Research on Grain Detection of Grain-Oriented Silicon Steel Based on Two-Dimensional X-Ray Diffraction

Li Xiao-ning^{1,2}, He Fei^{1,*} and Xu Ke¹

¹Collaborative Innovation Center of Steel Technology, University of Science and Technology Beijing, Beijing 100083, China

²Institute of Engineering Technology, University of Science and Technology Beijing, Beijing 100083, China

In order to improve the electromagnetic properties of transformer cores, grain size and boundaries detections of grain-oriented silicon steel are necessary. Conventional electron microscopic grain size detection is off-line and destructive, which cannot meet the requirements of modern production. X-ray detection is non-destructive and the sample can be dynamic. In this paper a new method of the grain boundaries detection of the Hi-B grain-oriented silicon steel with large grains has been proposed based on the change of images capturing by the two-dimensional X-ray diffraction system. The grain boundaries map is calculated with diffraction information which is extracted from the images based on a series of image processing algorithms. The results are basically consistent with the electron microscopy. Compared with the traditional method, the dynamic and non-destructive detection method is able to increase the detection efficiency and improve the overgeneralization of sampling. Compared with X-ray diffraction contrast tomography, this method is more efficient and low-cost. The sample can also be used after the detection, especially in high-end applications. When the grain boundaries are determined, the necessary processing method can be adopted to further enhance its electromagnetic performance in subsequent processing. [doi:10.2320/matertrans.M2017252]

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1. Introduction

Grain-oriented silicon steel is the most important soft magnetic material used as core material of transformers¹⁾. The material is manufactured through many complicated steps, leading to such electrical steel product being honored as an 'artistic product of iron'^{2,3)}. The commercially important properties are the high permeability and low core loss at a high electromagnetic induction. The core loss consists of hysteresis loss and eddy-current loss^{6,7)}. Its magnetic properties are closely related to the sharpness of the secondary recrystallization texture, especially $\{110\}<001>$ (Goss texture)^{4,5)}. The Hi-B (high-permeability grade) is an important product with sharp Goss texture accounting for more than 85% of the volume, which makes it as a great magnetic material⁸⁾. Usually the sharpness can be detected by using stacks of slices⁹⁾. So in this paper, the aim is not the texture sharpness but the difference between adjacent grains.

In order to reduce the core loss, two ways had been applied in processing previously. One is improving $\{110\}<001>$ alignment and the other is reducing the thickness of silicon steel sheet. But these ways are more and more difficult with the development of production technology. Adjusting the magnetic domain structure can also achieve the purpose of reducing core loss. The grain size in the product of Hi-B is so large (10~30 mm), resulting in large magnetic domains, which give rise to a large eddy-current loss as the domain walls move back and forth under the action of alternating field¹⁰⁾. In recent years, it has been studied extensively to decrease large eddy-current loss, and a number of techniques have been developed. Laser or other mechanical methods can refine of the magnetic domain effectively, if the grain boundaries can be detected and determined¹¹⁻¹³⁾.

There have been some methods for detecting grain size

and boundaries, such as metallurgical microscope, electron backscatter diffraction (EBSD)¹⁴⁾, Transmission electron microscope¹⁵⁾, scanning electron microscope (SEM)¹⁶⁾, laser-ultrasonic measurement¹⁷⁾. But these methods have many disadvantages. Some of the traditional methods are destructive because the sample need to be polished before testing. Others can only obtain the average grain size without grain boundaries.

X-ray diffraction has been widely used in texture analysis¹⁸⁾. Poly¹⁹⁾ or monochromatic²⁰⁾ synchrotron X-ray sources and polychromatic laboratory X-ray sources are applied to different methods of detection. In the 1990s, the two-dimensional X-ray diffraction detector diffraction systems was designed and now have been used in texture and residual stress detection^{21,22)}. Pole figure can be calculated from the data of X-ray diffraction. X-ray diffraction contrast tomography (DCT) has been used to map the grains orientations in 3D²³⁾. These methods are accurate enough and increasing applied in the field of material testing and characterization, but it is difficult to apply in the field of production monitoring because of the cost and the complexity. Compared with electron backscatter diffraction and metallographic methods, X-ray is non-destructive for the sample and efficient. Compared with one-dimensional X-ray diffraction, two-dimensional X-ray diffraction has the characteristics of high-speed and large data volume, which can improve the detection speed and be expected to be used for online detection²⁴⁾. The average grain misorientation of Hi-B oriented silicon steel is about 3 degrees¹³⁾, therefore orientations of different grains lead to a change of the diffraction images. In this paper, two-dimensional X-ray diffraction system was built to obtain the images. The different images depending on the misorientation can be used to calculate and identify the different grains. The grain map containing the information of grain size and grain boundaries can be produced with image processing methods. The reliability of the detection method is verified by metallographic results.

