Journal of Achievements in Materials and Manufacturing Engineering
VOLUME 21
ISSUE 2
April
2007

Processing and properties of sinters prepared from 316L steel nanopowders

H. Krztoń[a,*], D. Kolesnikow[b], J. Paducha[a], R. Molenda[a]
[a] Institute for Ferrous Metallurgy, ul. K. Miarki 12, 44-100 Gliwice, Poland
[b] Institute of High Pressure Physics Polish Academy of Sciences, ul. Sokołowska 29/37, 01-142 Warsaw, Poland
* Corresponding author: E-mail address: hkrzton@imz.gliwice.pl

Received 14.11.2006; accepted in revised form 15.11.2006

Manufacturing and processing

ABSTRACT

Purpose: The results of the research work on processing the sinters obtained from nanocrystalline powders of 316L steel are presented.

Design/methodology/approach: The 316L steel powder has been mechanically alloyed from a set of elementary powders with use of Fritsch Vario-Planetary Mill Pulverisette 4. The time of 12 hours of milling has been needed for producing the powder. The X-ray diffraction has been used for controlling of the mechanical alloying process. The Rietveld method has been used to calculate the contents of the components of the powder. Cold and hot isostatic pressing have been applied to make the compacts. The pressure of 500 MPa and 900 MPa of cold pressing, and 150 MPa of hot pressing have been used. The green compacts have been pressed isostaticly using liquid aluminum in the temperature of 950°C (1223 K). The X-ray diffraction have been used to identify the phase components of the sinters. The structure of the sinters have been observed using scanning electron microscope. The hardness values have been obtained by Vicker's test.

Findings: The mechanically alloyed powder has consisted of about 94 wt.% of austenite, 5 wt.% of ferrite and not more than 1 wt.% of not alloyed molybdenum. Two kinds of sinters have been produced, one kind made of pure 316L powder, second one obtained with aluminum infiltration within the volume of the sinters. The observed porosity of the sinters has depended on the applied pressing conditions strongly, mainly on the value of cold isostatic pressure. The hardness of the first kind of sinters have achieved a value of 380 HV (98N), the hardness of the second kind - more than 400 HV (98N).

Practical implications: The Al infiltrated sinter has been proposed as a material for a part of Diesel engine. As an example, a part of a fuel injection has been produced.

Originality/value: The nanocrystalline 316L powder has been obtained using mechanical alloying process. The original method of hot isostatic pressing in liquid aluminum has been proposed. This method enables to produce infiltrated sinters with low porosity and high hardness values.

Keywords: Mechanical alloying; Cold and hot isostatic pressing; X-ray diffraction; Hardness

1. Introduction

Powder metallurgy, despite of relatively high costs of production, is still a very important way of producing precision metal parts, e.g. the sintering products for automotive components [1]. The austenitic stainless steels, like 316L steel, are taken into account in these certain applications.

Steel powders are produced by various techniques, e.g. by pressurized gas atomization technique [2] and also by the mechanical alloying (MA) process. MA can be used to produce a very wide range of powder materials of various properties and applications as amorphous [3], nanocrystalline, intermetallics [4,5] or magnetic materials [6]. The starting materials can be also of various kinds, depending on the expected final result as an elementary powder, an elemental metal ribbon or a mixture of elementary powders.

The mechanical alloying process have been also proposed for producing the austenitic steel powders under a nitrogen atmosphere [7,8]. The mixtures of elementary powders with a chemical composition equivalent to chemical composition of a steel of interest, have been milled in an attrition mill with N_2 atmosphere under a constant flow rate. Two kinds of alloys have been investigated: Fe18Cr11Mn and Fe-18Cr-11Mn-5Mo-xN with various amounts of nitrogen. The austenitic steel powders have been obtained in both experiments.

There are many consolidation techniques in which the powder - also metal powder – can be formed, namely forging, rolling, extrusion, or pressing. All of them, especially isostatic pressing, are very expensive techniques, so the different numerical modelling have been applied to predict the properties of sintered powders. Also the densification behavior of 316L powder steel has been investigated using different models of finite element calculations [9, 10]. The best agreement between the model and the experimental results have been found for the isostastic pressing.

The isostatic pressing is the method which can fabricate the compacts of the highest density. In this study, both of isostatic pressing methods have been used – cold CIP and hot HIP – to produce compacts of the mechanically alloyed 316L powders. The steel powder has been processed from elementary powders in a planetary ball mill.

