

Microstructure and Mechanical Properties of Nano ZrO₂-dispersed Fe Sintered Bodies

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Abstract

The injection molded Fe sintered bodies were fabricated using two kinds of nano Fe powders, Fe-5vol.%ZrO₂ and Fe-10vol.%ZrO₂ powders. The relationship between microstructure and mechanical properties depending on the ZrO₂ contents and sintering temperature were characterized by SEM and TEM techniques. In the wear test, the Fe-0vol.%ZrO₂ sintered bodies showed mainly adhesive wear, but in the Fe-5vol.% ZrO₂ and Fe-10vol.% ZrO₂ composites the main wear behavior showed abrasive wear mode.

Keywords: Injection molding process, Fe nano powder, Dispersion strengthened

1. Introduction

Recently, there have been many studies on the nano-particles due to their great potential applications in the research fields and high technology industries [1]. Especially, the nano-materials showed remarkably different and new material properties compared with those of the micrometer scaled crystalline materials [2]. Therefore, it needs to investigate on their sintering behavior and mechanical properties depending on the crystalline size and structure of grain boundary [3]. For example, when the fabrication of small-sized Fe components using nano-sized powder, it should be considered on the high specific surface area of raw powders as well as the sintering conditions to get the nano crystalline structure.

Among the several processes for making the Fe bulk bodies, the metal injection molding (MIM) process has some advantages such as easily making the small sized and complex components as well as its high mass and low cost production [4]. In this work, as a basic study on the ZrO₂ dispersion strengthening of nano crystalline Fe bodies, we focused on the fabrication of Fe-ZrO₂ composites using MIM process, in which the ZrO₂ contents were verified (0vol.%, 5vol.%, 10vol.%). To investigate the relationship between microstructure and material properties of Fe-ZrO₂ composites depend on the sintering temperature, SEM and TEM techniques were used.

2. Experimental and Results

Nano Fe powder was fabricated by plasma arc discharge process (PAD) process and its average particle size was about 50 nm in diameter [5]. To prevent an explosion when nano Fe powder contact with the air due to the rapid oxidation, the nano Fe powder flowed in the mixed gas (Ar+1% O₂) for 2

hours. The 5vol.% and 10vol.% m-ZrO₂ powders (Tosoh, Japan, about 80nm diameter) were added into Fe nano powder and then their mixture powders were obtained by ball mill process. As the polymers binders, the ethylene vinyl acetate (EVA210, DuPont), paraffin wax (PW, DNW-135P, Dongnam Chem.) and stearic acid (SA, CH₃(CH₂)₁₆COOH, Daejung Chem. & Metals) were used. The composition ratio of polymer binders was fixed with 50 (EVA): 45 (PW): 5 (SA). The volume percentage of Fe-(m-ZrO₂) composite powder and polymer binder was 50:50.

To make the Fe-ZrO₂ powders / binder mixture, the shear mixing was carried out at following conditions; 100°C, 60 rpm, 20 minutes and in Ar atmosphere. The disk type brown bodies were made using these mixtures and in this time, the injection molding was carried out at 60°C and 130 MPa pressure. To remove the polymer binders, the debinding process was carried out at 530°C for 30 minutes in a flowing hydrogen atmosphere and then the sintered bodies were obtained at 700°C, 800°C, 900°C (duration time : 1hour, heating rate : 5°C/min). The values of relative density and hardness of brown and sintered Fe-ZrO₂ bodies were measured using Archimedes principle and Vickers hardness (HV-112, Akashi, Japan), respectively.

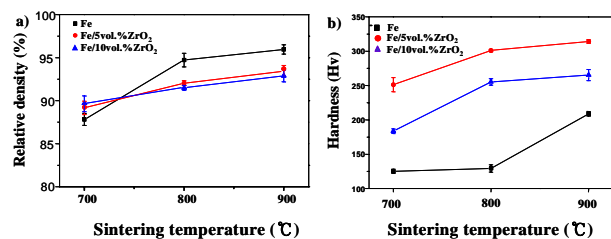


Fig. 1. a) Relative density, b) Hardness of Fe-ZrO₂ sintered bodies depending on the sintering temperatures and ZrO₂ contents.

Fig. 1 shows the values of relative density (a) and hardness (b) of Fe-ZrO₂ sintered bodies depending on ZrO₂ addition contents and sintering temperature. The relative density increased as increasing the sintering temperature. In the samples containing 0, 5 and 10vol% ZrO₂ (at 700°C), the relative densities were 87.8%, 88.9% and 89.5%, respectively. However, as increasing the sintering temperature (800°C and 900°C), the relative densities of Fe-0%ZrO₂ composite remarkably increased while in the Fe-5vol% and Fe-10vol% ZrO₂ composites, these values increased slowly as increasing the ZrO₂ contents. The main reason for showing the low relative density is basically due to the dispersion of nano ZrO₂ particles which are acted as an obstacle for the full densification.

