



## Short Communication

# Formation process of the {001} fiber texture on iron particles using simple ball milling



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## ARTICLE INFO

## Article history:

Received 26 April 2017

Received in revised form 24 July 2017

Accepted 6 August 2017

Available online 08 August 2017

## Keywords:

Ball milling

Fine iron particle

Texture

Electron backscatter diffraction (EBSD)

X-ray diffraction (XRD)

## ABSTRACT

The process of texture formation on iron particles was investigated via simple ball milling using graphite particles as milling aid. During milling, the shape of the iron particles changed from granular to platelets owing to repetitive compression and balls rolling in a direction perpendicular to the platelet faces. Simultaneously, a {001} + {111} double-fiber texture was formed, which is characteristic of a metal with a bcc structure deformed under uniaxial compression and multidirectional rolling. Based on these observations, it is concluded that the texture obtained through simple ball milling was induced by uniaxial compression and a multidirectional-rolling-like deformation. Since the main component of the double-fiber texture is {001} and the easy magnetization axes {001} are oriented toward the in-plane directions with the platelet face in the texture, the iron particles are ideal candidates for use as magnetic cores in motors, inductors, and other electromagnetic parts.

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## 1. Introduction

A laminated core comprising electrical steel sheets is commonly used in generators, alternators, and electric motors [1]. The ideal texture for these sheets is {100}⟨0vw⟩ because its plane normal direction is parallel to the easiest magnetization ⟨001⟩ axes [2] and fulfills the requirement for rotating magnetization. Dust cores, which are fabricated by compacting insulated soft magnetic powder, have an advantage over laminated cores as they can suppress eddy current losses. This is because powder can be insulated in a three-dimensional manner to increase the electric resistance, which is not the case with electrical steel sheets [3]. However, hysteresis losses in a dust core are larger than those in a laminated one because the easy magnetization axes in a powder are not controlled. Therefore, to reduce these losses, particles in the easy magnetization axes that can be controlled are desirable for dust cores.

The ball milling technique is widely used in many fields, such as metallurgy, chemistry, and other fields related to material science. As the behaviors of contents and phenomena in a milling vessel are very complicated, they have been theoretically [4] and practically [5] analyzed by many researchers. With advancements in the understanding of the behavior and structure of the resultant material from ball

milling, researchers have begun to use milling techniques to not only break down materials but also to achieve more favorable structures such as composite structures [6], nanostructures [7], and amorphous structures [8].

Recently, we proposed a process that uses a conventional ball mill with the addition of graphite particles to obtain iron particles with a platelet shape and a texture that parallels the easy magnetization axes of the platelet faces [9]. However, the formation mechanism of this texture when using the milling process is not fully understood. In this study, various microstructures and crystal structures were investigated to confirm the texture-formation process and discuss its mechanism. It was found that the texture obtained via simple ball milling was induced by uniaxial compression and a multidirectional-rolling-like deformation.

## 2. Experimental section

Commercial iron particles (average diameter: approximately 70 μm, purity: >99%) and graphite (Gp) microparticles (average diameter: approximately 10 μm, purity: >99%) were processed using ball milling. The iron particles having 2 g in weight and Gp particles having 0.014 g in weight were placed in a stainless steel vessel. Steel balls (AISI 52100, diameter: 9.52 mm) were also added as the milling medium. The vessel was mounted on a ball mill and then swung at 59.1 rad/s for 1.0 h.

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The morphology and crystallographic texture of the resultant particles were characterized using scanning electron microscopy (SEM), electron backscattered diffraction (EBSD), and X-ray diffraction (XRD). The EBSD data were processed using the TSL 6 OIM analysis software (TSL Co., Ltd.). Subsequently, inverse pole figure (IPF) maps were obtained. Prior to the SEM/EBSD observations, the particles were embedded in Technovit 5000 conductive resin (Kulzer Co., Ltd.) and mechanically polished using emery papers and a diamond paste. Finally, to remove any residual near-surface deformations, Ar ion milling was performed using an ion milling system (IM4000, Hitachi Co., Ltd.). This was a necessary surface preparation for the EBSD scans. To observe cross-sections of the resultant platelet particles, the particles were glued with their surfaces parallel to a stainless steel plate. This plate was also embedded in conductive resin, and its cross-section was polished. The cross-sections of the resultant particles were then obtained. Based on a logarithmic strain used for evaluating the amount of plastic strain on the compressed rod, the amount of plastic deformation of the milled particle was evaluated as follows:

$$\varepsilon = \ln(T/D_i) \quad (1)$$

where  $T$  is thickness of the platelet particles and  $D_i$  is average diameter of the iron particles. XRD patterns were recorded using a Rigaku Smart Lab X-ray diffractometer (Rigaku, Co. Ltd.) equipped with a Cu- $K\alpha$  radiation source (1.542 Å). Pole figure measurements were performed using an in-plane pole figure measurement method. This method allowed a complete pole figure to be recorded from  $\alpha = 0^\circ$ – $90^\circ$  (where  $\alpha$  is the tilt angle from the sample-surface normal direction) and did not require the sample to be tilted. The details of this measurement can be found in the literature [10]. Based on the pole figure measurements of {011}, {002}, and {211}, the orientation distribution function (ODF) was calculated using the Dahms and Bunge method [11]. The main components of the obtained textures were evaluated by examining the IPF derived from the ODF.

