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Phase Transformation of Metastable Austenite in Steel during Nanoindentation

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1. Introduction

Recently, nanoindentation has been employed to probe the small-scale mechanical behavior of materials for a wide range of academic or engineering applications. The response of a material to the nanoindentation is usually represented as a form of the load-displacement curve. When metallic materials undergo irreversible permanent deformation, the discrete physical events, such as dislocation nucleation, dislocation source activation, phase transformation or mechanically-induced twinning, can be detected as discontinuities of displacement or load during nanoindentation. These can produce geometrical softening accompanied by a sudden displacement excursion during load-controlled nanoindentation, which referred to in the literature as a pop-in. In this study, phase transformation of metastable austenite to stress-induced ε martensite which causes pop-ins during nanoindentation of steel will be reported and discussed.

2. Experimental

The chemical composition of the high nitrogen TRIP steel used in this study was designed as Fe-0.02C-5.06Mn-0.19Si-0.23Ni-0.28N-20.08Cr (wt.%). With this composition, the stacking fault energy of austenite is approximately $15 \mathrm{mJ \cdot m^{-2}}$, which is known to have the initial deformation microstructure as ε martensite [1]. Sequential experiments were carried out, first using electron backscattered diffraction (EBSD) to map the phase and orientation distributions of the grains, followed by nanoindentation of individual austenite grains in the mapped region, then sectioning through an indent using focused ion beam (FIB) milling and finally transmission electron microscopy (TEM) to confirm the formation of ε martensite from austenite under the indent. In addition, the load-displacement curves of the metastable austenite phase were analyzed to identify signatures of phase transformation.

3. Results and Discussion

3.1 Deformed microstructures

Figure 1 shows the change of deformation microstructure of austenite as tensile strain increases. In the beginning, ε martensite formation is predominant deformation mode and they finally transform to α′ martensite as deformation proceeds. From an analysis of the diffraction patterns, ε and α′ turned out to have S-N and N-W orientation relationship with parent austenite, respectively.

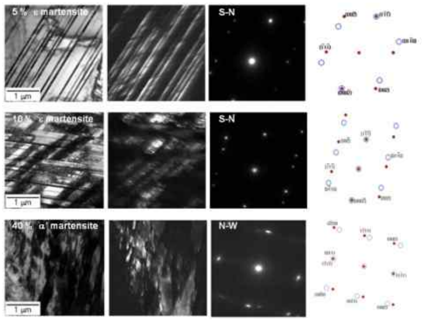


Fig. 1. Microstructure change in austenite as tensile strain increases.

3.2 Nanoindentaiton

Figure 2 gives the load-displacement curve obtained by nanoindentation of a metastable austenite grain. Stepwise pop-ins were measured in the early stage of deformation. On carefully polished metal surfaces the deformation before the first pop-in has been shown to be perfectly elastic (e.g. [2]). Before the first pop-in, the initial elastic portion of the loading curves can be fitted to the Hertzian elastic contact solution assuming that the indenter tip is spherical at shallow depths [2]. In the present case, the pop-ins in the early stage of plastic deformation may be related to ε martensite transformation because stress-induced ε martensite transformation is the predominant deformation mode in the early stage of plastic deformation. Moreover, stress-induced ε martensite forms in monopartial stackings [3], which causes the largest lattice shape change, geometrical softening is expected if a sufficient number of ε martensite bands form at the same time.

3.3 TEM works

Figure 3(a) shows a plan-view image of the cross-section sample, there is an obvious indent on the surface and deformed regions under the indent. From an analysis of the diffraction patterns, the region close to the indent (marked as A) consisted of two phases, transformed α' martensite and the parent austenite. Because region A corresponds to the highly stressed zone by nanoindentation [33,34], severe deformation must have been introduced in the region. Therefore, it is considered that in region A, austenite had already transformed to α' martensite by large amount of strain, which corresponds to microstructure of 40% tensile deformation in Figure 3.5.

