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Short communication

Misorientation/local plastic strain manifestations in chemical etching color

Xianguang Zhang[a,*], Kiyotaka Matsuura[b], Munekazu Ohno[b]

[a] Graduate School of Engineering, Hokkaido University, Kita 13, Nishi 8, Kita-ku, Sapporo, Hokkaido 060-8628, Japan
[b] Division of Materials Science and Engineering, Faculty of Engineering, Hokkaido University, Kita 13, Nishi 8, Kita-ku, Sapporo, Hokkaido 060-8628, Japan

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ABSTRACT

Cold plastic deformation produces misorientations inside the crystal grains, and the distribution of the misorientation is quite crucial to understand the deformation behavior of the metals or alloys. The misorientation manifestations in chemical etching contrast are investigated in this study in the case of cold-deformed iron. The chemical etching is performed by using nital, while the crystal orientation is determined by electron backscatter diffraction (EBSD). The correlation between the chemical etching contrast and crystal orientation have been studied in both cold-deformed and undeformed iron. The results clearly show that the chemical etching contrast strongly reflects the crystallographic orientation. The gradual change in chemical etching contrast inside the individual deformed grains gives information of both the misorientation and local plastic strain within the grains. This method can provide an easy and alternative way to qualitatively understand the misorientation and local plastic strain distributions in the microstructures.

1. Introduction

Microstructures including crystal grains and constituent phases are made visible by etching during a metallographic investigation. Two main types of etching are applied: the classical chemical etching and color etching. The chemical etching using an etchant is a very simple technique and is most widely used in the metallographic investigation, which preferentially attack the crystal grain boundaries to reveal the microstructures. On the other hand, the so-called color etching is carried out by deposition of a thin surface film on the metal, causing light interference between the different reflections from the metal surface and the film surface (Vander Voort, 1984), which is relatively complicated compared with the chemical etching.

It is widely known that plastically deformed metal materials maintain the geometric continuity of its structure by creating dislocations, which leads to variations in lattice orientation (misorientation) in the crystal grains. The degree of local plastic strain can be evaluated by the local average misorientation due to the proportional relationship between them (Kamaya et al., 2006; Zhang et al., 2013).

The correlation between chemical etching and crystallographic orientation has been investigated in many works. The dependence of the etching rates of single-crystal silicon on crystallographic orientation was reported by Sato et al. (1999). Szabó and Bonyár (2012) studied the effect of grain orientation on the effectiveness of chemical etching. Just as mentioned above, the chemical etching is used to reveal the microstructure of the metals or alloys through preferentially etches the grain boundaries. Different morphology of microstructures can be observed by chemical etching depending on the degrees of etching: (1) with light etching only the grain boundaries can be revealed; (2) while with further etching both grain boundaries and grain shading can be manifested (Vander Voort, 1999). Since the rate of etching is affected by the grain orientation, the etching creates contrast in the different oriented grains through differences in reflectivity (Vander Voort, 1999). Zhu et al. observed the grain etching contrast in α-titanium (2004). Orientation-dependent topography formation was observed in FIB cutting (Lenius et al., 2011) and mechanical polishing (Zhu et al., 2004a,b). The dependence of the shade of color etching on crystal orientation was investigated by previous researchers (Szabó and Kardos, 2010). However, it remained unclear whether or not the variation in the crystal orientation (misorientation) inside the individual grains can be revealed by the etching color. Since the chemical etching is an easy way to reveal microstructures, if such misorientation can be revealed by the chemical etching contrast, it would provide a novel and simple way to understand the misorientation and local plastic strain distributions in the microstructures. On the other hand, the dependence of chemical etching contrast on crystallographic orientation itself actually has not been seriously studied yet.

* Corresponding author. Tel.: +81 117066345; fax: +81 117066345.
E-mail addresses: zhangxg@ec.hokudai.ac.jp, zhangxianguang08@163.com (X. Zhang).

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In this study, we aim at manifesting the misorientation caused by the local plastic strain using the chemical etching contrast. For this purpose, the chemical etching contrast dependence of crystallographic orientation is first examined in the undeformed iron, and then the distribution of the misorientation inside the grains of the deformed iron is studied. The results clearly show that the chemical etching contrast strongly reflects the crystallographic orientation and the gradual change in chemical etching contrast within the individual deformed grains give information of both the misorientation and local plastic strain inside the grains.