### 3. Results and discussion

Fig. 1 shows representative SEM images of the appearance ((a-1) to (a-4)) and cross-section ((b-1) to (b-4)) of the milled iron particles as a function of milling time. As shown in Figs. 1 (a-1) and (b-1), the iron particles at 0 h exhibited a spherical shape. The shape of the particles changed from spherical to platelet. This indicates that the iron particles

deformed plastically. Furthermore, iron particles deformed more severely with increasing milling time, as shown in Figs. 1 (a-1) to (a-4). The thickness of a platelet particle (shown in Fig. 1 (a-4)) is approximately 3  $\mu\text{m}$ , and the initial average diameter of the iron particle is approximately 70  $\mu\text{m}$ . Therefore, the logarithmic strain defined in Eq. 1 of the platelet particle is  $-3.15$ . At a later milling stage, the deformed platelet particles piled up on each other and displayed a lamellar structure, as shown in Figs. 1 (b-3) and (b-4). The interface of the piled particles was in a linear arrangement. As far as could be observed, there was no lamellar structure that was folded back on itself, indicating that the platelet iron particles were repeatedly subjected to forces originating from the milling medium in a direction normal to the platelet plane.

Fig. 2 shows representative SEM and corresponding IPF maps of the milled iron particles along the direction normal to the platelet plane and along an in-plane direction. As shown in Figs. 2 (b-1) and (c-1), the iron particles before milling were polycrystalline, with a crystalline size of the order of several tens of micrometers; moreover, no texture was observed. The relative intensity of the diffraction peaks in the XRD pattern appears similar to that of polycrystalline  $\alpha$  iron mentioned on Joint Committee on Powder Diffraction Standards card no. 01-071-3763, as we previously reported [9]. Therefore, the EBSD and XRD observations indicate that the iron particles had no preferred orientation prior to milling. Figs. 2 (b-1) to (b-4) show IPF maps along a direction normal to the platelet face. Regions in which the {001} planes (red region) and {111} planes (blue region) dominate expand with increasing milling time. At 1.0 h (Fig. 2 (b-4)), almost all of the area on the particle surfaces is dominated by {001} or {111}, and the {001} region is larger than the {111} region. Figs. 2 (c-1) to (c-4) shows IPF maps along a direction that is in-plane with the platelet face. Traces of several crystallographic planes are observed on the IPF map. This means that no strong preferred orientation along the in-plane direction was developed. These EBSD observations indicate that the milling treatment results in the formation of the {001} + {111} double-fiber texture on the platelet iron particles and that the texture developed with increasing milling time. As can be seen in Fig. 1, the total amount of plastic deformation of the particles during milling increases with milling time. Therefore, the resulting texture is a type of deformation texture.

Fig. 3 is a set of IPF maps derived from the ODF at directions normal and parallel to the glass holder for XRD measurements. Particle orientation occurred in the holder during the XRD measurement because of the platelet shapes of the particles and the platelet faces being parallel to

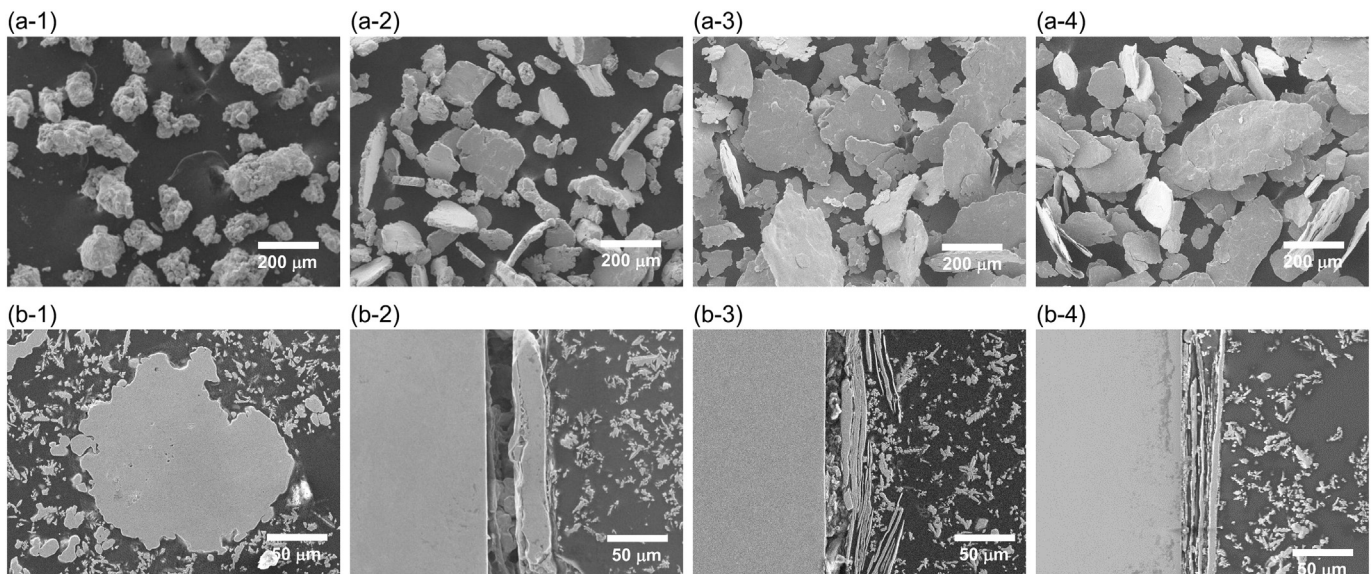


Fig. 1. Representative SEM images of iron particles milled for 0 h (a-1), 0.10 h (a-2), 0.50 h (a-3), and 1.00 h (a-4) and their cross-sections (b-1) to (b-4).

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