Rev.Adv.Mater.Sci. 8 (2004) 143-146

PHASE TRANSFORMATIONS IN BALL MILLED AISI 316L STAINLESS STEEL POWDER AND THE MICROSTRUCTURE OF THE STEEL OBTAINED BY ITS SINTERING

Agnieszka Szymańska[1]**, Dariusz Oleszak**[1]**, Agnieszka Grabias**[2]**,**
Marcin Rosiński[1]**, Krzysztof Sikorski**[1]**, Jan Kazior**[3]**, Andrzej Michalski**[1] **and**
Krzysztof J. Kurzydłowski[1]

[1]Warsaw University of Technology, Faculty of Materials Science and Engineering, 141 Wołoska Str.
02-507 Warsaw, Poland
[2]Institute of Electronic Materials Technology (ITME), 133 Wolczynska Str. 01-919 Warsaw, Poland
[3]Cracow University of Technology Institute of Materials Science and Metal Technology, 24 Warszawska Str.
31-155, Cracow, Poland

Recfeived: December 14, 2004

Abstract: The paper shows the influence of the 100 hours ball milling process of commercial AISI 316L stainless steel powder and the mixture of powders of its components eg. (Fe, Cr, Ni…) on the structure on the milling products. The impulse plasma sintering process of the powders has also been carried out. The results of X-ray diffractometry showed that ball milling in both cases leads to obtaining nanocrystalline two phase (austenitic-martensitic) stainless steel powders of the average size of crystallite about 40 nm. The fraction of martensite in the milled powders was estimated from Mössbauer spectra and their microstructure was observed on scanning electron microscope. The milled powders were sintered by impulse plasma sintering. The structure of the sintered material was characterized by X-ray diffractometry and light microscopy. It was found that impulse plasma sintering of nanocrystalline powders obtained by ball milling of pure components enable to obtain a nanocrystalline austenitic stainless steel of very low porosity.

1. INTRODUCTION

Mechanical properties of stainless steel can be improved by grain size refirement, now down to nanocrystalline structure. Ball milling is regarded as an effective way to obtaining nanocrystalline materials. However, decreasing of crystallite size during ball milling of steel powders is usually accompanied by phase transformations of the material studied [1-4] Nano-ferrite formation and strain-indused-ferrite transformation in austenitic stainless steel were reported in [1,2]. Also the formation of martensite in ball milled 316L austenitic steel was observed as a result of ball milling [3]. The ball milling process of tool carbon steel powders led to the dissolution of cementite and formation of nano-ferrite, characterized by crystallite size and the lattice strain of 10 nm and 1%, respectively.

The aim of this work to check the possibility of obtaining nanocrystalline austenitic stainless steel by impulse plasma sintering of nanocrystalline stainless steel powders obtained by (i) ball milling of commercial Höganäs austenitic AISI 316L stainless steel powder and (ii) mechanical alloying of elemental powders mixture.