The hardness values also increased as increasing the sintering temperature, but the maximum value (316.7 Hv) of hardness was obtained in the Fe-5vol% ZrO₂ composite sintered at 900°C. The low hardness values in the Fe-10%ZrO₂ composite are mainly due to the existence of many residual pores in the sintered body.

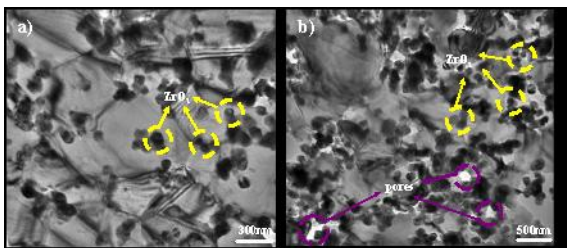


Fig. 2. TEM images of Fe-ZrO₂ sintered bodies at 900°C
a) Fe-5vol.% ZrO₂ and b) Fe-10vol.%ZrO₂

Fig. 2 shows TEM images of Fe-5vol.%ZrO₂ (a) and Fe-10vol.%ZrO₂ (b) bodies sintered at 900°C. The ZrO₂ powders were homogeneously dispersed at grain boundaries and in Fe grains. Especially, as increasing the ZrO₂ contents in the Fe matrix, some agglomeration of ZrO₂ particles and residual pores were observed. The grain size of Fe matrix was about 300~500nm in diameter and some amounts of dislocations were also observed as internal defects. The homogeneous dispersion of ZrO₂ fine particles in the Fe composites can be acted as the pinning sites for the dispersion strengthening of nano crystalline Fe sintered body.

Fig. 3 is SEM micrographs showing the wear surfaces of Fe-ZrO₂ composites depending on the ZrO₂ contents. The wear test was carried out using the ball on disk type as following conditions; 1Kg load, 60 rpm speed and for 2hours. As a counter ball, the steel ball with 2 mm in diameter was used. In Fig. 3 a), b) and c), it is confirmed that the wear widths decreased as increasing the ZrO₂ contents; i.e., in the Fe-0vol%-, 5vol%- and 10vol%- ZrO₂ composites, their widths were 440 μm, 200 μm, and 180 μm,

respectively. Fig. 3 d), e) and f) shows high magnification images of wear surfaces in the Fe-0vol%, 5vol%- and 10vol%-ZrO₂ composites, respectively. In the Fe-0vol%ZrO₂ composite (d), the main wear mechanism was adhesive wear mode, but in the Fe-5vol.%ZrO₂ (e) and Fe-10 vol.%ZrO₂ (f), the abrasive wear was mainly occurred due to the dispersion of hard ZrO₂ nano particles.

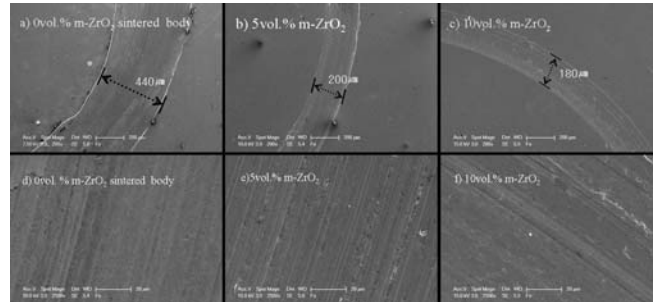


Fig. 3. Wear tested surfaces SEM images of Fe sintered bodies depending on the ZrO₂ content (a, b, c) low magnification and (d, e, f) high magnification.

3. Summary

In this work, the relationship between microstructure and mechanical properties of Fe-ZrO₂ composites fabricated by MIM process was investigated depending on sintering temperature and ZrO₂ content. Nano ZrO₂ particles were homogeneously dispersed at grain boundary and in the Fe grains. As the internal defects, some amounts of dislocations were observed in the Fe matrix and especially in the Fe-10vol%ZrO₂ composite, some amounts of residual pores were found. The relative density and hardness increased remarkably as increasing the sintering temperature. The maximum value of hardness was obtained in the Fe-5vol%ZrO₂ composite sintered at 900C, while the highest wear resistance was obtained in the Fe-10vol%ZrO₂ composite due to the dispersion of hard ZrO₂ particles

4. References

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