2. Experimental procedure

2.1. Material

The mixture of elementary powders, produced by Alfa Aesar and Merck, of chemical composition corresponding to chemical composition of the austenitic 316L steel, namely Fe-0.03C-1.0Si-2.0Mn-11.5Ni-2.0Mo-17.0Cr-0.11N (wt-%), have been prepared. As a source of nitrogen, a nitrided FeCr alloy has been chosen.

2.2. Processing

Mechanical alloying MA has been performed in Fritsch Vario-Planetary Mill Pulverisette 4 in argon atmosphere. The stainless steel balls have been used with the ball mass to powder ratio of 17:1. The total time of milling has been 12 hours. The milling procedure has consisted of repeating cycles of milling and breaks, both of 20 minutes long. Every second cycle, the direction of a vector of the angular velocity has been changed.

The processed powders have been compacted by cold isostatic pressing (CIP) in a room temperature. Two values of pressure have been used: 500 MPa and 900 MPa.

The hot isostatic pressing (HIP) have been performed in a specially designed steel containers, filling up with aluminum. The green compacts have been closed in welding steel dies. The temperature of HIP process have been 950^{o}C (1223 K) with a compaction pressure of 150 MPa. Because the melting point of aluminum is equal 660^{o}C (933 K), the process of pressing in a temperature of 950^{o}C (1223 K) has been performed in liquid aluminum.

2.3. Methods

The progress in mechanical alloying process has been controlled by X-ray diffraction, using Philips PW 1140 diffractometer. The application of the Rietveld method[11-13] to quantify the phase components of the powders has been used with SIROQUANT^{TM} software[14]. The morphology of the agglomerates of the powders and the microstructure of the sinters has been studied by scanning electron microscope Philips XL30 with EDAX-DX4i for microanalysis option . The electrochemical method has been used to obtain residues from the sinters. The hardness values have been obtained by Vicker's test.

3. Results

The result of 12 hours of milling of the mixture of elementary powders of chemical composition of 316L steel is shown in a diffraction pattern form in Fig. 1. There is still some amount of ferrite and molybdenum present in the powder. The Rietveld refinement in application to quantitative phase analysis indicated that the content of ferrite in the powder is at 5 wt-% level and the no alloyed molybdenum do not exceed 1 wt-%.

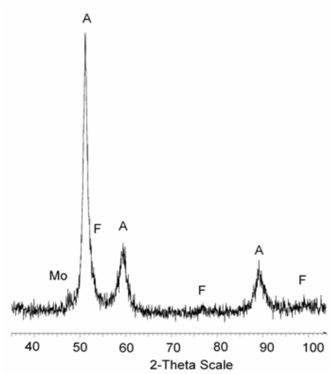

Fig. 1. The experimental diffraction pattern of a powder mixture of 316L chemical composition after 12 hours of milling. The diffraction lines are marked by letters, according to the phases which they belong to.

A austenite
F ferrite
Mo molybdenum

The green compact, processed by CIP method, is shown in Fig. 2. No lubrication or binder additions have been used for preparing the compact.

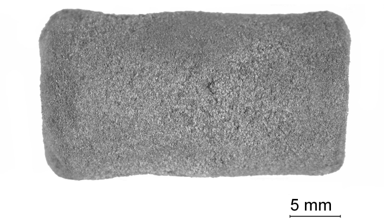

Fig. 2. The green compact of 316L steel powder obtained by CIP

One of the final compacts, after HIP process, is shown in Fig. 3.

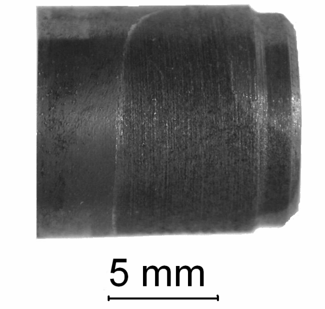

Fig. 3. The final compact of 316L steel powder obtained by HIP

Figure 4 shows the microstructure of the sinter made of pure MA 316L steel powder. The surface of the sinter is shown in polished and chemically treated conditions. The density of the sinter is 7.5 g/cm^3

