the paper was presented by the authors during the 11<sup>th</sup> International Scientific Conference on Contemporary Achievements in Mechanics, Manufacturing and Materials Science CAM3S'2005 in Gliwice-Zakopane, Poland on 6<sup>th</sup>-9<sup>th</sup> December 2005.

## References

[1] K.S. Narasimhan, Sintering of powder mixtures and the growth of ferrous powder metallurgy, *Materials Chemistry and Physics* 67 (2001) 56-65.

[2] L.A. Dobrzański, J. Otręba, M. Actis Grande, M. Rosso, Mechanical and microstructural properties of Ni-Mo and Ni-Mo sintered steels, *Proceedings of the 13<sup>th</sup> Scientific Conference „Achievements in Mechanical and Materials Engineering” AMME'2005*, Gliwice-Zakopane, Poland, 2005, 240-246.

[3] G.B. Jang, M.D. Hur, S.S. Kang, A study on the development of a substitution process by powder metallurgy in automobile parts, *Journal of Materials Processing Technology* 100 (2000) 110-115.

[4] K. Yamaguchi, N. Takakura, S. Imatani, Compaction and sintering characteristics of composite metal powders, *Journal of Materials Processing Technology* 63 (1997) 364-369.

[5] R.J. Causton, The development of high performance P/M steels, *Technical Bulletin*, Hoeganaes Corporation NJ 08077 (1996).

[6] M. Actis Grande, D. Ugues, M. Rosso, L.A. Dobrzański, Z. Brytan, The PM route for duplex stainless steels, *Proceedings of the 12<sup>th</sup> Scientific Conference „Achievements in Mechanical and Materials Engineering” AMME'2003*, Gliwice-Zakopane, Poland, 2003, 5-8.

[7] L.A. Dobrzański, Z. Brytan, M. Actis Grande, M. Rosso, E. J. Pallavicini, Properties of vacuum sintered Duplex Stainless Steels, *Journal of Materials Processing Technology* 157-158 (2004) 312-316.

[8] V. Stoyanova, C. Xu, L. Blanco, A. Molinari, H. Danninger, J. Torralba, Y. Yu, B. Lindquist, Influence of microstructure and porosity on tensile and impact properties of high performance PM steels, *Proceedings of Powder Metallurgy World Congress PM2004*, Vienna, Austria, 2004, 47-62.

[9] T.M. Cimino, C. Schade, R.J. Causton, High performance P/M stainless steel, *International Conference on Powder Metallurgy & Particular Materials PM2TEC'99*, Vancouver, Canada, 1998, 4-10.

[10] A. Molinari, V. Stoyanova, Sintering and sinter-hardening of low alloyed steels in vacuum, *Proceedings of Powder Metallurgy World Congress PM2004*, Vienna, Austria, 2004, vol. 1, 291-296.

[11] *Metals Handbook*, 9th edition, vol.7, ed. ASM Warrendale.

[12] T. Sakamoto, Effect of nickel iron and chromium on sintering of molybdenum powders, *Powder Metallurgy* 44 (1997) 689-693.

[13] F. Chagnon, L. Tremblay, Effect of post-sintering cooling rate on properties of diffusion bonded steel materials, *Proceedings of Powder Metallurgy World Congress PM2004*, Vienna, Austria, 2004, vol. 3, 244-248.

[14] J.M. Capus, Sinter hardening looks ahead, *Metal Powder Report* 55 (2000) 19-21.

[15] L.A. Dobrzański, Z. Brytan, M. Actis Grande, M. Rosso, Structure and properties of sintered duplex steels, *Proceedings of the 12<sup>th</sup> Scientific Conference „Achievements in Mechanical and Materials Engineering” AMME'2003*, Gliwice-Zakopane, Poland, 2003, 211-215.

[16] L.A. Dobrzański, J. Otręba, M. Actis Grande, M. Rosso, Sinter-hardening of Ni-Mo pre-alloyed powders with tungsten addition, *Journal of Achievements in Materials and Manufacturing Engineering* 20 (2007) 427-430.

[17] L.A. Dobrzański, J. Otręba, M. Actis Grande, M. Rosso, Microstructural characteristic and mechanical properties of Ni-Mo-(W) steels, *Journal of Achievements in Materials and Manufacturing Engineering* 18 (2006) 347-250.

[18] H. Zhang, et al., *Advances in Powder Metal and Particulate Materials* 1 (2001) 695-708.

[19] N. Khattab, J. M. Torralba, E. Gordo, V. Trabadelo, I. Iturriza, Development of HCxO stainless steel - based materials for its application as valve seat inserts in diesel engines, *Proceedings of Powder Metallurgy World Congress PM2003*, Valencia, Spain, 2003, 225-231.