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Orientation contrast of secondary electron images from electropolished metals

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ABSTRACT

Orientation contrast obtained by an in-lens secondary electron detector in a scanning electron microscope from electropolished/etched metals is reported. The imaging conditions for obtaining such orientation contrast are defined. The mechanism responsible for the formation of the orientation contrast is explained, and an application example of this new imaging method is given.

1. Introduction

In the scanning electron microscope (SEM), secondary and backscattered electrons are the two most often used imaging signals. Secondary electron image (SEI) is the most widely used imaging mode in the SEM because the large depth of field enables 3D images to be obtained [1–3]. Backscattered electrons are often used to obtain compositional and crystallographic contrast of a specimen [1–3]. Crystallographic contrast obtained by backscattered electrons arises from the channeling effect of the incident electrons, which causes the backscattered electron coefficient to vary with the orientation of the crystals examined [4,5]. The crystallographic contrast obtained by backscattered electrons is low, typically ~2–5% [2]. Weak crystallographic contrast may also be seen in SEI, since secondary electrons induced by backscattered electrons (the so-called SE2 [6]) are detected. SEI has not been used to obtain crystallographic contrast because of its low crystallographic contrast. In this paper, it will be reported that strong orientation contrast[1] can be obtained by SEI from metallic specimens under suitable imaging conditions and specimen preparation method.

2. Experimental

An austenitic steel with a composition, 24 wt% Mn, 3 wt% Al and 0.6 wt% C was used in this study. Before examination in the SEM, the specimen was electropolished/etched[2] using an electrolyte of 20% perchloric acid and 80% glacial acetic acid at room temperature, at 25 V. The SEM used in this research is a Zeiss Supra 55, which is equipped with a Schottky-type field emission source. A beam booster voltage of 8 kV is added to the landing voltage chosen by the operator throughout the whole column. After passing through the scanning system, incident electrons are decelerated to the landing voltage by an electrostatic lens at the end of the electron column. There are two secondary electron detectors. One is an annular in-lens detector located above the objective lens inside the column. Secondary electrons emitted from the specimen surface are accelerated by the electrostatic lens and focused onto the annular in-lens detector. The other one is a conventional Everhart–Thornley (E–T) detector mounted on the wall of the specimen chamber. A quadrant solid state backscattered electron detector (BSD) is mounted on the bottom of the objective lens. In this SEM, there is a high current mode (HC) available.

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[1] Orientation contrast is used to differentiate the crystallographic contrast obtained by backscattered electrons, and secondary electrons induced by backscattered electrons.

[2] It will be shown later that to judge whether the specimen is polished or etched depends on the magnification used.