*Corresponding author. E-mail: hefei@ustb.edu.cn

Meanwhile, the grain boundaries detection of grain-oriented silicon steel can also provide a reliable method for non-destructive detection of other materials.

2. Principle of the Detection

2.1 Principle of Two-dimensional X-ray Diffraction

The principle of two-dimensional X-ray diffraction^{25,26)} is shown in Fig. 1. According to the Bragg's Law:

$$2d \sin \theta = n\lambda \quad (1)$$

Where λ is the wavelength, d is the distance between each adjacent crystal planes (d -spacing), θ is the Bragg angle at which one observes a diffraction peak, and n is an integer number, called the order of reflection.

When polycrystalline material is irradiated with monochromatic X-ray, the diffraction pattern is produced as a series of cones with different θ . As shown in Fig. 1(a), different cones can be calculated to get different pole figures. With the change of the χ and ϕ , the pole figure as shown in Fig. 1(b) can be efficiently detected by two-dimensional detector.

Hi-B grain-oriented silicon steel is polycrystalline material with Goss texture, so in order to obtain the grain size and boundaries. If the diameter of X-ray is small enough, the result of the diffraction received by detector is usually one or several spots.

2.2 Diffraction System

Grain boundaries dynamic detection system based on two-dimensional X-ray diffraction is shown in Fig. 2. The equipment is D8 discover from BRUKER with the detector vantec500, and the software is DIFFRAC.SUITE. But the mechanical structure and the platform was redesigned²⁷⁾. The m in Fig. 2(a) is related to different material and calcu-

Fig. 1 Schematic of two-dimensional data collected by the area detector (a) X-ray diffraction diagram (b) Detection angle of X-ray corresponding to the texture position.

Fig. 2 Mechanical structure of online detection system for micro structure of material 1—Half circle stent; 2—Mobile bar; 3—Rail; 4—X-ray detector; 5—X-ray sources; 6—Rotation axis; 7—Sample platform; 8—Lifting appliance; 9—X-ray.

lated from θ in Bragg's Law. It determines the different facet diffraction and the type of poles. Depending on the Co (cobalt) anode target and lattice parameter a of grain-oriented silicon steel, 2θ is determined as 77.6 degrees and m is 102.4 degrees. The χ is determined by adjusting the q in Fig. 2(b) and ϕ is determined by the angle between the X-ray generator and the detector. The position of Goss texture calculated by using the MTEX open-source package²⁸⁾ is shown in Fig. 3, so the χ is selected as 45 degrees and ϕ is 90 degrees. When the rolling direction is in accordance with y axis in Fig. 2(a), q is set as 45 degrees, and then Goss texture can be detected. The component numbered as 7 in Fig. 2 is an electrically controlled rail platform that can control movement steps in x and y directions precisely. The grain misorientation of grain-oriented silicon steel sheet in different position can be reflected by the detection on the two-dimensional plane. The lifting device numbered as 8 makes it possible to detect the samples with different thicknesses effectively.

2.3 Principle of Grain Boundaries Detection

X-ray is generated and goes through the collimator. And then the sample is irradiated with the X-ray. The diffraction information can be received by the two-dimensional detector as shown in Fig. 4. The sample platform moves along the x and y axes, and diffraction measurement is performed at each step. Due to the tiny misorientation, the change of diffraction spot position indicates that there are different grains.

Fig. 3 Position of the Goss texture on pole figure from standard polar figure.

Fig. 4 Diagrammatic sketch of different grain diffraction under X-ray.

