3D microstructural analysis in the steel industry

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Abstract. Since steel products have complex microstructures and chemical composition to achieve high strength and high functionality, it is important to characterize the internal structure with analytic techniques and to be able to predict their properties using simulation techniques. It is now possible to characterize and analyze the structural arrangements in raw steel materials and steel products, including the shapes of defects such as voids and cracks, as well as the crystallographic grain structure, by use of powerful new 3D characterization and analysis techniques, which can provide information on the internal structure on the micron or even submicron scale. It is expected, therefore, that application of new methods and results should lead to improvements in the mechanical properties of steel products. In this article, 3D analysis of the internal structure in a duplex stainless steel, and of strain propagation in pure nickel are presented as examples of the industrial application of 3D microstructural analysis.

1. Introduction
Steel products have long been used as base products to support the infrastructure of human society. In the development of steel products, microstructure analysis provides important information in the quest to improve both mechanical properties and functionality. Since steel products have complex microstructure and chemical composition in order to achieve their high strength and superior functionality, it is important in research and development of steel products to be able to predict their properties using both analytic and simulation techniques. Such simulations require detailed knowledge of material parameters influencing internal structure formation and mechanical properties, such as the dislocation structure, the boundary structure, precipitates, and grain shapes, sizes and orientations, as well as crack and void shapes. In order to obtain these important properties, 2D techniques such as optical microscopy (OM), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and atom probe field ion microscopy (AP-FIM) have been used for many years, and these techniques are complementarily used to obtain multi-scale information. Recently, the importance has been recognized of developing these 2D techniques into 3D techniques, such as X-ray computed tomography (CT), serial sectioning OM and SEM, TEM/STEM-CT and atom probe tomography (APT) for materials analysis, as shown in figure 1.

In this article, a 3D analysis of the internal structure in a duplex stainless steel, which has a complex phase domain shape, and of strain propagation in pure nickel are presented as examples of the industrial application of 3D microstructural analysis.
2. 3D analysis techniques in the characterization of steels

Several 3D analysis techniques, such as serial sectioning and tomography techniques, have been developed to obtain the 3D surface morphology and internal structure. The simple way to take 3D structure images of objects is to use the serial sectioning technique. In this technique, a sample block is sliced by mechanical polishing and/or ion beam machining, and 2D images of cross-sections of the object are acquired by microscopic techniques, such as OM and SEM. Slicing of the sample block and acquiring of images are alternatively carried out, such that a 3D image can be reconstructed from a series of 2D images. Therefore, an important requirement in the technique is to cut each slice parallel to the sample block face, with each slice the same thickness, as precisely as possible. Furthermore, drift correction of 2D images is required in case the sample block moves during acquisition. Serial sectioning is a destructive analysis technique, and as such the sample block is lost disappeared after data acquisition. In contrast, tomography techniques using X-rays or TEM/STEM can be used to obtain 3D images of a sample block non-destructively, because the technique does not destroy the sample block during measurement. In these techniques, transmitted images are acquired by rotating a sample block around an axis perpendicular to the incident X-ray or electron beam. A 3D image of the sample is reconstructed with a series of the transmitted images by reconstruction software.

Since steel materials typically have a very fine internal structure, down to the micro- or sub micro-meter size, application of such 3D analysis techniques to observation of the fine structures in steels requires high spatial resolution. Serial

![Figure 2 3D image of rolling contact fatigue cracks (light blue) around inclusions (yellow and orange) reconstructed with serial sectioning SEM images.](image)
Sectioning using a focused ion beam (FIB)/SEM system is useful to allow information to be obtained at the micro- or sub-micro meter scale, due to the precise sectioning that can be achieved using a FIB system and the high resolution available by SEM. Furthermore, EDS and EBSD measurement are also available in such a system [1]. Figure 2 shows an example of 3D image obtained by FIB/SEM. Thin fatigue cracks can be observed around inclusions in the steel sample. Tomography techniques using TEM/STEM can provide still higher resolution 3D images [2], though it is necessary to use a sample thin enough to be electron beam transparent. For a steel, this limits the sample thickness to a maximum of 300nm if information on the internal structure is required. As such it is difficult to obtain crystalline grain shapes in steels using this approach. Since X-rays can transmit through steel of almost 1 mm in thickness, it is possible to obtain non-destructively information on the crystalline grain structure, as well as the phase domain shape and non-metallic inclusions in bulk steel material using an X-ray tomography technique.

