Pulsed Electric Current Sintering of Nano-crystalline Iron-base Powders

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Abstract

A new process of pulsed electric current sintering was developed. It combines compaction with activated sintering effectively and can manufacture bulky nano-crystalline materials very quickly. A nano-structured steel is obtained with high relative density and hardness by this process. The average grain size of iron matrix is 58nm and the carbide particulate size is less than 100 nm. The densification temperature of ball-milled powders is approximately 200ºC lower than that of blended powders. When the sintering temperature increases, the density of as-sintered specimen increases but the hardness of as-sintered specimen first increases and then decreases.

Keywords : pulsed electric current sintering, nano-crystalline, powder metallurgy

1. Introduction

In the 1980s, a novel sintering technology known as Pulsed Electric Current Sintering (PECS) was developed. Plasma Activated Sintering (PAS) and Spark Plasma Sintering (SPS) are typical sintering methods of this category. Compared with conventional sintering, hot pressing and hot isostatic pressing, PECS can consolidate powders to near-full dens ity under a lower temperature and within a much shorter sintering duration [1-3], which is beneficial to the refine ment of microstructures. Furthermore, the electric discharges and plasma generated by the high pulsed currents may purify and activate the particle surfaces [4]. Therefore PECS was considered to be a potential technology. To manufacture bulky nanocrystalline materials with near-full density and clean grain boundaries. Some attempts have be en made to fabricate the nanocrystalline iron and steel b y PECS [5-6]. It is found that the grain growth is still v ery quick even in the short sintering duration of PECS. This work is aimed to fabricate a Fe-2Cu-2Ni-1Mo-1C nan ostructured steel. The nano-crystalline iron-base powders are prepared by high-energy ball-milling and consolidated by means of PECS. The sintering behavior of nano-crystalli ne powders as well as the microstructures and mechanical properties of sintered samples are studied.

2. Experimental and Results

The elemental powders are blended according to the composition of Fe-2wt.%Cu-2wt.%Ni-1wt.%Mo-1wt.%C in a V-type mixer for 5h. Then the blended powders are put into a planetary high-energy ball-milling machine and milled for 20h under the protection of argon gas. The weight ratio of milling balls to powders is 15:1. The rotating speed applied is 226 r/min.The blended and ball-milled powders are then sintered on a PECS system, as described in our previous paper [7]. Powders are loaded into an Al_2O_3 ceramic die and pressed by graphite punches. Consequently a square-wave pulsed current flows through the powders. A constant pressure level of 27.7MPa is applied throughout the sintering process.

The XRD analysis results show that the α-Fe peaks of ball milled powders broaden compared with those of the blended powders. This phenomenon is attributed to the grain refinement and lattice distortion caused by the high-energy impact and grinding. Calculation results reveal that the average grain size of iron powders is reduced from 20μm to 15.2nm after ball milling for 20h. Simultaneously the lattice distortion of iron powders increases from 0.03% to 0.48%.

The ball-milled elemental powders are consolidated to near-full dense (relative density 99.9%) at a sintering temperature of 912ºC, whereas the relative density of specimen sintered from blended powders only arrives at 98.1% even at a sintering temperature of 1123ºC.

The microstructures of samples sintered from ball-milled powders are shown in Fig. 1. When the sintering temperature is 828ºC, the porosity of sintered specimen is relatively low (relative density 96.1%). Tiny carbide particulates are dispersed evenly in the iron matrix. The grain of iron matrix is so fine that it can hardly be recognized by optical microscopy, as seen in Fig. 1a. By TEM observation, as shown in Fig. 1b, it is found that the grain size of the sintered specimen ranges from 30 to 100 nm, and the average grain size is approximately 58nm. EDAX result of this region confirms that the main composition is Fe, with a little bit Ni and Cu content in it. When the sintering temperature increases to 912ºC, although the specimen is sintered to near the theoretical density, some of the iron matrix grains grow dramatically to

