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## In situ X-ray microdiffraction study of deformation-induced phase transformation in 304 austenitic stainless steel

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#### Abstract

The traditional phenomenological crystallographic theory of martensitic transformations can only explain the change in the shape and crystallographic orientation of a martensitic plate within a single parent crystal. It cannot predict the detailed transformation scenario for preferred selections of martensitic variants or the contributions of partial slip/twinning to local lattice distortion, especially in polycrystalline metals/alloys that exhibit grain-to-grain interactions throughout deformation-induced phase transformation. In this work, synchrotron-based X-ray microdiffraction was used to characterize changes in the local orientation, morphology and strain distribution inside individual martensitic plates, as well as the effect of parent orientation on variant selection in bulk polycrystalline 304 stainless steel (SS) during in situ uniaxial tensile loading at the low temperature of 210 K. It was directly verified that the martensitic phase transformation in the studied 304 SS has two stages, transformation first from  $\gamma$  to  $\varepsilon$  in the nanoscaled lamella, and then from  $\varepsilon$  to  $\alpha'$  in the microbands. The selection of martensitic variants was predicted well by the minimum strain work criterion. Phase transformation-induced stress relaxation was evidenced by fluctuations in the (111) plane lattice strain accompanied by a strain gradient inside the martensitic plate, indicating a load transfer from the transformed grain to its neighbor. This leads to good stress/strain accommodation, as stresses can dissipate from the matrix into martensitic plates and nearby grains. Our experimental observations and theoretical analysis provide an in-depth understanding of the micromechanical behavior, particularly phase transformation-induced plasticity enhancement, of metals containing the metastable phase.

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#### 1. Introduction

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Metastable austenitic stainless steels with low stackingfault energies have been widely used in many industrial applications, as they have a good combination of strength and plasticity in addition to excellent corrosion resistance. It is generally recognized that strain/stress-induced martensitic phase transformations enable good strain

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accommodation during deformation, thus enhancing the plasticity of materials [1–3]. Typical examples of these alloys are the transformation-induced plasticity (TRIP) steels, which exhibit both high strength and excellent plasticity due to deformation-induced martensitic phase transformation behavior. The in situ three-dimensional (3-D) high-energy X-ray diffraction (HE-XRD) method was successfully used to characterize the thermal stability of the retained austenite and to monitor the martensitic transformation kinetics of individual austenite grains in TRIP steel as a function of temperature and/or applied stress [4–7]. It

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was found that the mechanical stability of retained austenite in TRIP steel is closely related to complex interactions between the austenite's composition (i.e. the distribution of carbon, aluminum and phosphorus), grain orientation, grain size, temperature and stress state. Although these findings could reveal the overall or specified transformation kinetics in each phase or individual austenite grain, it is difficult to obtain detailed crystallographic information during the phase transformation. To optimize the mechanical performance by enhancing both strength and plasticity using metastable microstructures, it is essential to first understand the changes that occur in microstructural features, such as the geometric arrangement of martensitic variants, the orientation relationship (OR) between the parent austenite and the daughter phase, and the strain/ stress distribution inside parent grains and martensites during plastic deformation. The above-mentioned features strongly reflect the mechanisms of martensitic phase transformations and affect the micromechanical behavior of alloys. Therefore, it is necessary to reveal the mechanisms of strain/stress accommodation and the detailed transformation scenario during deformation. Due to the existence of complex multiphase in TRIP steel, i.e. ferrite-bainite matrix and metastable retained austenite, metastable austenitic stainless steel with only an austenitic matrix is an ideal target for exploring the local crystallographic features, including the strain/stress distribution during martensitic transformation.