The working voltage of X-ray diffraction system is 35 kV, and the current is 40 mA. The diameter of collimator is 1 mm. Because the sample is irradiated with X-ray obliquely, the sample area involved in the diffraction is elliptical as shown in Fig. 5. The angle A_h between X-ray and the horizontal plane is calculated with θ and q :

$$A_h = \arcsin \left[\sin \theta \times \sin \left(\frac{\pi}{2} - q \right) \right] \quad (2)$$

Therefore, the angle between y axes and the long axis of an ellipse is:

$$A_y = \arctan \left[\tan \theta \times \cos \left(\frac{\pi}{2} - q \right) \right] \quad (3)$$

So, the A_h is 26.30 degrees and the A_y is 29.62 degrees. The long axis of ellipse is 2.26 mm. The long axis direction is not parallel to the direction of x or y direction, and 2.26 mm is decomposed into x and y direction which are 1.12 mm and 1.96 mm. Therefore, the moving step of the electrically controlled rail platform in the direction of x and y axes is chosen to be 1 mm, which means a diffraction image is acquired at an interval of 1 mm. That can ensure the most area can be detected by the X-ray system.

The misorientation is complicated, but the process can be simplified. X-ray is irradiated on grain A, after the sample moving it will be grain B as shown in Fig. 5. Because of tiny misorientation, the angle between crystal planes of the different grains lead to different diffraction spots. The spots in images received by detector have different positions and intensities which can be calculated to map the grains. The diameter of detector is 140 mm, and the distance l_d between the sample surface and the detector is 200 mm as shown in Fig. 6. When the angle between two crystal planes is t degrees:

$$t = \theta_{i1} - \theta_{i2} \quad (4)$$

Where the incident X-ray angle θ_{i1} and the diffraction X-ray

Fig. 5 Step selection principle according to the shape of X-ray on sample.

Fig. 6 Range of the detector covering the angle of X-ray diffraction.

angle θ_{d1} are satisfied with $\theta_{d1} = 2\theta_{i1}$. So the angle of diffraction X-ray of two grains is:

$$\theta_{d1} - \theta_{d2} = 2t \quad (5)$$

Then the distance l_s of the diffraction spots is approximately:

$$l_s \approx l_d \sin(2t) \quad (6)$$

When $t = 3$ degrees, the angle of diffraction X-ray is 6 degrees, then $l_s = 20.91$ mm. The m and q ensured that the detector is centered on the texture, and according to the diameter of detector and the distance l_d , the angle between the diffraction X-ray covered by the center and edge of detector is t_{max} as 19.29 degrees. It means when the angle of the crystal plane deviating from $\{110\}<001>$ direction is less than half of 19.29 degrees, the diffraction spot can be detected. The resolution of the detector is 2048×2048 . In order to obtain a good quality image, the diffraction time is set to 3 seconds at each step and then detection sequence is preserved. Because of the tiny angle of the crystal planes, the received images of different grains are different as shown in Fig. 7.

The diffraction spot position and intensity are obtained after the preprocessing and feature extraction. Each diffraction spot indicates one grain under the incident X-ray. A plurality of spots in the diffraction image means that there are a plurality of grains participating in the diffraction, and the diffraction area is divided according to the diffraction intensity. When there is no diffraction spot in the image, it means that the grain involved in the diffraction is non-Goss orientation.

The program of entire processing is designed as shown in Fig. 8, specifically including the following steps:

- (1) Read the image and preprocess: Preprocess the image by binarization, then filter the noise by median filtering which selected template is square (8×8 pixel). To ensure that the diffraction spot is a single connected region, the morphological closed operation is performed. The closed operation is on the basis of morphological image processing²⁹⁾, where dilation ($\oplus$) and erosion ($\ominus$) are applied in order:

$$X \cdot S = (X \oplus S) \ominus S \quad (7)$$

Where X is original image, S is the structuring element. The center point of the diffraction spot in the figure represents the position of the current diffraction spot, and the intensity information in the diffraction spot area is

Fig. 7 The actual diffraction results of grain-oriented silicon steel of different grains.

calculated from the gray level image.

