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Martensitic phase transformation and pop-in in compression of austenitic steel nanoplates observed in situ by transmission electron microscopy

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ABSTRACT

To explore small-scale martensitic phase transformation and its relation to pop-ins observed in stress–strain curve, *in situ* compression experiments in transmission electron microscope were performed on austenitic steel nanoplates. Diffraction mode test revealed that the transformation indeed occurs at the nanoscale, but the pop-in seems not related with sudden microstructural change. Additional imaging mode test suggests that pop-ins may be caused by rapid formation of slip band and/or shearing-off of the nanoplate.

1. Introduction

Recent development of various nanomechanical testing techniques has continuously deepened the knowledge of small-scale metallurgical phenomena in structural steels and alloys. Among the techniques, nanoindentation has been the most popular for analyzing plastic deformation and strengthening behavior that is controlled by crystalline defects such as dislocations, grain boundaries, and precipitations [1–4]. During nanoindentation with spherical or rounded indenter, the load–displacement (P–h) curve often exhibits sudden increase in displacement (or displacement excursion) under load-control mode or sharp decrease in load (or load drop) under displacement-control mode. From a viewpoint of physical metallurgy, this phenomenon, so-called pop-in, has gathered numerous interests since it (especially, the first pop-in) may indicate elastic-to-plastic transition by homogeneous/inhomogeneous nucleation and/or propagation of dislocations (for example, see [5,6]).

In some materials, the pop-ins are considered as a clue for other phenomena rather than the dislocation activities. An interesting example can be found in the steels exhibiting a solid state phase transformation of metastable face-centered cubic austenite (γ) into base-centered tetragonal martensite (α') during deformation. In these materials, the nanoindentation pop-in is often thought to be closely related to the martensitic transformation [7–10]. This speculation seems appropriate since those steels exhibit continuous pop-ins (or serrations) during conventional tensile test of a bulk sample [11]. It is believed that the pop-ins of the bulk sample are caused by an instantaneous increase in shear strain of martensite phase and in turn an instantaneous increase in specimen length [11]. Because this strain-induced martensitic phase transformation can provide a good combination of high strength (by increasing strain hardening rate) and good ductility (by preventing the local necking) [12], its detailed mechanism has drawn both scientific and technological interests. Recently, research in the field has accelerated with the increasing interests in transformation-induced plasticity (TRIP) steels for automotive and abrasive applications [7,8,13]. Furnémont et al. [7], who examined nanohardness of each phase in a high silicon TRIP steel, reported a large pop-in during nanoindentation on austenite phase, and argued that the pop-in is corresponding to indentation-induced martensitic transformation. Very recently, Ahn et al. [8] and Misra et al. [9] reported a series of pop-ins in nanoindentation P–h curves of a modified TRIP steel and a type 301LN austenitic stainless steel, respectively. While the first pop-in is believed to be a clue for the dislocation-mediated plasticity onset, they proposed that the sequent pop-ins may be the results from martensitic transformation. However, since the previous studies were limited to the postmortem *ex situ* experiments, there has been no clear evidence for the direct relation between the martensitic transformation and the nanoscale pop-ins of the steels. Additionally, for a very small volume under stress, whether martensitic transformation occurs in the same manner as for a bulk sample or not is also somewhat controversial [14,15]. With this in mind, here we performed *in situ* compression test within a transmission electron microscope

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(TEM) for a better understanding of small-scale martensitic plastic transformation of an austenitic steel and its relation to pop-ins of stress–strain curve.