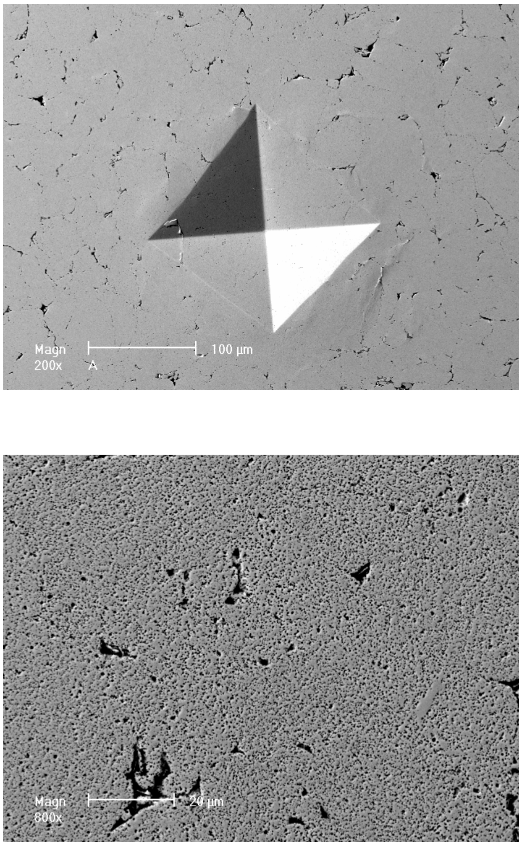

Fig. 4. The microstructure of the sinter of pure 316L powder. CIP pressure- 900MPa.The polished surface is shown above with an imprint of pyramidal indenter. Below – the electrolytically treated surface. SEM – BSE.

The second type of sinters is characterized by the presence of aluminum within the volume. The typical structure of this kind of compacts is presented in Fig. 5.

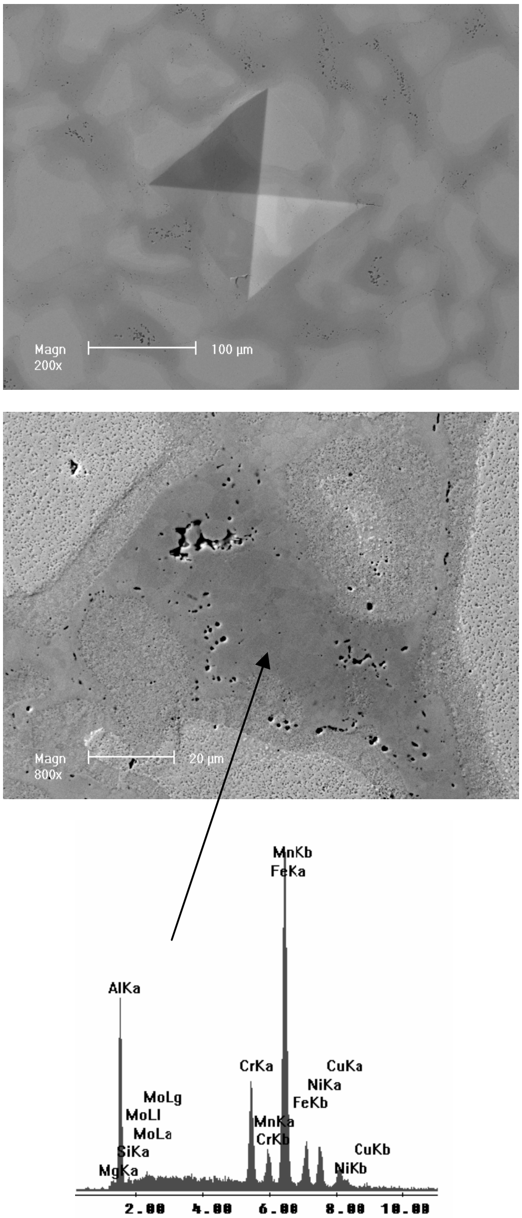

Fig. 5. The microstructure of the sinter with volume infiltration of aluminum. CIP pressure- 900MPa.The polished surface is shown above with an imprint of pyramidal indenter. Below – the electrolytically treated surface and the corresponding EDS spectrum, collecting in the point indicating by the arrow. SEM – BSE.

The density of infiltrated compacts is in the range from 6.9 to 7.3 g/cm^3, depending on the porosity mainly.

The mean values of Vickers hardness are equal 380 HV (98N) for the first kind of sinters and 430 HV (98N) for the second type.

X-ray diffraction patterns of the sinters have consisted of austenitic diffraction lines and – additionally in the second type-of FeAl lines. The small amount of some other phases (or a phase) has been revealed on the diffraction patterns. The residues have been prepared to identify these phases.

The diffraction pattern of a residue, obtained from the part of the sinter of the second type, has shown the presence of $M_{23}C_6$ as a main component, very close to $Cr_{23}C_6$ standard pattern. The small amounts of M_7C_3 has been detected and also some traces of CrN.

