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STRUCTURAL PROPERTIES OF NANO- STEEL POWDERS PREPARED BY POWDER METALLURGY

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ABSTRACT: In this paper, the first results of preparation and structural characterization of the nanostructured strengthened steels are presented. The samples were prepared by powder technology. A high energy milling process at different parameters has been applied to strengthened steel powder production. The high efficient attrition mills are on the basis of this work assuring grains with nanostructure. Powder samples were investigated by scanning electron microscopy (SEM). The structural changes during milling steps have been described. It was demonstrated that 4 hours milling in wet atmosphere are enough to realize steel powders with nano dimensions.

KEYWORDS: nanostructured strengthened steels, powder technology, scanning electron microscopy (SEM)

❖ INTRODUCTION

Oxide dispersion strengthened (ODS) FMS are promising materials with a potential to be used at elevated temperatures due to the addition of extremely thermally stable oxide particle dispersion into the austenitic or martensitic matrix. ODS steels show high-strength at high-temperatures [1]. Oxide-dispersion-strengthened steels have attracted attention for advanced nuclear power plants applications such as fast and fusion reactors, because of their superior high temperature mechanical properties [2, 3]. ODS steels are being developed and investigated for nuclear fission and fusion applications in Japan [4, 5], Europe [6, 7] and the United States [8, 9].

Powder metallurgy of stainless steel (PM SS) components constitutes an important and growing segment of the PM industry. The PM processing provide a feasible and economic manu-facturing of au-stenitic stainless steels components with complex shape and advantages such as good dimensional precision, high surface finish and good mechanical properties [10 - 14]. The production of oxide dispersion- strengthened steel involves many processes, such as mechanical alloying, degassing, canning, hot extrusion, and heat treatments. In the procedures, the hot extrusion process strongly affects precipitation behavior of oxide particles and their dispersion. [15]. Fundamental studies concerning optimization of mechanical milling (MM) processing as well as effects of alloying elements on the high-temperature mechanical strength had been carried out in cooperation with fabrication vendors [16]. In this work, the structural and morphological properties of nanostructured ODS steel powders prepared by powder metallurgy methods are presented.

❖ THE STUDY

An efficient dispersion of ODS steels will be achieved by employing a high efficient milling process, namely the attritor milling (Figure 1). In this paper the wet coating process of fine ceramic particles is proposed by the help of mechano-chemical processes assured by attrition milling. In the case of our model



Figure 1. Horizontal high efficient attritor mill

experiments, for some of the powder mixtures a high efficient attritor mill (DMQ-07 Union Process) was employed. This apparatus allowed a high rotation speed (2000-2800 rpm) and a contamination free mixing process, because of stainless steel parts (tank, arm, balls) as in Figure 1.

Based on our former observations the attritor mill has more advantages to conventional planetary mill. In the wet process, the attritor may work at higher speeds as 3000 rpm in comparison to planetary mill, 500 rpm. The delta discs employed in the attritor, as well as the small media 0.1- 1 mm assure a very efficient dispersion. The commercial austenitic powder (Hoganas 316L) were milled by attritor for 1, 2, 3, 4 and 5 hours in propanol (wet milling).

Morphology and microstructure of the powder and sintered steels were studied by scanning electron microscope (Zeiss-SMT LEO 1540 XB and Jeol JSM-25-SIII).

❖ ANALYSES, DISCUSSIONS, APPROACHES AND INTERPRETATIONS

Structural characterization of starting austenitic steel powder was performed by scanning electron microscopy (Figure 2). Austenitic sample consisted of globular particles. The average size of particles is 50 - 100 μm . The composition of starting austenitic powder is Fe and 0,02% C, 13% Ni, 16,8% Cr, 0,85% Si, 0,20%O, 0,04% N and 2,2% Mo.

The powder structure after 1 hour milling showed considerable differences to starting powder. The forms of austenitic particles are globular. Their average size is lower, about 80 μm (Figure 3a). The structural investigations demonstrated the existence of small grain in few micrometer ranges among globular grains (Figure 3b). Figure 4 showed the morphology of austenitic sample after 2 hours milling time. The average size of globular particle is about 60 μm . 1 hour intensive high efficient milling decreased the particle size about 15 - 20 μm . The shape of grains has not changed.