2. Material and methods

The material used in the present study is commercially pure iron with a purity of 99.95 mass% (Si < 0.01, Mn < 0.01, *P* < 0.002, *S* < 0.002 and Al < 0.001 mass%.). The as-received pure iron bars were annealed at 1273 K for 60 min and then slowly cooled to room temperature to obtain ferrite grains structure. The deformation was carried out by a ball-pressing (BP) test at room temperature; the set-up and procedure of which can be found in our previous report (Zhang et al., 2013). After the deformation, longitudinal mid-plane sections were prepared for both electron backscatter diffraction (EBSD) measurement and optical microscopy (OM) observation. The surface was mechanically polished using silicon carbide paper to grade 2000, then on a polishing cloth with a liquid suspension of 40 nm alumina in order to achieve flat surfaces without any damage associated with the sample preparation. It is an important step to produce high quality images. The EBSD measurements were done with a JEOL JSM-6500F field-emission scanning electron microscope (FE-SEM) equipped with a TSL software. The step size was set to 3 μm in all the cases. The grain boundary was defined as a boundary involving misorientation larger than 5°. After the EBSD measurement, the samples were chemically etched by 3% nital at room temperature for 5–8 s followed by OM observation (KEYENCE VHX-1000). The etching time should be carefully controlled to get a clear etching contrast, and to avoid overetching. The difference in height of each grain on the etched surface was measured using an atomic force microscope (AFM; KEYENCE VN-8000) working in the tapping mode.

3. Results and discussion

Fig. 1(a) and (b) shows an optical image of the undeformed sample after chemical etching and the corresponding crystal orientation map of the same area, respectively. The individual grains are numbered in both figures. It can be seen in Fig. 1(a) that the etching colors of the grains are varying over the observation area. By comparing Fig. 1(a) with (b), it is very interesting that the different etching colors of the grains correspond to different grain orientations, which clearly indicates that the variety of the etching-contrasts of the grains is a manifestation of the variety of the grain-orientations. This shows good agreement with the previous report (Vander Voort, 1999).

In order to determine the dependence of chemical etching color on the grain orientation, the correlation between the luminance of each grain in the chemically etched image and the orientation angles between surface normal and directions of [0 0 1], [1 0 1] and [1 1 1] is investigated, and the result is shown in Fig. 2. The luminance (*Y*) can be calculated by the following equation (Cheng, 2001):

$$Y = 0.299\,R + 0.587\,G + 0.114\,B \quad (1)$$

where *R*, *G* and *B* are color components of red, green and blue and their values can be measured for each grain in Fig. 1(a). It can be clearly seen that when the grain orientation is getting close to [0 0 1] direction, the luminance tends to increase, which means that bright colored grains in the etched structure has crystal orientation close to [0 0 1] direction. While the grain orientation is getting close to [1 0 1] and [1 1 1] directions, the corresponding etching color tends to be darker. Hence, the chemical etching contrast clearly depends on the crystallographic orientation, and the grain orientations can be roughly identified from their chemically etched color. Importantly, the variation in crystal orientation, viz. misorientation inside the grains caused by plastic deformation can be revealed by the chemically etched microstructures, as described in detail in the following part.

Fig. 3(a) shows the chemically etched image of the deformed sample and Fig. 3(b) is the corresponding crystal orientation map. From Fig. 3(b) it can be seen that the individual grains show varying shades of colors, indicating local rotation of crystal orientation,

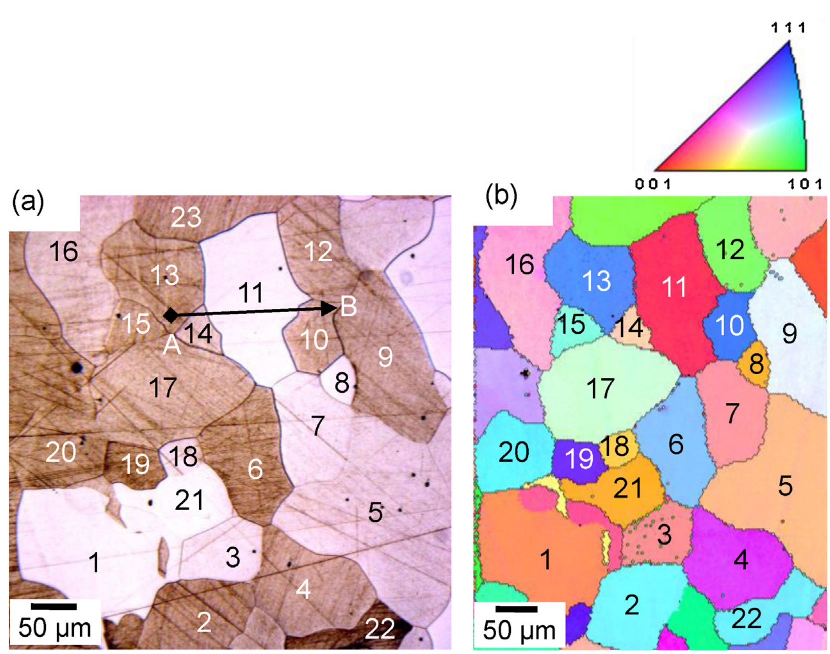

Fig. 1. (a) Optical image of chemically etched undeformed sample and (b) orientation map of the same sample area.