4. Conclusions

The mechanical alloying process has turned out to be the very effective tool to process austenitic steel powders from a mixture of α-Fe powder with some alloying elements. Unfortunately, some not alloyed amount of molybdenum has been present in the powder even after 20 hours of milling. The compressibility of the powders, especially in CIP process, has been increased by dividing them to three fractions depending on the agglomerates' sizes. The pressure used in CIP process has influenced the porosity of the final compacts strongly. In both presented above results, the pressure used in CIP process has been equal to 900 MPa. Despite of it, the porosity of the second type of compact is much smaller than in the first type. The reason is, that the infiltration of Al into the sinter structure, affected the quality of agglomerates' joints and decreased the porosity. The smaller porosity in Al-infiltrated compacts seems to be responsible for the much higher values of hardness (430 HV in comparison to 380 HV).

The HIP method has been applied in temperature of 950°C (1223 K). The time used to reach this value of temperature has been equal 4 hours which has promoted the precipitation of the carbides. The carbides are uniformly distributed within the agglomerates volume.

The method of HIP, in which not a gas but a liquid metal has been used for pressing, has proved to be a very effective tool to produce the compacts. Especially, the possibility of producing of infiltrated sinters has been possible.

As a final result of this study, a part of a fuel injection of Diesel engine has been made. The infiltrated sinter has been used as a material for it (Fig. 6).

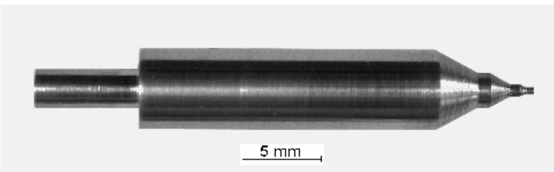

Fig. 6. A part of Diesel engine, made of the Al infiltrated sinter.

Acknowledgements

Financial support from the Polish Ministry of Science and Higher Education, project no PBZ-KBN-096/T08/2003 is gratefully acknowledged.

References

[1] G.S. Upadhyaya, "Sintered Metallic and Ceramic Materials", John Wiley&Sons, 2000.
[2] S.B. Lasday, Technology for Producing Ultrfine Metal at New Facility Expands P/M Applicability, Industrial Heating, June (1991) 17-19.
[3] B.S. Murphy, M. Mohan rao, S.Ranganathan, Milling Maps and Amorphization during Mechanical Milling, Acta metall.mater. 43(1995) 2443-2450.
[4] M. Riffel, J. Schilz, Mill Setting and Microstructural Evolution during Mechanical Alloying of Mg_2Si, J. Mater. Sc. 33 (1998) 3427-3431.
[5] J. Paduch, H. Krztoń, J. Wojtas, E. Barszcz, Study of Formation of Al-Fe Alloys by Mechanical Alloying, Proceedings of Int. Congr. X-ray Opitsc and Microanalysis, Manchester, 1992 , 303-306.
[6] G. Shi, L.X. Hu, B. Guo, E.D.Wang, Z.R. Wang, Phase and Structural Changes of $Nd_{12}Fe_{82}B_6$ alloy during Mechanical Milling in Both an Argon and Hydrogen Atmosphere, J. Mater. Process. Technol. 151 (2004) 258-262.
[7] M.M. Cisneros, H.F. Lopez, H. Mancha, D. Vasquez, E. Valdes, G. Mendoza, M. Mendez, Development of Austenitic Nanostructures in High-Nitrogen Steel Powders Processed by Mechanical Alloying, Metallurgical and Materials Transactions A, 33A (2002) 2139-2144.
[8] M. Mendez, H. Mancha, M.M. Cisneros, G. Mendoza, J.I. Escalante, H. F. Lopez, Structure of the Fe-Cr-Mn-Mo-N Alloy Processed by Mechanical Alloying, Metallurgical and Materials Transactions A, 33A (2002) 3273-3278.
[9] K.T. Kim, Y.C.Jeon, Densification Behavior off 316L Stainless Steel Powder under High Temperature, Mater. Sci. Engin. A245 (1998) 64-71.
[10] J. Song, J.C. Gelin, T.Barriere, B. Liu, Experiments and Numerical Modeling of Solid State Sintering for 316L Stainless Steel Components, J. Mater. Process. Technol. 177 (2006) 352-355.
[11] H. Krztoń, The Rietveld Method and its Applications , IMŻR eports , 3-4 (1991) 42-46 (in polish).
[12] R.J. Hill, C. J. Howard, J. Appl. Crystallogr. Vol. 20 (1987) 467-474.
[13] D.L. Bish, S.A. Howard, J. Appl. Crystallogr. Vol.21 (1988) 86-91.
[14] SIROQUANT™ Quantitative XRD Software, User's Guide and Reference, Ver. 2.5 for Windows, 2000.