3. 3D internal structure analysis of duplex stainless steel

Microscopy, such as SEM and TEM, has been widely used to observe the internal micro-structure of steel materials. Although the obtained data show very useful and important 2D information about the internal structure, 3D information is also needed to provide a complete picture of the true structure in the material, such as the crystalline grain structure, the phase domain structure as well as non-metallic inclusions. In the following, an internal structure analysis of a duplex stainless steel with a complex structure of ferrite phase (bcc) and austenite phase (fcc) is described as an application of the 3D characterization of steel. As a destructive analysis, 3D-EBSD is used to obtain sub-micron scale 3D information. X-ray micro-CT is additionally used to obtain 3D information of a volume larger than that available using 3D-EBSD.

3.1. Experimental procedure

A hot-drawn duplex stainless was used as a sample in this experiment. For the 3D-EBSD measurement, cross-sectional FIB machining was applied to a 100µm×100µm area. The cross-section was sliced to 200nm in thickness. FIB machining and EBSD measurements were carried out alternately. Each EBSD measurement was carried out over a 60µm×60µm area. For the X-ray micro CT measurement, a needle shaped sample of 500µm in diameter and 10mm in length were prepared. The surface of the sample was polished by electrolytic polishing. A polychromatic X-ray beam was used to take transmitted X-ray images. The images were taken by rotating the sample through a 360 degrees rotation in a total of 3201 steps.

3.2. Results and discussion

Figure 3 shows 3D images the hot drawn duplex stainless steel obtained from the 3D-EBSD data. The

![Figure 3 (a) 3D phase map and (b) IPF map of a duplex stainless steel taken using 3D-EBSD [3].](image-url)
reconstructed volume is $56.8\mu m \times 37.6\mu m \times 6.6\mu m$. Using information of the crystalline phase and orientation in obtained EBSD data, the 3D shape of the phase domains and of crystalline grains can be reconstructed as shown in figure 3(a) and 3(b), respectively. In figure 3(b), the orientations of the crystalline grains are indicated by inverse pole figure (IPF) coloring. Although the thickness of the obtained volume is too small to provide the complete shape of the crystalline grains of the ferrite and austenite phases, it is nevertheless possible to visualize the complicated 3D structure of the phase domain boundaries and grain boundaries by 3D-EBSD.

Figure 4(a) shows the crystalline structure for each grain. Twin boundaries are highlighted by red lines in the figure. Figure 4(b) shows a cross-sectional view of the section indicated by a yellow line in figure 4(a). The cross-section is perpendicular to the twin boundary. The twin boundary plane, indicated with a red arrow, lies approximately perpendicular to the surface of the observed volume. The twin boundary is not straight but has a complicated curvature. This is in fact an artifact of the serial sectioning technique. In order to reduce the influence of such artifacts, it is necessary to obtain a completely uniform thickness for each slice, with more precise drift correction between each slice. The angle between the plane orientation of twin boundary {111} and the normal orientation of the grains 1 and 2 is calculated to determine the plane orientation of the twin boundary. Table 1 shows calculated angular relationships. The angle between the twin boundary (1 1 -1) and the grains is approximately $90^\circ$. Therefore, the plane orientation of the twin boundary can be uniquely decided to be (1 1 -1).

As the austenite phase domain has a volume much larger than the volume that can be observed by 3D-EBSD, it is difficult to understand the whole of the domain. Therefore, absorption contrast tomography (ACT) using X-ray micro CT has also been used to obtain the whole shape of the austenite phase domain. Figure 5 shows cross-sectional views of the ACT image of the duplex stainless steel. The bright and dark contrast regions are identified as ferrite and austenite phases, respectively. Non-metallic inclusions are also observed as black particles. Figure 5(d) shows the 3D shape of the austenite phase domain extracted from figure 5(a). Non-metallic inclusions are also indicated in red in figure 5(d). It is revealed that austenite phase domain is elongated along the drawing direction, and each domain shows a cylindrical shape of 30 to 100$\mu m$ in diameter and 100 to 500$\mu m$ in length. Thus, it is certainly possible to obtain morphological information of phase domain in

Table 1 Angular relationship between plane orientation of twin boundary and surface normal direction of crystalline grain.

| Twin boundary | Grain 1 [315] | Grain 2 [256] |
|---------------|--------------|--------------|
| (1 1 -1)      | 95.6$^\circ$ | 85.9$^\circ$ |
| (1 -1 1)      | 46.9$^\circ$ | 77.6$^\circ$ |
| (-1 1 1)      | 73.0$^\circ$ | 49.9$^\circ$ |
| (1 1 1)       | 28.6$^\circ$ | 21.1$^\circ$ |

Figure 4 (a) Crystal structure for each crystalline grain and (b) cross-sectional view of a twin boundary.
metallic materials using ACT, but it is not possible with this technique to obtain crystallographic information, such as the grain shape, structure and orientation as can be achieved using 3D-EBSD. Recently, a new technique, diffraction contrast tomography (DCT), has been developed that allows the 3D shape and orientation of crystalline grains to be obtained [4-6]. Complementary use of ACT and DCT will be a powerful technique to observe the internal structure in metallic materials non-destructively.