2. Experimental detail

Examined material is a metastable austenite steel having a composition of Fe–12Cr–5Mn–0.4C (in weight percent), that was developed as an erosion-resistant hardfacing alloy with a deformation-induced martensite surface layer. The specimen surface was mechanically polished with fine SiC paper of grit number 2000, and then electrolytic-polished using Lectropol-5 instrument (Struers, Westlake, OH) with a solution of 20% perchloric acid in ethanol, which was helpful to eliminate the surface layer of martensite (transformed during mechanical polishing). For confirming the occurrence of martensitic transformation in the bulk sample of the steel, high-load instrumented indentation test was performed using AIS2100 (Frontics Inc., Seoul, Korea) with a spherical indenter having a radius of 250 μm. The volume fraction of indentation-induced martensite phase was roughly estimated by a ferritescope (Feritscope MP30 (Fischer GmbH, Sindelfingen, Germany). In the ferritescope experiment, a low-frequency alternating magnetic field is generated around a cylindrical-shaped iron probe (5 mm diameter), and the change in the surrounding magnetic field due to the presence of the sample can be measured using a coil wound around the probe. From the magnetic permeability measurement of the sample, one can estimate the martensite content through the rule of mixtures in a way analogous to that for the ferrite content estimation [16].

The focused ion beam (FIB; Nova 200 NanoLab, FEI Co., Hillsboro, OR, USA) milling was used to fabricate nanoplates (also called nanoblades) having a rectangular contact area (with ~80 nm×~250 nm) and a height of ~450 nm; bulk plates with ~20 μm width, ~5 μm height, and ~3 μm thickness were extracted from the bulk, then attached on TEM grid using nano-manipulator, and finally milled into the nanoplates without breaking vacuum in the focused ion beam chamber. *In situ* TEM compression tests were carried out using a PicoIndenter (Hysitron, Minneapolis, MN, USA) inside a JEOL 2010F TEM (JEOL Ltd., Tokyo, Japan) under diffraction or bright-field imaging mode. With a diamond flat punch with a diameter of 1 μm, compression tests were carried out under displacement-control mode where load-drops can appear instead of displacement-jump under load-control mode. Each nanoplate was loaded up to the maximum displacement of 100 nm under a constant displacement rate of 0.5 nm/s with holds at the maximum load for 10 s.

3. Results and discussion

As a preliminary test to confirm the occurrence of the martensite phase transformation, high-load indentation experiments were performed to the maximum displacement of 150 μm and the change in the martensite volume fraction by the indentations was roughly estimated by a ferritescope, as shown in Fig. 1. The detection area of the ferritescope is a circle with a diameter of 5 mm (for 2-mm-depth), which is much larger than an indentation impression size. Thus, if indentation-induced martensitic transformation occurs, increasing the number of indentation in a given detection area will bring an increase in the measured martensite fraction. Here, we increased the number of indentation within the detection area from 0 (before indentation) to 9 (see the inset picture of Fig. 1). The estimated volume fraction of transformed martensite was increasing continuously with the number of indentation from ~2% to 18%, indicating that the transformation can take place macroscopically in the steel. Note that there is small martensite fraction in the initial state (~2%) that may be attributed to the surface martensite formed during mechanical polishing.

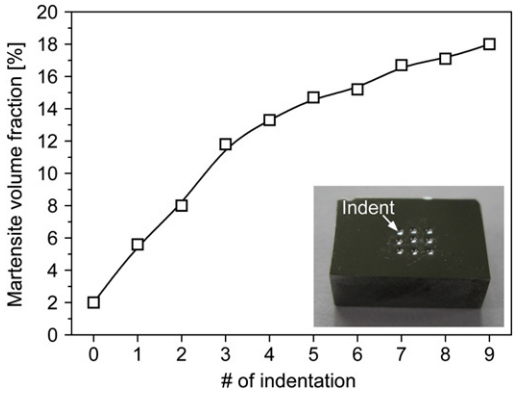

Fig. 1. Change in the estimated martensite volume fraction with increasing number of high-load indentation.

At first, *in situ* compression tests in TEM were performed under diffraction mode. Representative TEM image of the nanoplate taken before compression is shown in Fig. 2a. Fig. 2b and c (both of which are not from *in situ* tests) exhibit diffraction patterns with a zone axis of [111] martensite obtained before and after deformation, respectively. For clarifying the complex diffraction patterns, schematics (featuring closed and open symbol for austenite and martensite, respectively) are also provided in the figures. Most of austenite