Corresponding author: Krzysztof Sikorski, e-mail: sikorski@inmat.pw.edu.pl

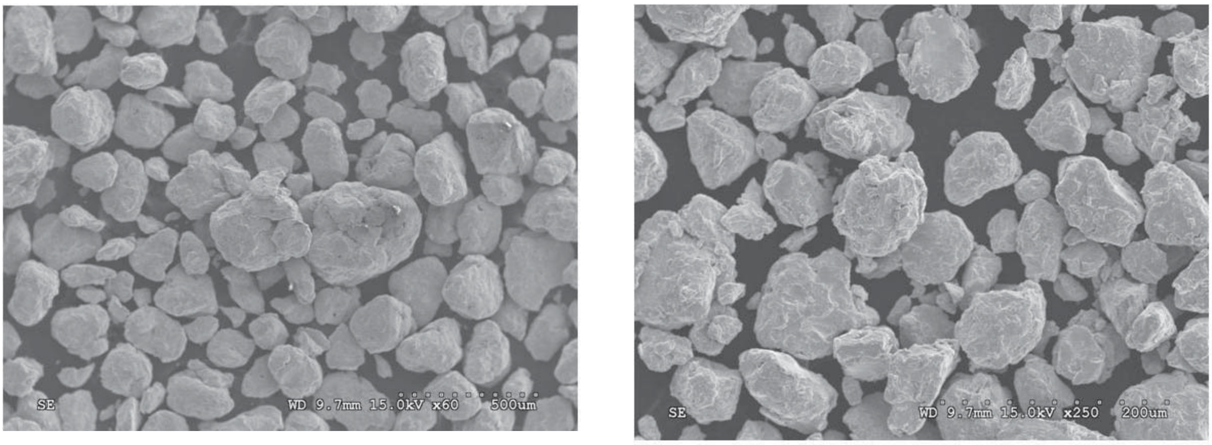

Fig. 1. SEM image of stainless steel powders after 100 hours milling of: commercial AISI 316L – a), austenitic steel obtained from pure elements – b).

2. EXPERIMENTAL

The water - atomised, Höganäs austenitic AISI 316L stainless steel powder and the mixture of pure component powders of this steel (e.g. Fe,Cr,Ni…) were subjected to ball milling process for 100 hours in a Fritsch P5 planetary ball mill equipped with hardened steel vials and balls. The ball-to-powder weight ratio was 10:1. All powder handling was performed in a glove box under argon atmosphere. Chemical composition of the mixture of elemental powders given in Table 1, was the same as the Höganäs austenitic AISI 316L powder. The initial mean sizes of the powders were also comparable (50 and 52 μm). After ball milling process the powders were characterized using SEM and XRD methods. The X-ray investigations were performed on a Philips PW 1830 diffractometer, using CuK$_\alpha$ (λ=0.15418 nm) radiation. The average size of crystallites was estimated from broadening of the diffraction peaks using Scherer formula. The fraction of martensite in the milled powders was estimated from transmission Mössbauer spectra, measured at room temperature using a ^{57}Co-in-Rh source.

After milling the powders were sintered in vacuum at 1050 °C for 3 minutes, by impulse plasma sintering method [5]. The sintered specimens thus obtained were characterized using X-ray diffractometer and optical microscopy.

3. RESULTS

SEM images of the powders obtained by 100 hours ball milling of the commercial Höganäs AISI 316L powder and the powder obtained from pure components of the steel are given in Figs. 1a and 1b. After milling the particles form polyhedron agglomerates of the average size of 250 μm in the case of the milled commercial 316L steel powder and 100 μm in the case of the milled pure chemical components powder.

Comparison of X-ray diffraction patterns of the milled powders with that for the commercial powder is shown in Fig. 2. One can see that ball milling of the commercial AISI 316L powder as well as the mixture of component powders bring about two phases (austenitic-martensitic) powders. In the present case for both 316L powder and the mixture powders the diffraction patterns are similar. It proves that stainless steel powder can be obtained by mechanical alloying of elemental powders. On the other hand the broadening of the diffraction lines reveals nanocrystalline structure of the powders after milling. The average size of crystallites estimated from Scherer formula equals to 35 nm for ball milled 316L powder and 42 nm for the ball milled mixture of the component powders.

Transmission Mössbauer spectra of the analysed powder are illustrated in the Fig. 3. The

Table 1. Chemical composition of the used powders.

Element concentration [wt.%]							
symbol of steel	Cr	Ni	Mo	Mn	Si	C	Fe
316L	17	13	2.2	0.2	0.8	0.02	balance