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0304-3991/

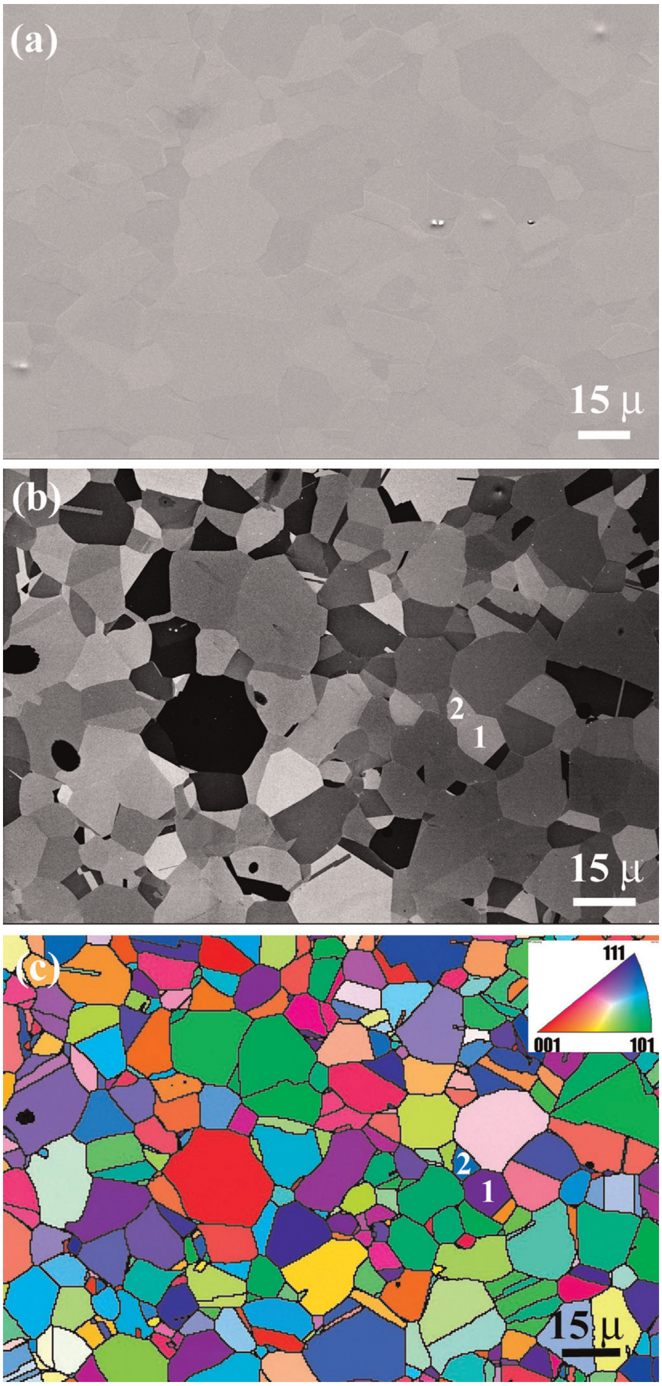

Fig. 1. (a) SEI of the specimen imaged using the E–T detector showing the featureless electropolished specimen surface. V_{ac}=15 kV, aperture size=30 μm, WD=5 mm, HC mode off, B=45.0%, and C=37.9%. (b) SEI of the specimen detected by the in-lens detector showing strong orientation contrast. V_{ac}=5 kV, aperture size=120 μm, WD=5 mm, HC mode on, B=32.3%, and C=24.1%. (c) OIM from the area of (b). V_{ac}: acceleration voltage, WD: working distance, B: brightness setting; C: contrast setting.

When the HC mode is applied, the condenser lens is switched on, and a beam current higher than when the HC mode is off can be obtained. Three objective aperture sizes are used, namely, 30, 60, and 120 μm. Working at 15 kV, 30 μm aperture, HC mode off is recommended by the manufacturer to obtain the highest resolution. No post image processing has been applied to all the SEM images shown in this paper, after they have been recorded in the SEM. Orientation image maps (OIMs) were obtained by an electron backscattered diffraction (EBSD) system, Aztec 2.3, from Oxford Instruments.

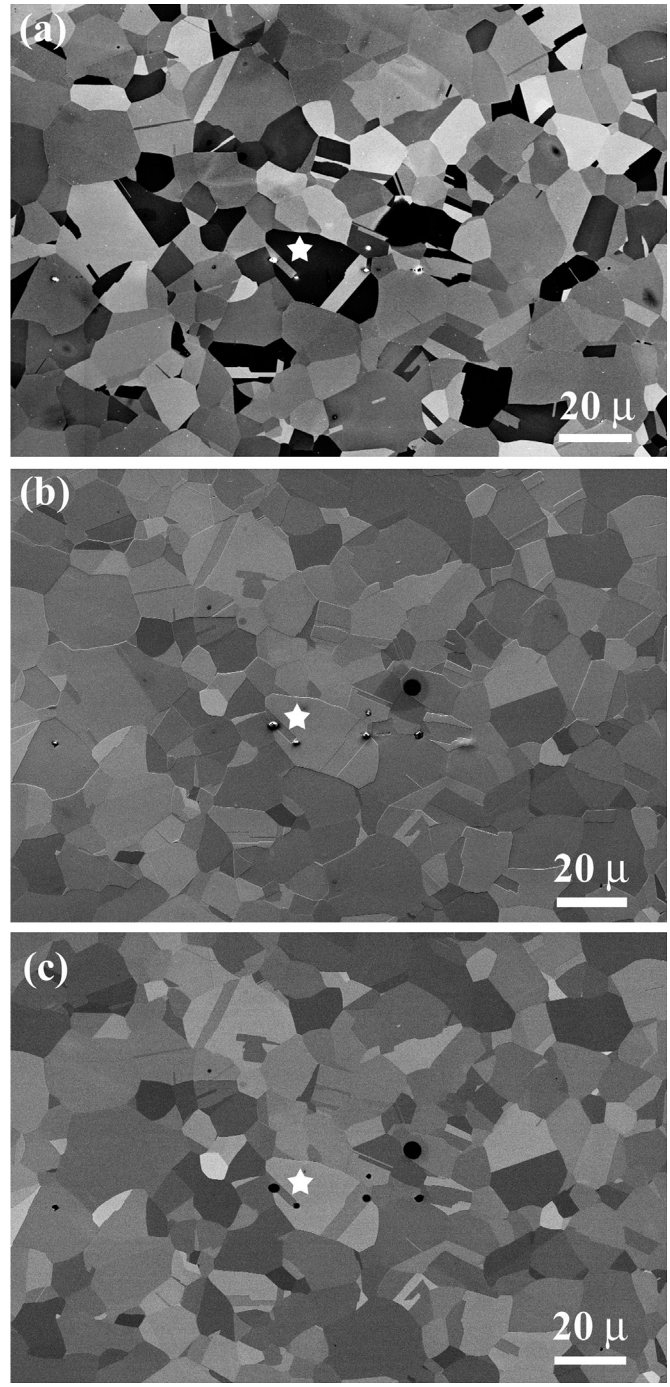

Fig. 2. SEM micrographs from the same area imaged using different detectors. White stars are registration marks. Acceleration voltage=5 kV, aperture size=120 μm, working distance=5 mm, HC mode on. (a) In-lens detector, B=31.6%, C=24.1%, (b) E–T detector, B=0%, C=33.5%, and (c) BSD, B=28.2%, C=86.9%. B: brightness setting; C: contrast setting.

The various imaging conditions are given for all images in the figure legends and are not repeated in the text.

3. Results

Fig. 1 shows the images of the specimen. Fig. 1(a) is a conventional SEI detected by the E–T detector at 15 kV, which shows that the specimen surface appears to be flat and featureless. Fig1(b) shows an image obtained at 5 kV using the in-lens detector which shows very strong orientation contrast between grains and between twinned