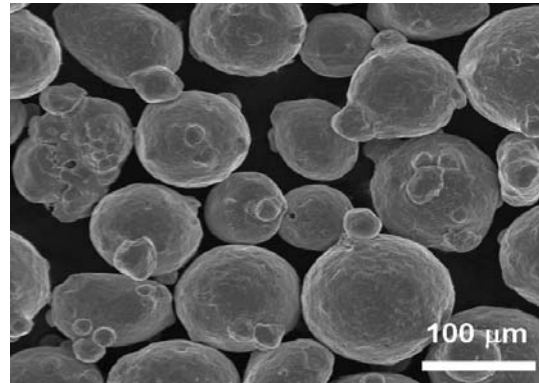
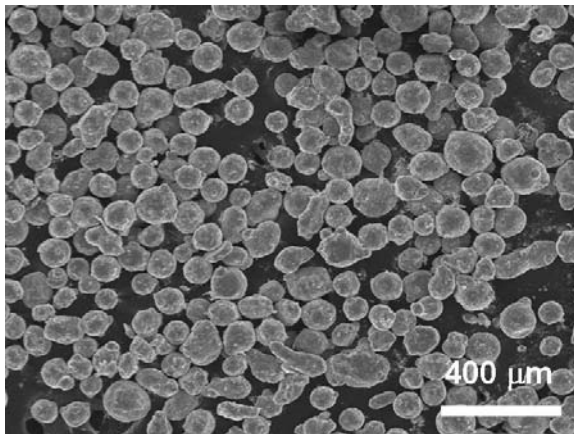
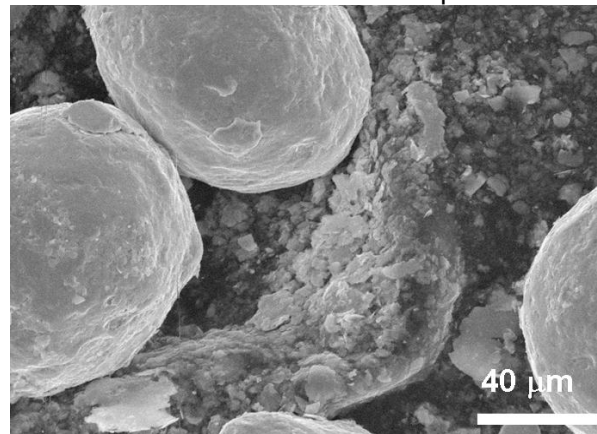


Figure 2. SEM image of starting commercial austenitic powder

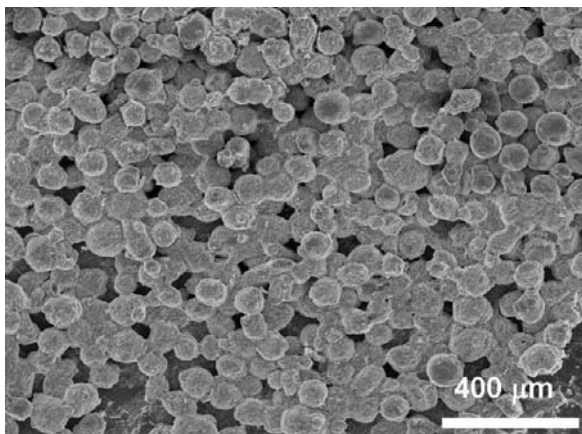


a)

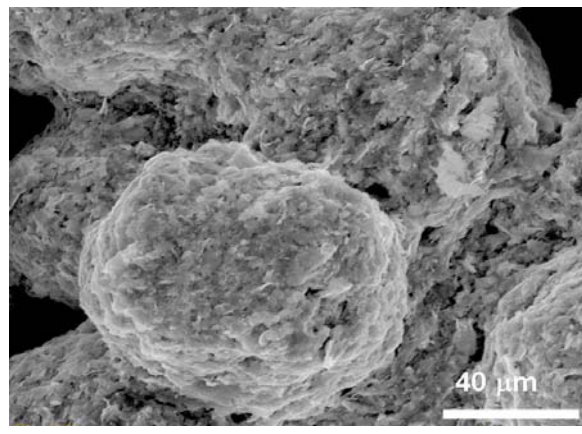


b)

Figure 3. SEM images of austenitic powder milled for 1 hour. a) low magnitude, b) high magnitude



a)



b)

Figure 4. SEM images of austenitic powder milled for 2 hour. a) low magnitude, b) high magnitude

The next 1 hour milling time (3 hours) decreased the particle size about $10\ \mu\text{m}$ as shown the Figure 5a. The parts of particles are globular, but the SEM investigation proved the developing of particles with rough surface (Figure 5b). This disintegration effect is on the basis of the evaluation of nanoparticles.