- (2) Image feature extraction: As shown in Fig. 9, the position and intensity of spots are extracted, and the position information indicates whether the orientation of the diffraction grains changes. The intensity information quantifies the division of the unit diffraction area in the detection result when there are multiple diffraction spots in the diffraction image.
- (3) Judge the quantity of diffraction spots: The number of spots indicates the quantity of the grains of current diffraction. Then every spot needs to be compared with the spots in surrounding images.
- (4) Judge the change of diffraction spot: According to the different positions of the current diffraction spot and the other known diffraction spots around, the pixel distance exceeds a threshold (100 pixel selected in this paper), which indicates that the diffraction grain has changed.
- (5) Generate grain map: According to the above process and the results of each diffraction, the grain map of the current sample is given, and different grains are marked with different colors.

3. Experiment and Analysis

3.1 Experiment

The samples used in this experiment were 0.3Q21G Hi-B

Fig. 8 The processing program structure of grain boundaries detection.

Fig. 9 Specific preprocessing and feature extraction in program.

grain-oriented silicon steel sheet. Detection was finished with the two-dimensional X-ray diffraction system, and the system parameters was introduced in section 2. The experiment was conducted offline and the data were processed by the program after all the images were collected.

In the metallographic experiment, the samples were polished by 2000# sandpaper until there was no scratch on the surface. The steel sheet is 0.3 mm thick and inorganic coating is about 3 μm thick. The coating should be polished off to make the erosion easily. After the sample was soaked with 4% hydrochloric acid alcohol solution for thirty minutes, the grain and grain boundaries were obviously observed.

3.2 Experimental Results

A lot of samples were detected by two-dimensional X-ray diffraction system. There are the similar results. Three typical samples (a), (b), (c) are showed here. After processing, the grain boundaries results are obtained in Fig. 10. Different colors represent different grains, but the orientations of the grains are not related to any color. And the black indicates that the diffraction spots are not detected in the diffraction images because the orientations of some grains are not Goss texture. The grain size with Goss is about 20~30 mm. Meanwhile, the grain boundaries are approximately in agreement with the metallographic results showed in Fig. 11.

3.3 Analysis and Discussion

- (1) In the experiments, some diffraction information of the other orientation cannot be accurately detected due to the position limitation of the detector, but the impact on the detection results is limited which benefits from the sharpness of Goss texture. The diameter of the collimator, the moving step of the sample platform and the diffraction time also have some influences on the experimental results. Therefore, it is necessary to optimize the parameter selection of the relevant conditions comprehensively in the future.
- (2) In Fig. 11, some of the boundaries have not been de-

Fig. 10 Grain boundaries processing results (Different colors only indicate different grains, not the orientations, and the black area indicate that there is no spot in experimental images.).

tected. In the results of diffraction images, there may be some overlapping diffraction spots that cannot be differentiated, probably because the orientation of several grains is so similar, or some small spots are covered by big spot. The results should be expressed clearly by using higher precision equipment and advanced image

Fig. 11 Metallographic of oriented silicon steel (50 × 20 mm) (The lines are grain boundaries from Fig. 10.).

processing algorithm.

- (3) In the experiment, the accuracy of 1 mm is not sufficient for the accurate detection of grain boundaries where further improvement is required. And each step the diffraction area on the surface of the sample is overlapped with another step, so the shape of the collimator also can to be adjusted in the future.

4. Conclusions

- (1) Based on the principle of two-dimensional X-ray diffraction, a non-destructive method for detecting grain size and boundaries of Hi-B grain-oriented silicon steel has been proposed, and dynamic data collection system built in the experiment has satisfied with the detection. The results has been corroborated by traditional metallographic method. The dynamic non-destructive detection can be used in the production and processing of high-end products.
- (2) In the method, the moving step precision of the device is 1 mm, and if the grain boundaries are required to be detected with high precision, it can be realized by adjusting the moving step precision, the diameter of the X-ray beam and the time of diffraction synthetically. So in further research, the misorientation can also be detected accurately. Secondly, it is necessary to increase the detector number and ensure that more diffraction information can be obtained. Finally, more refined and efficient image processing algorithm is required for dealing with the overlap of diffraction spots, which can improve the accuracy of detection, and achieve the goal of high precision online detection.
- (3) There are already many advanced equipments can map the grains in 2D or 3D. But the cost and complexity make it difficult to be applied on-line. So the method in

this paper is a good choice for enterprises.

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