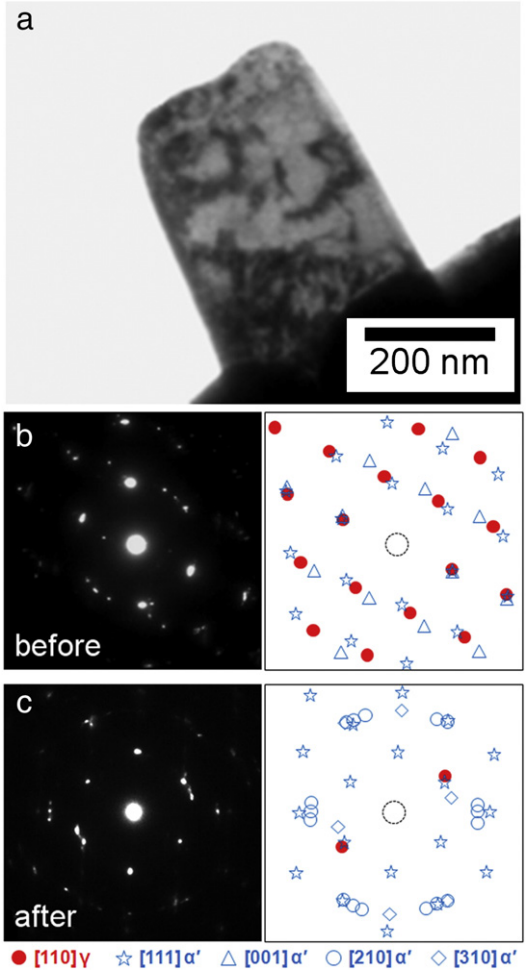

Fig. 2. Representative *ex situ* TEM images. (a) Bright-field image of the nanoplate before compression; (b)–(c) the diffraction patterns taken (b) before and (c) after compression with corresponding schematics.

spots disappeared after compression, which may imply that martensitic transformation also occurs in the nanoscale compression.

Fig. 3 shows the engineering stress–strain curve obtained from *in situ* compression test and the captured video frames (with *P*–*h* curve in the upper right side) corresponding to the points marked on the curve. Despite the existence of small taper in the nanoplate, engineering stress and strain in Fig. 3a were simply calculated as the load divided by initial contact area and the displacement divided by the initial height of the plate, respectively. To make certain contact between tip and plate, main test started at an elastic pre-load, and the zero-stress point was adjusted in consideration of both the linear slope of elastic loading curve and unloading curve, as shown in Fig. 3a. The softening-like behavior of the curve may be induced by slight buckling due to either plane-stress condition of the very thin nanoplate or misalignment between the plate top and loading axis. Note that similar behavior is not observed in Fig. 4 below. The yield