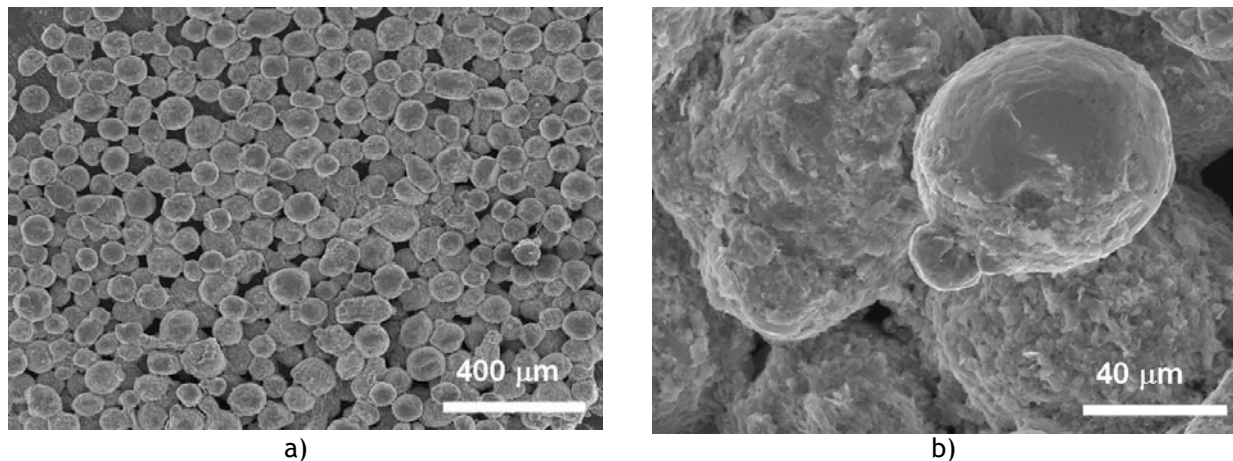


Figure 5. SEM images of austenitic powder milled for 3 hour. a) low magnitude, b) high magnitude

After 4 hours wet milling, the structure showed the drastically change in morphology. The sample consisted of very small austenitic grains with lamellar structure and of few $80\ \mu\text{m}$ size globular particles (Figure 6a). The average size of lamellar particles in one dimension is nanometer range, their length is few micrometers (Figure 6b).

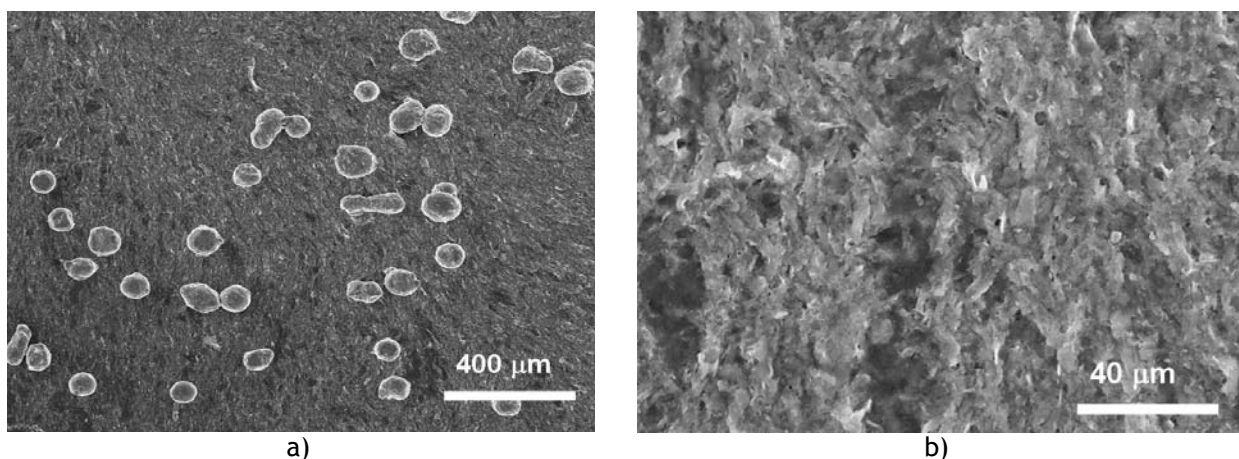


Figure 6. SEM images of austenitic powder milled for 4 hour. a) low magnitude, b) high magnitude

The final nanostructure was achieved after 5 hours milling time (Figure 7a). The sample consisted from very thin lamellar particles, the existing of larger grains is not shown. Figure 7b show the very fine austenitic structure.

