Multimodal assessment of mechanically induced transformation in TRIP steel using X-ray micro- and nano-CT and pencil-beam XRD-CT

There has been growing interest in multi-phase steels with meta-stable retained austenite. The retained austenite phase, which is embedded as a minor phase in a ferrite-based microstructure, is stable at room temperature. The retained austenite phase transforms into martensite with volume expansion under external loading, which has a beneficial effect on the strength-ductility balance of multi-phase steels. In retained austenite steels, traditional metallographic preparation, such as cutting and polishing, inevitably introduces austenite-to-martensite transformation due to plastic deformation. A great deal of work has therefore been undertaken employing quantum beams for *in situ* observation. *In situ* diffraction experiments offer a method of measuring morphological and crystallographic characteristics of individual grains in 3D [1]. Remarkable spot blurring and spot overlap however occur during plastic deformation in XRD experiments [1]. Recently, the present authors proposed a multi-modal technique called diffraction-amalgamated grain boundary tracking [2]. In the present study, the technique was used to quantitatively assess the role of individual austenite grains in relation to austenite-to-martensite transformation behavior during plastic deformation. Imaging of the dual phase microstructure has been utilized to directly visualize retained austenite grains by utilizing the phase-contrast X-ray tomography that has recently been improved to 150 nm spatial resolution at 30 keV owing to the development of a type of X-ray Zernike phase-contrast X-ray nanotomography (XNT) by Takeuchi *et al.* at SPring-8 BL20XU [3].

A multi-phase steel consisting of a retained austenite interconnected in a reticulate manner was used (V_f of 22.1%). Both the XNT and XRD experiments were performed at SPring-8 **BL20XU**. A projection-type X-ray micro-tomography (XMT: 1.0 μm in spatial resolution) setup was placed side-by-side with the XRD and XNT setup to switch the three setups repeatedly. A specimen was loaded in tension and its deformation and transformation behavior was observed by the XMT, XNT and XRD techniques. A monochromatic X-ray beam of 20 keV was used for the XMT and XNT experiments. In the XNT Experiment, a large-scale X-ray microscope, which has *L* of 165 m, was used together with Köhler illumination to establish the XNT setup with Zernike phase-contrast mode [4]. A spatial resolution of 0.16 μm was achieved in the reconstructed images. In the XRD experiment, an X-ray pencil beam, which was collimated to 2.0 μm in diameter with a FZP, was raster scanned (50 rows and 50 columns in 1 μm increments) with the sample being rotated over 180° in 1° increments and the raster scan was repeated at each angle, resulting in a total of 450000 diffraction patterns being captured for each loading step. The X-ray energy was tuned to 37.7 keV for XRD.

3D morphology and initial crystallographic orientation of all the retained austenite grains were successfully obtained as demonstrated in Figs. 1 and 2 [5]. Complex austenite morphology was observable with their annihilation behavior during loading. The morphological change in the retained austenite is clearly seen in Fig. 1. The onset of the austenite-to-martensite transformation was seen between the applied strain of 0.3–0.4%. Localized transformation, which is indicated by an arrow in Fig. 1(a), is observed at an applied strain of 1.3% for the green grain. The local transformation then propagated, progressing vertically into a wedge-shape at 2.5%, followed by an abrupt transformation between 2.5 and 3.5%. It is also observed that an upper adjacent gray austenite grain transformed at roughly the same time as the green austenite grain, whereas the lower right gray austenite grain remained up to 3.5%, implying a variety of transformation behaviors for individual grains.

The change in austenite volume fraction was classified according to the initial crystallographic

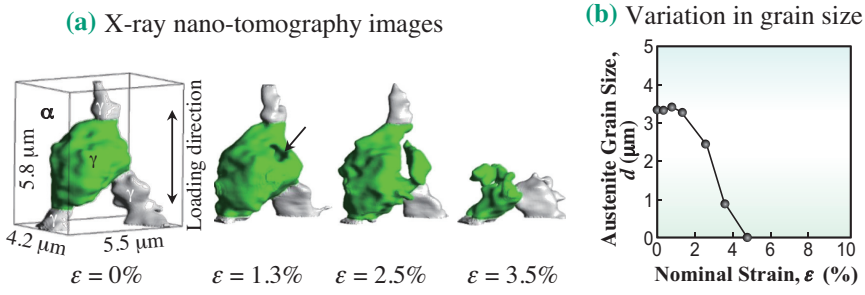

Fig. 1. **(a)** Typical example of austenite-to-martensite transformation during loading, together with the variation in grain size of the grain shown in **(b)** expressed in terms of equivalent sphere diameters. The green retained austenite grain was connected to the three adjacent austenite grains highlighted in gray.

orientation, and the annihilation rates were readily comparable among different orientations: retained austenite grains with a tensile axis within 15° of their initial <110> direction exhibit rapid transformation, whereas those within 15° of the initial <111> direction exhibit a low transformation rate.