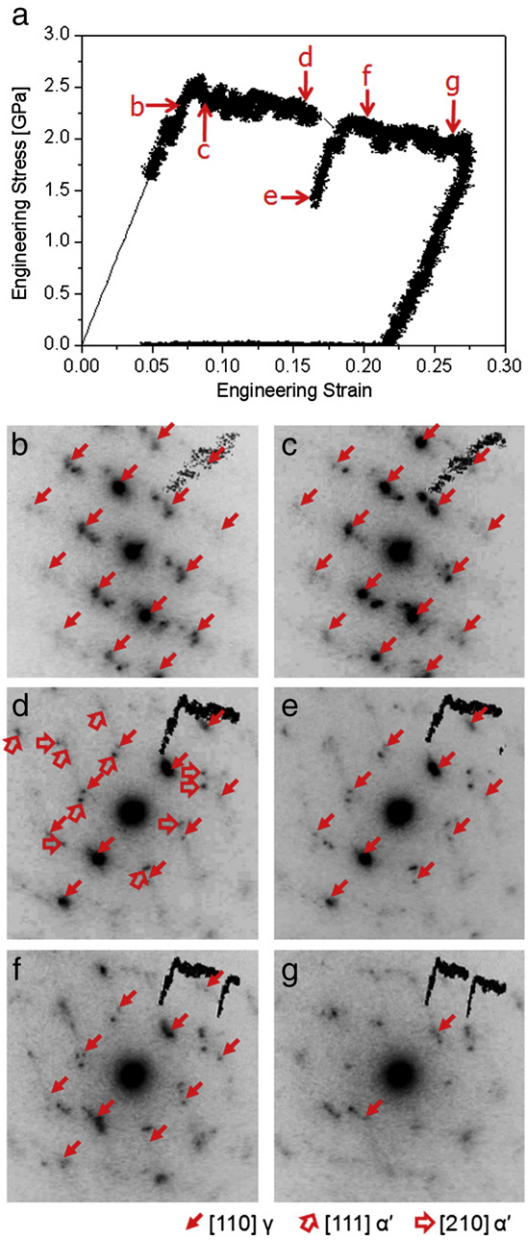

Fig. 3. Results from *in situ* TEM compression test under diffraction mode. (a) Engineering stress–strain curve; (b)–(g) the captured video frames exhibiting diffraction patterns for the points marked on the curve. Note that the martensite spots in (d) are also seen in (e)–(g), but not arrow-marked to avoid complexity.

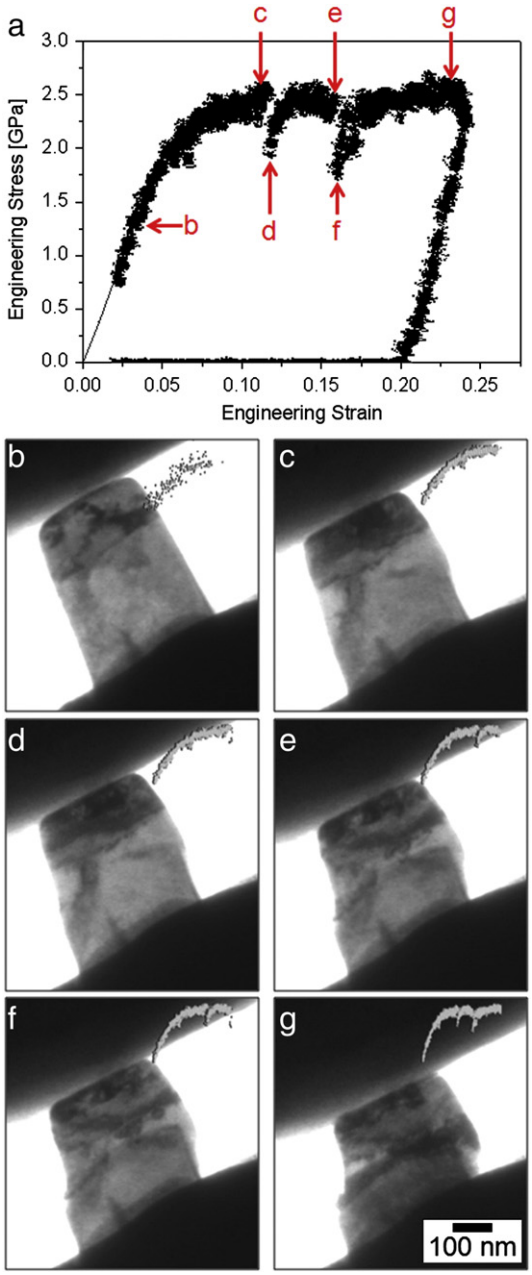

Fig. 4. Results from *in situ* TEM test under bright-field mode. (a) Engineering stress–strain curve; (b)–(g) the captured video frames showing bright-field images for the points marked on the curve.

strength of the nanoplate estimated from Fig. 3a is about 2.5 GPa that is 10 times higher than that of bulk counterpart (~250 MPa), which can be expected according to the rule of "smaller is stronger" in literature (for example, see [17,18]). The first important feature in the video frames is that the recognizable phase transformation began far after the onset of plastic deformation; i.e., the transformation required some amount of plastic strain. The new diffraction spots for martensite phase are evident at the point (d). Note that the diffraction spots for the martensite phase shown in (d) are also shown in (e)–(g) (though they are not arrow-marked to avoid complexity). Requiring some amount of plastic strain for initiation of martensitic transformation is inconsistent with the reported behavior of the transformation during conventional uniaxial tests of bulk sample. For example, Tao et al. [19], who performed tensile tests with *in situ* neutron diffraction measurements on a steel of Fe–10Cr–5Ni–8Mn–0.1C (in weight percent), observed the initiation of

