

## Microscopic 3-dimensional stress distribution inside crystalline grains of bulk steel under plastic deformation measured by scanning 3-dimensional X-ray diffraction microscopy

Assuring the reliability of smaller, lighter, and more functional components is becoming an increasingly important issue in transportation industries. One of the key properties in reliability analysis is mechanical stress, which is categorized into three types. Type I stresses are macroscopic stresses that can be measured using commercial X-ray diffraction equipment and are widely used in mechanical designing and computer-aided engineering. Type II stresses are grain-averaged stresses, which represent individual grains; therefore, the value differs from grain to grain. On the other hand, type III stresses are intragranular stresses, i.e., local stresses inside a grain, which have a distribution within the grain. The measurement of type III stresses is important because the life and strength of a component are considered to depend on the type III stresses that affect the microscopic crack propagation behavior. Although measuring the 3-dimensional microscopic distribution of stress is necessary for improving the reliability of components, conventional techniques are not capable of accomplishing it nondestructively. Synchrotron X-ray-based techniques, such as 3-dimensional X-ray diffraction (3DXRD) [1-3], have been realized in nondestructive measurements using high-energy and super-brilliant X-rays. In this research [4], we developed a scanning 3DXRD microscopic methodology that combines a highly transmissive synchrotron X-ray microbeam with techniques of extracting the internal information of single grains.

The schematic of the scanning 3DXRD measurement system constructed at SPring-8 BL33XU [5] is shown in Fig. 1. The size of the incident synchrotron X-ray microbeam is 1×1 µm<sup>2</sup> at the measurement volume in the specimen. To detect diffracted beams from only the measurement volume, a conical slit was placed between the specimen and area detector. The conical unit of concentric slits blocks the diffraction from the outside of the measurement volume. Diffraction images were obtained every 0.6° while the specimen was rotated for 180° around the vertical axis. The stresses at the measurement point can be analyzed from the diffraction images. The 3-dimensional distribution of the stresses was obtained by scanning the specimen at intervals of 1.2 µm. The measured specimen is low-carbon steel with a tensile strength of  $\sigma_{TS}$  = 330 MPa. The sample has a cross-sectional size of  $1 \times 1 \ \mu m^2$  with a mean grain size of 20 µm. Scanning 3DXRD measurements were conducted while the specimen was plastically deformed by 5.1% under tensile loading.

We succeeded in nondestructively measuring the 3-dimensional distribution of type III stresses inside the grains of bulk polycrystalline steel under tensile deformation using a 1  $\mu$ m X-ray beam, as shown in Fig. 2. The macroscopic type I stress, calculated as the average for all voxels, was 240 MPa, which approximately agrees with the applied tensile load. The type II stresses in the cubic area were not more than the tensile strength of  $\sigma_{TS}$ = 330 MPa. On the



Fig. 1. Schematic of internal stress measurement by scanning 3DXRD microscopy with a conical slit at BL33XU.



Fig. 2. Measured 3-dimensional distribution of type III stresses in the vertical direction. The size of the cube is  $44.4 \times 44.4 \times 44.4 \mu m^3$ .

other hand, the type III stresses exceeded  $\sigma_{\text{TS}}$  and reached 500 MPa after the 5.1% elongation.

An internal 2-dimensional map of type III stresses

on a plane normal to the tensile direction is shown in Fig. 3(a). The distribution of microscopic stresses is shown in Fig. 3(b) along line A in (a). Since the type II stresses are independent of position in each grain, they are represented by a constant value indicated by the blue line. The type III stresses have the distribution shown by red dots. Error bars were evaluated using the deviations of forces from equilibrium. The type III stress inside the grain exceeds  $\sigma_{TS}$ = 330 MPa and reaches 520 ± 160 MPa. The results show that the local stresses greatly deviate from the average stresses measured by conventional methods.

The developed technique will enable the extraction of weak points inside components and the identification of their degradation and deformation mechanisms. This should contribute to the development of even more reliable components and manufacturing processes for vehicles, home appliances, and information/ communications equipment. Since this technique enables the nondestructive measurement of stresses from the microscopic to the macroscopic scale, it should also facilitate the development of multiscale material modeling that expresses both fractures and deformation, as well as simulations that predict the life of components.



Fig. 3. (a) Internal 2-dimensional map of type III stresses on a plane normal to tensile direction. (b) Distribution of type III stresses along line A in (a) markedly deviated from that of type II stresses.

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