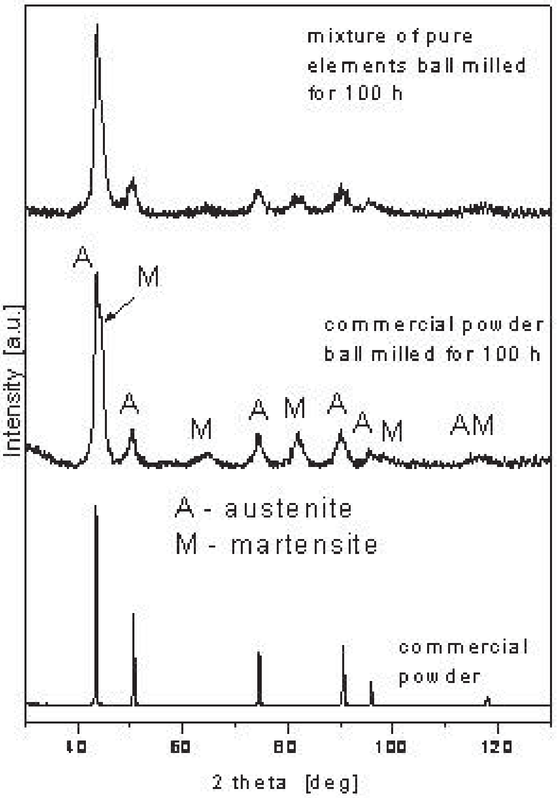

Fig. 2. X-ray diffraction patterns of the examined stainless steel powders.

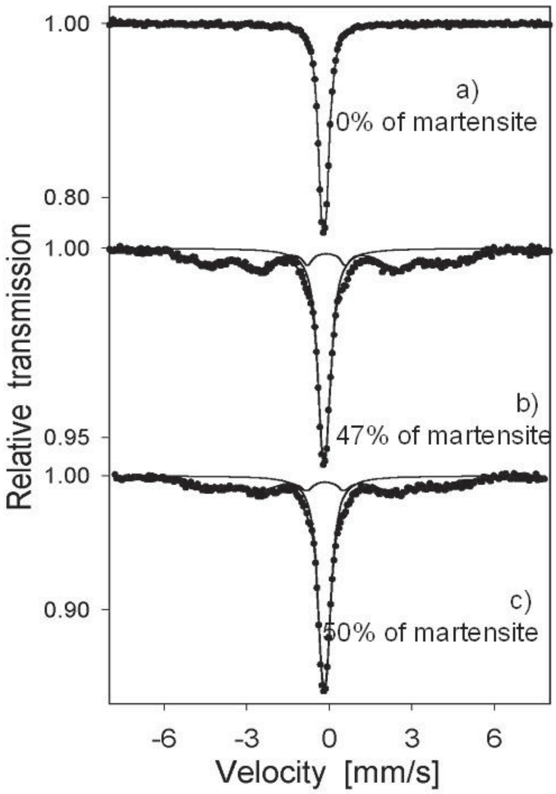

Fig. 3. The Mössbauer spectra of the examined powders: commercial AISI 316L – a), obtained by mechanical alloying of the mixture of element powders – b), obtained by milling of the AISI 316L powder – c).

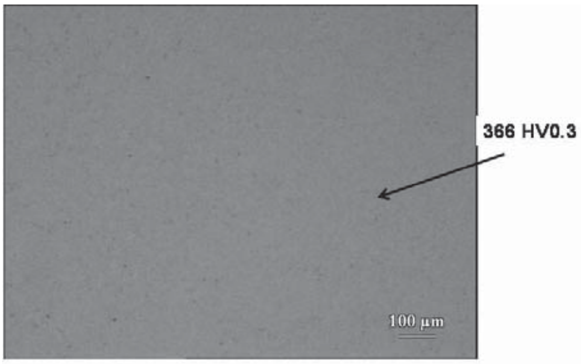

Fig. 4. Polished cross section of the steels obtained by sintering the milled mixture of pure components powders.

Mössbauer spectrum of the starting commercial powder (Fig. 3a) consists of the paramagnetic component characteristic for the austenitic phase. The quadrupole doublet observed at the centre of the spectrum is characterized by a small quadrupole splitting of 0.18 mm/s and isomer shift of -0.10 mm/s. After 100 h of milling a magnetic six-line component appears in the spectrum (Fig. 3b). This sextet with characteristic broad lines and the hyperfine field of 26.8 T is related to the martensite phase formed from the austenite during the milling process. Its relative content of 50% was calculated as a ratio of the area of the sextet to the total spectral area. The Mössbauer spectrum of the mixture of pure component powders milled for 100 h is very similar to that obtained for the as-milled commercial powder (Fig. 3c). The hyperfine parameters related to the austenite and martensite phases formed due to the milling correspond very well to those calculated for the commercial powder. The relative content of the martensite is slightly lower (46%) for the alloy prepared by ball milling of the mixture of pure component powders. Hyperfine parameters: quadrupole splitting (QS), hyperfine field (H_{hf}), isomer shift (IS), and relative content (A) for the phases observed in the 316 steel powders determined from the Mössbauer spectra are quoted in the Table 2.