martensitic transformation at the very beginning of the plasticity or when the applied stress reaches yield strength. As mentioned earlier, although the stress state underneath the indenter is totally different from uniaxial stress state, the maximum shear stress at the first pop-in during spherical indentation is often thought to be comparable to the shear yield strength under uniaxial loading [5,6]. Thus, the first pop-in phenomenon during a spherical indentation can be analyzed in a way somewhat analogous to the pop-in behavior during uniaxial loading. In this regard, the post-yield martensitic transformation in the present work is partially agreed with previous nanoindentation studies [7–9] in which it was suggested that the first pop-in in *P–h* curve is dislocation-controlled yielding, but subsequent pop-ins may correspond to martensitic transformation.

More important feature in Fig. 3 is the correlation between diffraction pattern change and pop-in (or discontinuous large load-drop) in the stress–strain curve. One may imagine that this load-drop is a direct evidence for the transformation in the same manner as serrations of bulk tensile tests are analyzed in previous works [11]. Interestingly, however, the diffraction patterns observed before and after the load-drop (i.e., (d) and (e)) are almost the same, and no clue for the microstructural transition was detected. During further loading, there is no appearance of new martensite spots, but intensity of diffraction spots (that is related with a volume fraction of a phase) for martensite increases while that for austenite continuously decreases. At point (g), most of the austenite spots disappeared and only two spots are remaining, which implies that martensitic transformation progressed continuously during deformation after pop-in. It is noteworthy that there are also small serrations (i.e., continuous small load-drops) in the curve of Fig. 3a. However, no close relation between each serration and the diffraction pattern change was found in analysis of video frames, implying that the serrations may be caused by dislocation slips or even by experimental noise [15].

An important question arising from the above results is “unless the pop-in is caused by the transformation, where does it come from?” To gain a clue for the answer, additional *in situ* TEM compression tests were conducted under bright-field imaging mode, as shown in Fig. 4. In the engineering stress–strain curve of Fig. 4a, two pop-ins are exhibited in plastic regime. The snapshots of Fig. 4 captured from live video suggest that the first pop-in between (c) and (d) may result from the rapid formation of large slip band and in turn observable slip step at the surface (see the upper right part of the plate in Fig. 4c). As deformation progresses, the dislocation structure dramatically changes, and subsequently the nanoplate is sheared off, which resulted in the second pop-in in the curve between (e) and (f).

It is constructive to compare our results with recent *in situ* TEM compression studies on martensitic transformations and related mechanical responses of structural metals. Ye et al. [15] investigated the small-scale martensitic transformation of NiTi shape memory alloy nanopillars through *in situ* TEM compression test. They reported the first pop-in at ~1 GPa may be corresponding to the transformation, but also proposed that the evidence of new diffraction spots is clearer at the second pop-in. Indeed, in the TEM diffraction image in Ref. [15], it is not easy to find new spots right after the first pop-in. Withey et al. [20], who performed *in situ* TEM tests on Ti–Nb–Ta–Zr–O alloy nanopillars, also observed a post-yield martensitic transformation (noticed by the appearance of new diffraction spots). They reported that, while the transformation in a high strength nanopillar was accompanied by a noticeable load-drop, there was no obvious relation between the pop-ins and the transformation in a low strength nanopillar. This may imply that the pop-ins are not necessary for martensitic transformation, which is in an agreement with our observation.

4. Conclusion

In the present study, we have performed *in situ* TEM compression tests on a metastable austenitic steel, for a better understanding of the small-scale martensitic plastic transformation and its relation to pop-ins in real-time. *In situ* test under diffraction mode revealed that martensitic transformation indeed occurs in the nanoscale compression, but the point of pop-in in the stress–strain curve seems not closely related with the transformation detected in the diffraction patterns, at least in the material examined here. Additional *in situ* TEM test under bright-field imaging mode suggests that the pop-ins may be the clue for the rapid formation of large slip band (and in turn observable slip step at the surface) and/or the shearing-off of the nanoplate.

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