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Variant selection during mechanically induced martensitic transformation of metastable austenite by nanoindentation

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Multiple pop-in events which indicate a gradual transformation from a metastable austenite grain to martensites in steel were detected during nanoindentation. It was observed by means of an automotive mapping technique with TEM that the partial volume of prior austenite had transformed into several martensite blocks with different variants. From a finite element calculation combined with phenomenological approach for martensitic transformation, it was confirmed that each variant corresponded to those for which the transformation strain effectively accommodates external stress.

Keywords: Martensitic phase transformation; Nanoindentation; Pop-in; Transmission electron microscopy (TEM); Metastable phases

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The special crystal orientation relationship (OR) [1–3] between the parent austenite (γ) and the transformed phase is observed when martensite (α') or bainite structures are formed by displacive transformation. For example, the Kurdjumov–Sachs (KS) OR, consisting of 24 equivalent variants expressed as $\{1\,1\,1\}_{\gamma}\|\{0\,1\,1\}_{\alpha'}$, $\langle 1\,0\,1\rangle_{\gamma}\|\langle 1\,1\,1\rangle_{\alpha'}$, has been reported by many researchers [4–6]. In principle, the probability of selecting a variant from among 24 equivalent variants is identical, but under certain circumstances, such as in the presence of external stress, specific variants can be preferred.

Martensitic transformation, which occurs under external stress, has long been studied [7–11]. Patel and Cohen [7] discussed the effect of mechanical work by external stress on the martensite starting temperature. Kundu and Bhadeshia [8,9] showed that possible orientations of transformed α' can be predicted by considering the interaction energy associated with the transformation. Recently, Perdahcıoğlu et al. [10] considered the mechanical driving force in simulation of mechanically induced martensitic transformation (MIMT) in stainless steel. However, there are few studies of MIMT under inhomogeneous external stress at a single-grain level.

The authors had suggested that the pop-in event observed on the load–displacement curve during nanoindentation of metastable austenite grain corresponds to the onset of MIMT [11]. In this paper, a multiple pop-in phenomenon, which is attributed to MIMT from metastable γ grain to several α' blocks during nanoindentation, was presented. The crystal OR between the two phases and variant selection during the transformation were quantitatively investigated by an automotive mapping technique with transmission electron microscope (TEM). The interaction energy between the applied stress and the transformation strain was evaluated by means of a finite element (FE) simulation and a phenomenological theory of martensitic transformation (PTMT).

TRIP steel with a chemical composition of Fe–0.08C–0.5Si–1Al–7Mn (wt.%) was used in the present work. A sequential procedure used here consisted of, the phase mapping of a polished surface using electron backscattered diffraction (EBSD), the nanoindentation of individual γ grain, the cutting out of the cross-section of the indented region using a focused ion beam, and the confirmation of the phase formation using a transmission electron microscope (TEM). Additionally, crystallographic orientation and phase mapping were performed by nanobeam diffraction in scanning mode using a TEM equipped with a NanoMEGAS ASTAR system. Other experimental details of processes such as specimen preparation, the equipment used, and the specific working conditions can be found in the earlier studies [12,13].

Figure 1 shows the nanoindentation load–displacement curve of the indented γ grain. The blue broken line

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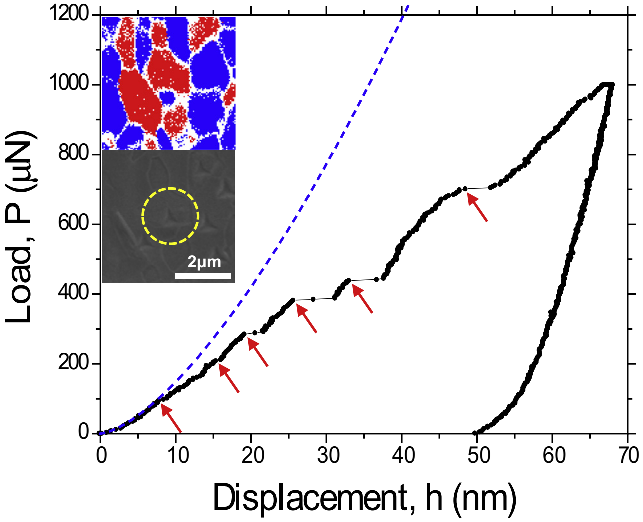