The polished cross section of the steel obtained by sintering of the milled mixture of pure component powders is shown in Fig. 4. One can see that

Table 2. Hyperfine parameters of the phases observed in the 316L steel powders determined from the Mössbauer spectra.

Sample	Austenite			Martensite		
	QS [mm/s]	IS [mm/s]	A [%]	H_{hf} [T]	IS [mm/s]	A [%]
commercial AISI 316L powder	0.18	-0.10	100	—	—	—
powder after 100 h of milling	0.18	-0.08	50	26.8	-0.01	50
mixture of pure component powders after 100 h of milling	0.18	-0.09	54	26.5	-0.05	46

the sinter of materials is almost free of pores (the hardness is 366 $HV_{0.3}$). X-ray diffraction pattern of the steel is shown in Fig. 5. The average size of crystallites in the steel, estimated from Scherer formula, equals to 72 nm. The result of X-ray diffractometry showed that the austenitic nanocrystalline stainless steel can be produced by impulse plasma sintering of commercial AISI 316L powder or by sintering of mechanically alloyed elemental powders mixture.

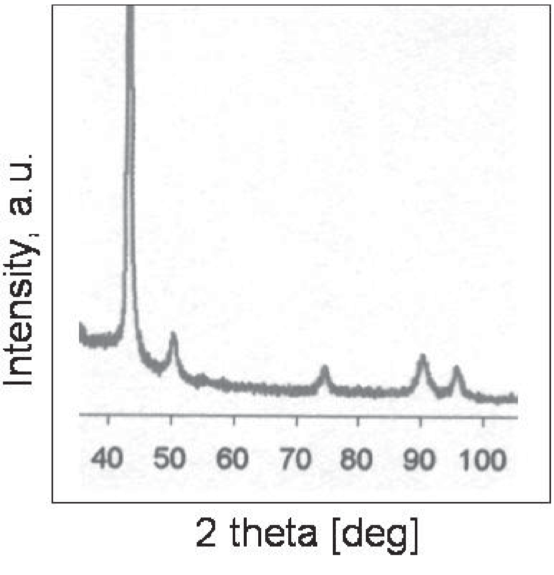

Fig. 5. X-ray diffraction patterns of the steel obtained by sintering of the ball milled mixture of pure component powders.

4. CONCLUSIONS

1) Ball milling of the mixture of pure elemental powders leads to obtaining of the nanocrystalline two phase (austenitic-martensitic) stainless steel powder;
2) Ball milling of the commercial AISI 316L steel powder brings to appearing of the martensitic phase;
3) The average size of crystallites, estimated from Scherer formula, equals to 35 nm for ball milled 316L powder and 42 nm for the ball milled mixture of pure elemental powders;
4) Impulse plasma sintering of nanocrystalline powders obtained by ball milling of the mixture of pure component powders enable to obtain nanocrystalline austenitic stainless steel.

ACKNOWLEDGEMENT

This work was supported by State Committee for Scientific Research - Projects No 4T08D 048 24 and No 4T08B 040 23.

REFERENCES

[1] Z. G. Liu, X.J. Hao, K. Masuyama and M. Umemoto // *Scripta mater.* **44** (2001) 1775.

[2] M. Umemoto, Z. G. Liu, Y. Xu and K. Tsuchiya // *Mater. Sci. Forum* **386-388** (2002) 323.

[3] D. Oleszak, A. Grabias and T. Kulik // *Arcives of Materials Science*, in press.

[4] D. Oleszak, A. Janczewki and A. Grabias // *Solid State Phenomena* **101-102** (2004) 165.

[5] A. Michalski, M. Krasnowski, M. Rosinski and D. Siemiaszko, In: *Proceedings of the 15-th International Symposium on Plasma Chemisrty*, ed. by A. Bouchoule, J.M. Pouvelsle, A.L. Thomann, J.M. Bauchire and E. Robert (Gremi, CNRS/University of Orleans, 2001, vol. VII) p. 2885.