Figure 2 successfully identifies crystallographic orientations of all 58 austenite grains throughout transformation [5]. The arrows in Fig. 2 indicate rotation paths during transformation. When the transformation is protracted up to 3.5% or above, the majority of the grains are rotated to a non-negligible extent; for example, 2.0° in rotation angle on average for 5.8%. It is also noteworthy that some of the grains reversed their paths in the middle of transformation, for example, the typical reversal by the grain circled in black.

The respective variations in dislocation density in the retained austenite grains are also measured. The dislocation multiplication behavior of austenite grains was strongly affected by their crystallographic orientation; the austenite grains close to the <111> orientation exhibit a gradual increase in dormant dislocation density, whereas those close to the <110> orientation exhibit rapid multiplication. The <100> direction appears to represent an intermediate tendency between the <111> and <110> directions.

Figure 3 illustrates the variety of behaviors observed during the increase in dislocation density and transformation [5]. It is evident that rapid transformation is apt to occur for austenite grains that exhibit abrupt dislocation density increase (grains G1, G3, and G6), whereas those with moderate dislocation density increase (grains G2, G4, and G5) tend to show slow and gradual transformation.

In summary, the gradual transformation, plastic deformation, and rotation behaviors of the individual austenite grains were clearly observed in 3D. It was revealed that the early stage of the transformation was dominated by the stress-assisted transformation that can be associated with measured mechanical driving force, whilst the overall transformation was dominated by the strain-induced transformation that is interrelated with measured dislocation multiplication. The transformation behavior of individual grains was classified according to their initial crystallographic orientation and size. It was conclusively demonstrated that such characteristic behavior partly originated from interactions with surrounding soft and hard phases.

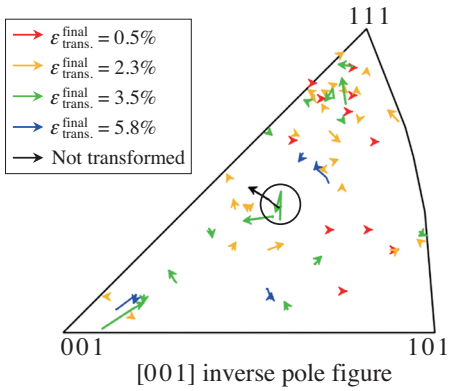

Fig. 2. Crystallographic orientation distribution and rotation behavior on a [001] inverse pole figure. All 58 austenite grains that were successfully identified are color-coded according to the external strain level at which the austenite-to-martensite transformation is completed.

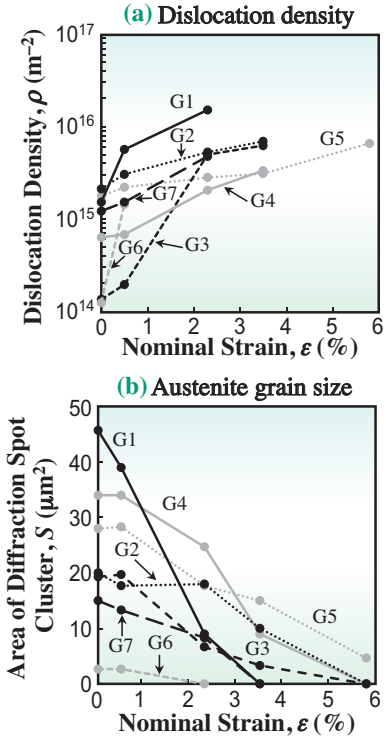

Fig. 3. Changes in **(a)** the dislocation density and **(b)** the retained austenite grain size with applied strain. Seven characteristic grains (G1–G7) have been extracted from the total number of grains obtained. The retained austenite grain size is expressed as the average of the projected spot-cluster areas obtained for three incident beam orientations; diffractions from the {111}, {200}, and {220} planes.

Hiroyuki Toda* and Hiro Fujihara

Department of Mechanical Engineering, Kyushu University

*Email: toda@mech.kyushu-u.ac.jp

References

[1] N. H. Jimenez-Melero *et al.*: Scripta Mater. **56** (2007) 421.
[2] H. Toda *et al.*: Acta Mater. **107** (2016) 310.
[3] A. Takeuchi *et al.*: Rev. Sci. Instrum. **92** (2021) 023701.
[4] H. Toda: X-Ray CT Hardware and Software Techniques (2021) Springer Nature Singapore.
[5] *H. Toda, K. Hirayama, K. Okamura, T. Suzuki, A. Takeuchi, M. Uesugi, H. Fujihara: Acta Mater.* ***234*** *(2022) 117956.*