Figure 1. Nanoindentation load–displacement curve of the indented γ grain (yellow circle). The blue broken line represents the Hertzian elastic contact solution. Red arrows indicate the starting points of several pop-ins. The insets show a phase map (red: fcc γ, blue: bcc α′ or ferrite (α)) of the indented surface and a scanning electron microscope image after indentation. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

represents the Hertzian elastic contact solution [14], which was calculated with indenter tip radius of 400 nm determined from a calibration using standard fused silica. There are several discontinuous displacement bursts. The first pop-in event is likely the result of dislocation nucleation in the annealed austenite grain based on the deviation from the Hertzian solution. The other pop-ins might be caused by strain-induced phase transformation from the parent γ to the transformed α′. This type of pop-in event had been detected and described as resulting from geometrical softening due to the selection of a favorable martensite variant [12]. Therefore, the multiple pop-in implies that the prior γ grain is transformed gradually as indentation proceeds. Figure 2(a)–(c) respectively shows bright-field image, and phase and orientation maps in the normal direction of the cross-section of the indented region. The rectangular white broken line on Figure 2(a) corresponds to the frame of Figure 2(b) and (c). The black lines represent the grain boundary or the phase interface when the misorientation angle exceeds 15°. With a partial volume of untransformed γ, the rest of prior austenite grain was transformed to several α′ blocks, extended from the grain boundary or free surface to the phase interface between the remaining γ.

Seven blocks of α′ phase, M1–M7, with a clear phase interface with the remaining γ, were identified based on the high angle boundary as shown in Figure 2(c). Note that small blocks consisting of less than 20 pixels were excluded. In this study, the KS OR was considered adopting the sequence suggested by Takayama et al. [15]. The misorientation angles between the ideal KS OR and the experimental OR were calculated from the deviation matrix D^k, which is described as follows [16]:

$$D^k = M^k_{KS}(S^i g_A)(S^j g_M)^{-1} \qquad (1)$$

Here, M^k_{KS}, g_A, g_M, S^i and S^j are the matrices which represent the k-th variant of the KS OR, the γ orientation, the α′ orientation, and i-th and j-th symmetric operator in cubic system, respectively. The variant with the smallest misorientation angle θ was then determined. It can be written as follows:

$$\min_k \theta = \cos^{-1}\left(\frac{tr(D^k) - 1}{2}\right) \qquad (2)$$

The calculations above were performed for all the pixels within each defined α′ block, and the mean orientation of γ was used for g_A.

The determined KS OR variants and corresponding close-packed plane (CP) groups, Bain groups and mean misorientation angles for M1–M7 blocks are shown in Table 1. M1–M4, which belong to the same CP group, i.e., CP4, form a packet altering the Bain group between neighboring blocks. M1 consists of the V20/V23 variant pair, equivalent to V1/V4, in which the rotation axis and

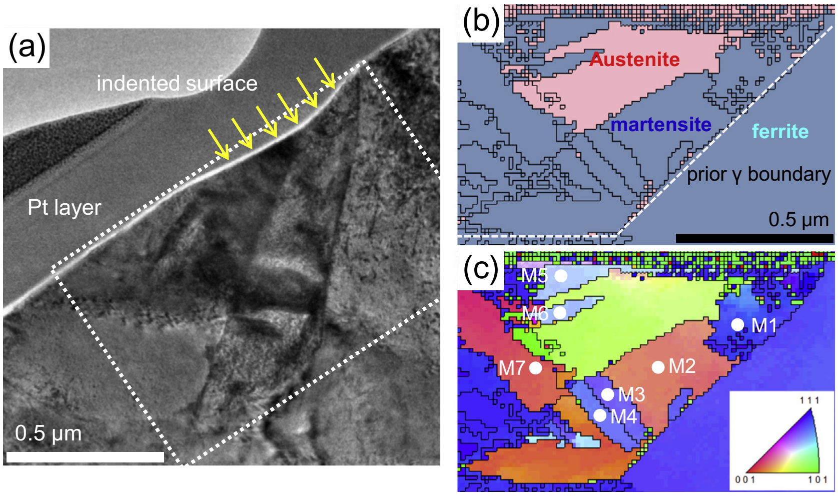

Figure 2. (a) Bright-field image, (b) phase map (pink: fcc γ, steel blue: bcc α′ or α) and (c) orientation map of the cross-section of the indented region from ASTAR in TEM. The black lines in (b) and (c) represent the grain boundary or phase interface when the misorientation angle exceeds 15°. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)