Region B is about 900nm away from the center of the indent, where it is considered that less stress was applied than region A. Figure 3(b) shows an high resolution image of region B from $[\bar{1}10]_\gamma$ zone, there are many thin parallel bands lying in the same direction (marked as a dotted line in the figure) distributed all through the examined area. From analyses of this lattice image and the numerical diffractogram of the image (Figure 3(c)), this banded structure turned out to be a lamellar structure consisting of a mixture of ε and γ phases. Although thickness of single lamellae of each phase was only a few planes, this fine banded structure was observed through a wide range. A diffractogram obtained by fourier transformation of the high resolution image also presents the existence of both γ and ε phases, and the orientation relationship between them (N-W).

Possible displacement along indentation axis could easily be predicted by simple calculations based on the Schmid's law. The displacement along indentation axis for $(111)[11\bar{2}]$ slip system by formation of one ε martensite layer, which consists of two stacking faults, was calculated to be 0.16 nm. Considering the monopartial nature of stress-induced ε martensite, it can be easily imagined that if only 10~20 single ε martensite layers were made at the same moment, the amount of displacement along the indentation axis will be an order of several nanometers, which could be detected as pop-in. In the high resolution image in Figure 3.9, over twenty of thin ε martensite bands were already observed in such a small area of only about 20 nm x 20 nm. Therefore, these microstructural investigations strongly suggest that the pop-in behavior in the early stage of plastic deformation of austenite is closely related to the formation of ε martensite.

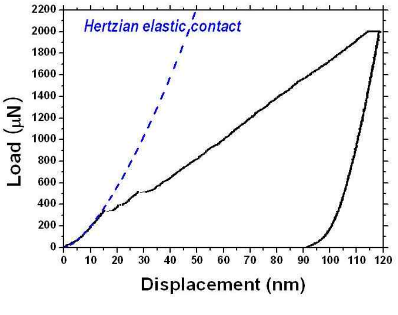
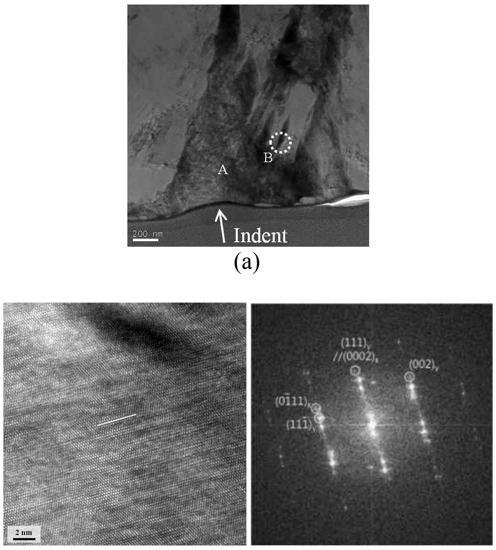


Fig. 2. Load-displacement curve obtained by nanoindentation of the metastable austenite grain.



(a)

(b) (c)

Fig. 3. (a) A plan-view image of the cross-section sample. (b) A high resolution image of region B (zone=$[\bar{1}10]_\gamma$). (c) A numerical diffractogram obtained by FFT of (b).

3. Conclusions

This study investigated the relationship between pop-in behavior of austenite in the early stage of nanoindentation and formation of ε martensite based on microstructural analyses. The load-displacement curve obtained from nanoindentation revealed stepwise pop-ins in the early stage of plastic deformation. From analyses of high resolution TEM images, a cluster of banded structure under the indent turned out a juxtaposition of (111) planes of γ austenite and (0001) planes of ε martensite. The calculation of displacement along indentation axis for $(111)[11\bar{2}]$ slip system by formation of ε martensite showed that geometrical softening can also occur by ε martensite formation when considering that the stress-induced ε martensite transformation is the predominant deformation mode in the early stage of plastic deformation and its monopartial nature as well. These microstructural investigations strongly suggest that the pop-in behavior in the early stage of plastic deformation of austenite is closely related to the formation of ε martensite.

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