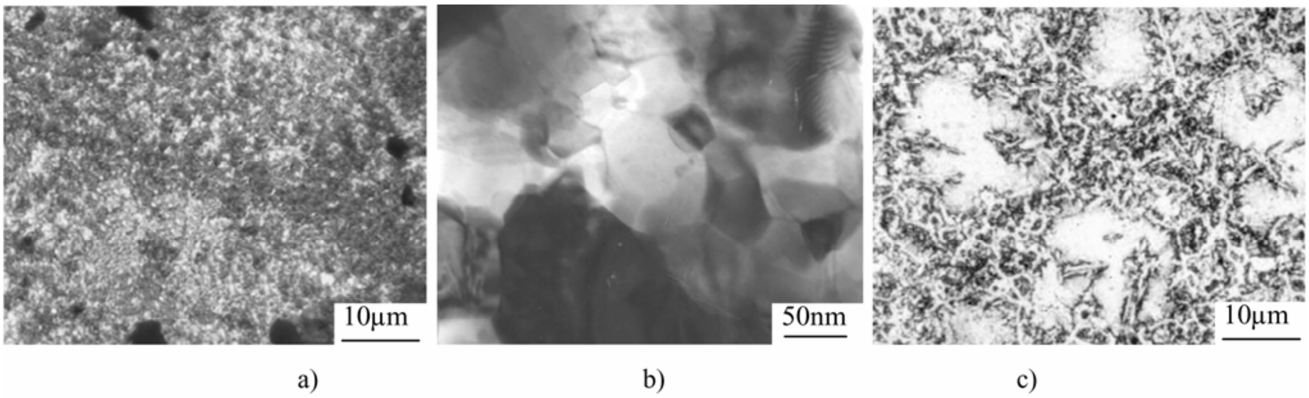

Fig. 1. Microstructures of specimens sintered from nanocrystalline iron-base powders (a), (b) Sintering tem perature 828ºC; (c) Sintering temperature 912ºC

20μm, as shown in Fig. 1c. Acicular bainite can be seen in the excessively coarsened grains.

When the sintering temperature is 828ºC, the fracture surface of sintered specimen consists of fine intercrystalline fracture facets and dimples. Some carbide particulates exist at the bottom of the dimples. The particulate size is smaller than 100nm. No obvious plastic deformation occurs before the fracture of the specimen. As the sintering temperature increases to 912ºC, the dimples on the fracture surface are deeper, and the carbide particulates are coarser than those in the specimen sintered at 828ºC. The average size of carbide particulates is approximately 100 nm.

When the sintering temperature increase, the hardness of as-sintered specimen first increases and then decreases. As the powders are subjected to an elevated temperature, the density of sintered specimen increases, the particle boundaries disappear gradually, the bonding strength between particles is enhanced, and alloying elements are diffused into the iron matrix. Therefore the hardness of sintered specimen increases gradually with the increase of sintering temperature. However, once the sintering temperature exceeds a critical level, nanograins will grow up excessively, and the hardness of the sintered specimen decreases instead.

3. Summary

In this work, nanocrystalline iron-based elemental powders with an average grain size of 15.2nm are prepared by high-energy ball-milling. The nanocrystalline powders are consolidated to near-full dense in 6 minutes by means of Pulsed Electric Current Sintering. During the final stage of densification, some of the iron matrix grains grow up quickly to about 20μm, with aciculate bainite phase dispersed in them. Hardness of the sintered specimen first increases and finally decreases due to the complex influence of densification and grain growth. A nano-crystalline sintered steel is obtained. The relative density arrives at 96.1%, the average grain size of iron matrix is 58nm, the carbide particulate size is less than 100nm, and the hardness reaches HRC 63.8.

4. References

1. J.R. Groza, A. Zavaliangos, Mater. Sci. & Eng. A, **287[2],** 171(2000).
2. M. Omori, Mater. Sci. & Eng. A, **287[2]**, 183(2000).
3. D.S. Perera, M. Tokita and S. Moricca, J. Eur. Ceram. Soc., **18[3],** 401 (1998).
4. V. Mamedov, Powder Metall., **45[3],** 322 (2002).
5. K. Satoshi, K. Hisashi, J. of the Japan Soc. of Powder and Powder Metall., **50[6],** 545 (2003).
6. R. Kalyanaraman, S. Yoo, Nano Materials, **10[11],** 1379 (1998).
7. Y.Y. Li, Y. Long, X.Q. Li, et al, Mater. Sci. Forum, **471-472[2]**, 225 (2004).