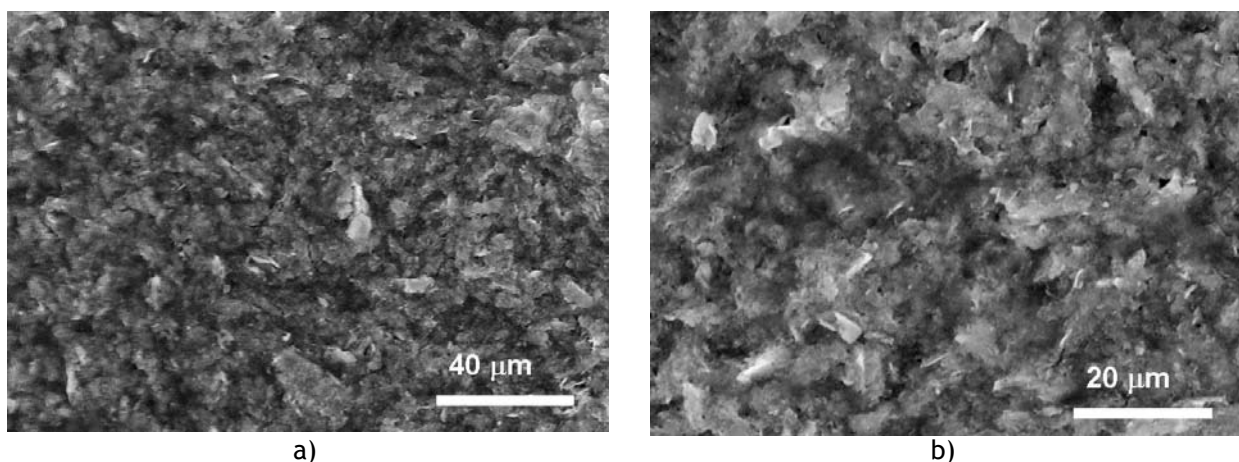


Figure 7. SEM images of austenitic powder milled for 5 hour. a) low magnitude, b) high magnitude

❖ CONCLUSIONS

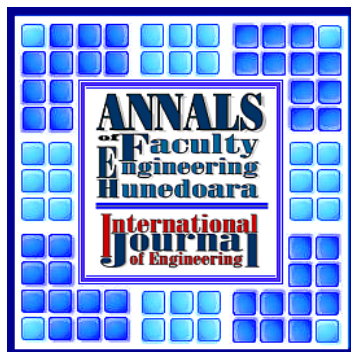
In this paper, the first results of preparation and structural characterization of the strengthened nanostructured steels prepared by the powder technology is presented. A high energy wet milling process in propanol for 1, 2, 3, 4 and 5 hours milling time has been applied to strengthened steel powder production. The structural changes have been observed. The average size of starting commercial austenitic powder was about 100 μm . The particle size of nanometer range in was achieved after 4 milling hours.

❖ ACKNOWLEDGEMENT

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❖ REFERENCES

- [1.] A. Kimura, T. Sawai, K. Shiba, et al. Nucl. Fusion 43 (2003) 1246.
- [2.] J.J. Huet, Powder Metall. 10 (1967) 208.
- [3.] J.J. Huet, V. Leroy, Nucl. Tech. 24 (November, 1974) 216.
- [4.] S. Ukai, T. Nishida, H. Okada, T. Okuda, et al., J. Nucl. Sci. Technol. 34 (1997) 256.
- [5.] S. Ukai, T. Yoshitake, S. Mizuta, et al., J. Nucl. Sci. Technol. 36 (1999) 710.
- [6.] A. Alamo, J. Decours, M. Pigoury, C. Foucher, Structural Applications of Mechanical Alloying, ASM International, Materials Park, OH, 1990.
- [7.] A. Alamo, H. Regle, G. Pons, L.L. Bechade, Mater. Sci. Forum 88–90 (1992) 183.
- [8.] D.K. Mukhopadhyay, F.H. Froes, D.S. Gelles, J. Nucl. Mater. 258–263 (1998) 1209.
- [9.] M.K. Miller, E.A. Kenik, K.F. Russell, et. al., Mater. Sci. Eng. A 353 (2003) 140
- [10.] F. Borgioli, E. Galvanetto, T. Bacci, et al., Surf. Coat. Technol. 149 (2002) 192–197.
- [11.] O. Sandberg, L. Jönson, Advances in Powder Metallurgy, Adv. Mater. Process. 12 (2003) 37–42.
- [12.] P. Lindskog, The future of ferrous PM in Europe, Powder Metall. 47 (2004) 6–9.
- [13.] Koszor O, Horváth A, Weber F, Balázsi K, Gillemot F, Horvath M, Fényi B, Balázsi Cs, KEY ENG MAT 409: pp. 237-243. (2009)
- [14.] H. Sakasegawa et al. / Journal of Alloys and Compounds 452 (2008) 2–6
- [15.] T. Okuda, S. Nomura, et al., Proc. Symp. Sponsored by the TMS Powder Metallurgy Committee, Indiana, 1989, p. 195.



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