The stacking-fault energy (SFE) of austenite, which is a function of the alloy composition and temperature, is one of the most important factors controlling deformation mechanisms [8,9]. A material with a low SFE prefers to follow mechanism (a), a  $\gamma$  (austenite with a face-centered cubic (fcc) structure)  $\rightarrow \varepsilon$  (martensite with a hexagonal close-packed (hcp) structure)  $\rightarrow \alpha'$  (martensite with a body-centered cubic (bcc) structure) phase transformation procedure. Thus,  $\varepsilon$ -martensite is generated as a precursor during the first stage of the transformation and is subsequently transformed into the  $\alpha'$  phase [9]. There are few cases that have traced this microstructural transformation on multiple scales, ranging from nanometers to several micrometers, during deformation in various loading and temperature environments.

Various traditional characterization methods, such as electron backscatter diffraction, XRD and transmission electron microscopy (TEM), are powerful tools for revealing the transformation mechanisms and selection principle for martensitic variants [10–15]. Due to the limited penetration of these techniques, it is very difficult to determine the exact transformation mechanisms inside bulk materials. Synchrotron-based microfocused beam X-ray diffraction ( $\mu$ XRD) is a powerful tool for measuring the local orientation and strain distribution inside individual grains of polycrystalline materials. Due to the high penetration depth of  $\mu$ XRD, the tested material can be tens of micrometers thick, which is a good simulation of bulk materials in their true serving conditions, rather than just the thin film or

surface tested by the traditional tools mentioned above. Furthermore, the beam size can be focused to  $0.5 \times 0.5 \,\mu\text{m}$ , offering high spatial resolution for measuring grains with large local lattice distortions [16–18].

In the present work,  $\mu XRD$  was used to measure the local orientation distribution inside individual martensitic plates and their parent grains in bulk polycrystalline 304 stainless steel during in situ uniaxial tensile loading. The experiment was conducted at a low temperature (210 K) just above the martensitic transformation temperature  $(M_s)$  to facilitate the presence of  $\varepsilon$ -martensite, a precursor in the transformation process [15]. The original lattice parameters of the  $\gamma$ ,  $\varepsilon$  and  $\alpha'$  phases in the 304SS used in study were  $a_{\gamma} = 0.3536 \text{ nm}, \quad a_{\varepsilon} = 0.2516 \text{ nm},$ this  $c_{\varepsilon} = 0.4064$  nm and  $a_{\alpha'} = 0.286$  nm. The Laue patterns of the  $\varepsilon$  and  $\alpha'$  martensitic variants revealed the transformation scenario, which is the key to understanding the deformation mechanisms. Our experimental results were in full agreement with the fcc-hcp-bcc step theory described previously [19]. The minimum strain work criterion [20,21] successfully explained the variant selection. Strain distributions inside martensitic plates were measured in this experiment; these verified the strain accommodation of martensitic plates. Synchrotron HE-XRD also confirmed the lattice strain fluctuations as a function of applied stress under in situ tension at 210 K, indicating that the stress in the transformed grain had relaxed and transferred into adjacent grains to some extent.

### 2. Material and experimental procedure

### 2.1. As-received material and sample preparation

The chemical composition of the as-received 304 stainless steel used in this study is shown in Table 1. The corresponding SFE is estimated to be  $\sim 15.5 \text{ mJ m}^{-2}$  according to previous literature [22]. Dog-bone-shaped tension specimens for HE-XRD, with a gage length of 7 mm and a width of 3 mm, were cut from the as-received steel sheet and polished with sandpaper to a final thickness of 1 mm. The dog-bone tension specimens for  $\mu$ XRD, with a gage length of 3 mm, were first pre-ground and polished into a thin plate, and then twin-jet electropolished to make a hole in the center of the sample, indicated by the red ellipse in Fig. 1(a).  $\mu$ XRD was performed near the hole edge, marked by the blue<sup>1</sup> square in Fig. 1(a). The thickness of the blue area was approximately 30-50 µm, indicating that the specimen was a couple of grains thick because the average grain size is approximately 20 µm. This area (blue square) was chosen for characterization for two reasons: (i) the thickness optimized the penetration ability of synchrotron X-rays and the simulation of bulk materials; and (ii) to spatially resolve the overlapping Laue diffraction

<sup>&</sup>lt;sup>1</sup> For interpretation of color in Fig. 1, the reader is referred to the web version of this article.

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