4. Propagation of plastic deformation in pure Nickel taken by 3D-EBSD
The distribution of plastic and elastic deformation in crystals effects their recrystallization behavior. Therefore, deformation behavior has been studied by several simulation techniques, such as finite element modeling and crystal plasticity simulations. Actually, deformation in crystals propagates three dimensionally. As such 3D-visualization techniques are therefore required to obtain the deformation behaviour of crystals experimentally. In order to discuss deformation and fracture behavior of metallic materials, it is important to understand the propagation of plastic strain and deformation in a crystal grain or in the vicinity of a grain boundary, as materials typically consist of many crystal grains. The 3D-EBSD technique has been applied to analyze the propagation of plastic deformation in metallic materials [7].
4.1. Experimental procedure

A well-polished pure nickel plate (10mm \( \times \) 10mm \( \times \) 1mm) was annealed in a vacuum furnace for 3 hours at 973K, and then a small impression was made on a surface of the plate using a Vickers indenter, which has a diamond tip in the form of a square based pyramid. A weight of 10 g was loaded on the indenter. The depth of the impression was smaller than 2 \( \mu \)m under the loading condition. 3D-EBSD measurements were carried out with an integrated instrument combining FE-SEM, FIB and EBSD. In the instrument, cross-sectioning and orientation mapping are automatically acquired under control by dedicated control software for 3D imaging. Figure 6 shows snap-shot taken images during the EBSD measurement and FIB machining. EBSD maps of 30 \( \mu \)m \( \times \) 30 \( \mu \)m area were obtained in the experiment. The step size of each EBSD map was 200 nm, and maps were taken using a slice thickness of 200 nm. In order to extract information of plastic deformation from EBSD maps, misorientation angles between the deformed and non-deformed areas were calculated using rotation matrix calculations [8].

4.2. Results and discussion

Figure 7 shows the distribution of plastic deformation in the nickel plate, where a small indent has been made in the vicinity of twin boundary. Figure 7(a) shows a 3D map reconstructed from a series of contour maps of misorientation angle obtained from the 3D-EBSD measurement results. In the 3D map it is seen that the nickel crystal is slightly deformed and still has a misorientation angle of several degrees at a depth of 10 \( \mu \)m from the surface. This indicates that the plastic deformation made with the indenter extends to a region five times as deep as the depth of the indent. Figure 7(b) shows a two-dimensional contour map of misorientation angle obtained at a depth of 1.8\( \mu \)m from the surface of the nickel plate. The deformed region around the impression has a misorientation angle larger than 5°. The region near the center of the impression, however, has a misorientation angle smaller than 1°. This indicates that severe plastic deformation occurs at in material touching the face of the pyramid shaped indenter, and that little deformation occurs at the apex of the indenter. Figures 7(c) and 7(d) show cross-sectional views across and along the twin boundary, respectively. Plastic deformation in the crystal shows anisotropic distribution, and propagation of the deformation is obstructed at the twin boundary and parallel to \{111\} planes. It is considered that relaxation of the plastic deformation caused by the slip planes, \{111\}, and the twining planes having the same index as the slip plane is responsible for the anisotropic propagation. Thus, 3D-EBSD is a powerful technique to visualize the

![Figure 6 Snap-shot images taken during (a) EBSD measurement and (b) FIB machining [7].](image-url)
3D propagation of plastic deformation and strain, and the obtained results should contribute to crystal plasticity studies.

5. Conclusion

3D analysis can provide a full picture of the morphology of the internal structure in steels, including the crystalline grain arrangement, the phase domains and the presence of non-metallic inclusions, as well as providing information on the propagation behavior of plastic deformation. In order to observe the evolution of such features and morphological changes during production processes, time-resolved 3D observations should be realized. Serial sectioning techniques can yield information at the micro- or sub micro-meter scale. It is difficult, however, to apply such techniques to in-situ observations, because serial sectioning is a destructive technique. Although tomography techniques can be used to observe a sample non-destructively, there are still issues regarding the ability for time-resolved observations, due to long acquisition times. Therefore, high intensity sources, such as synchrotron radiation, and high sensitivity detectors are required to improve the acquisition time. Collaborative analysis between in-situ observations and 3D analysis will be a key technique to observe the dynamical behavior of internal structure evolution and morphological changes, it is expected such combined data will support the development of advanced steel products in the near future.

**Figure 7** (a) 3D reconstructed image and (b) X-Y cross-sectional view of plastic strain distribution in the pure Ni plate indented by Vickers indentation. Cross-sectional views of (c) and (d) show the cross-sections along the red and yellow dotted lines indicated in (b), respectively [7].
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