



# Structural and phase transformation in a TWIP steel subjected to high pressure torsion



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## ABSTRACT

We report on the changes in microstructure and phase composition in a high-Mn steel due to severe plastic deformation realized via high pressure torsion at different temperatures. Severe straining conducted at 20 °C and 300 °C allowed producing nanostructural states with a mean grain size less than 100 nm. Besides, straining at room temperature led to formation of strain-induced  $\epsilon$ -martensite while at 300 °C fully austenite state was produced. Despite the revealed differences both states demonstrated similar, notably increased level of hardness up to ~580 Hv.

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## 1. Introduction

Severe plastic deformation (SPD) proved to be a powerful tool for nanostructuring various metallic materials down to ultrafine grain (UFG) range, leading to significant improvement of their strength properties [1]. Basically, by variation of SPD processing parameters one could produce different nanostructured states (different by grain size, phase composition, grain boundary features and so on) thus providing an opportunity to manifest different combinations of the material properties [1]. This approach could be promising in manipulation of microstructure of TWIP steels which recently started to draw an increased attention of materials scientists and industrial partners due to their attractive combination of properties [2].

Modern industrial needs require increasing strength properties of steels, where SPD-processing could be of a great assistance. So far there are only a few publications [345] devoted to SPD processing of high-Mn TWIP steels. Timokhina et al. studied the effect of SPD by equal-channel angular pressing (ECAP) at different conditions (strain, route, and temperatures) and showed that ECAP is capable of producing various structural states depending on the processing parameters [4]. It was found that various dislocation substructures (parallel microbands, dislocation cells, sub-grains, stacking faults and so on) were formed as a function of ECAP parameters [4]. Interestingly, the formation of nano-twins inside micro-twins was identified that indicated an opportunity to

construct a new structural architecture of TWIP type steels for the new properties. At the same time ECAP seemed to be not able to produce truly UFG homogeneous states with high-angle grain boundaries so far. In Ref. [3] it was testified that high-pressure torsion (HPT) at room temperature enables producing homogeneous UFG state in this steel (with the hardness considerably exceeding those of cold-rolled samples). The TWIP effect was found to be diminished by exhausting of twinning activity at late deformation stages. The researchers also noted formation of a notable fraction of strain-induced martensite.

However, the transformation from austenite to martensite could decrease the ductility and fatigue properties in such materials so that its manifestation should rather be avoidable. Previously, on the example of an austenite stainless 316 steel it was shown that HPT at elevated temperature could allow getting rid of strain-induced martensite formation in the course of deformation and to produce fully austenite UFG 316 steel [6]. In the present comparative study, we focus on the application of HPT with room and elevated temperatures to examine the capability of the high strain SPD to produce UFG TWIP steels with different structural and phase parameters.

## 2. Experimental

A 0.6%C–18%Mn–2%Al TWIP steel was provided by the industrial partner (POSCO company [7]) in the form of a warm-rolled sheet. Before the SPD processing initial disc-shaped steel specimens were cut out of the sheet and annealed at 1200 °C for two hours and then water quenched. The specimens were

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nanostructured by HPT at room temperature and at 300 °C under applied the pressure of 6 GPa. The number of HPT turns consisted  $n=10$  providing shear strain in the middle of a disc radius of about 300 as estimated by  $\gamma = 2\pi Rn/l$ . The HPT-produced UFG specimens had the form of discs 20 mm in diameter and 0.8 mm in thickness. All subsequent studies were carried out for the area corresponding to the middle of the radius of a specimen. The microstructure was studied by transmission electron microscopy (TEM) using a JEOL 2100 microscope operated at 200 kV, the length of the camera was 0.3 m for diffraction patterns. TEM foils were prepared by twin jet electro-polishing with 10% of perchloric acid in butanol at 25 °C and with a voltage of 50 V. Observations were made in both bright and dark field imaging modes, and selected area electron diffraction (SAED) patterns were collected using an aperture of  $1 \mu\text{m}^2$ . The average grain size was calculated using TEM dark field images with the help of mean intersect lengths over 100 grains for each state. X-ray analysis was performed using Rigaku D/MAX-2500/PC diffractometer at 40 kV 200 mA with  $\text{CuK}_\alpha$  irradiation. The microhardness measurements were carried out using a microhardness tester Micromet with a load of 0.1 kg per 10 s.

### 3. Results and discussion

A TWIP steel in an initial state had a microstructure characterized by large density of twins inside large grains  $150 \pm 10 \mu\text{m}$  in diameter (Fig. 1) and its phase composition was estimated to be fully austenite.

After HPT at 20 °C we observed severely refined nanostructured states (Fig. 2a) with a mean grain size of about 50 nm with a few elongated grains  $\sim 100$  nm in a bigger dimension. HPT at 300 °C temperatures resulted in formation of a UFG state with a mean grain size of 90 nm (Fig. 2b). Both states were formed by predominantly high angle grain boundaries as followed from SAED (Fig. 2a, b) showing diffraction spots uniformly distributed over ring position. Another difference between the two UFG states was found to be in phase composition – spots corresponding to martensite were detected for the UFG state produced by HPT at room temperature and no such spots were observed for the case of 300 °C HPT one. No twins inherited from initial quenched state were found in the nanostructured states. However, some nano-sized grains in both UFG states contained secondary nanotwins with 10–15 nm twin spacing, this feature was much more typical for the 300 °C HPT TWIP steel (Fig. 3). Thus, in both states the

TWIP effect was restricted as stated in [3] for the room temperature HPT-processed TWIP steel.

The results of XRD phase analysis (Fig. 4) of both UFG states were in agreement with the TEM observations. The room temperature HPT steel exhibited notable  $\epsilon$ -martensite peaks which indicated the presence of a considerable volume fraction of this phase (exceeding 5–7% considering sensitivity of an XRD technique). This observation is in a good agreement with the result for the room temperature HPT TWIP steel reported in Ref. [3]. At the same time X-ray profile line 300 °C HPT TWIP steel consisted of only austenite X-ray peaks, that testified to the fact that at elevated temperature both TWIP and phase transformation effects were suppressed. Generally, it is the stacking fault energy value which is a key parameter for manifestation of deformation mechanisms (including phase transformation) in Mn–Al–Si steels [8]. In our case different phase composition of UFG TWIP steels produced at different temperatures can be understood in terms of stress/deformation–temperature–transformation diagrams which show that the martensite formation is suppressed when reaching  $M_d$  – the maximum temperature at which transformation by deformation occurs, see, for instance [9].

Microhardness measurements have shown that both nanostructured states demonstrate outstanding increase in hardness – from  $275 \pm 20$  Hv for the initial quenched state to  $580 \pm 45$  Hv and  $570 \pm 40$  Hv for 20 °C HPT and 300 °C HPT TWIP steels, correspondingly. These values exceed not only those (360 Hv) for cold-rolled TWIP steel [10] but also 450 Hv measured for Fe–24Mn–3Al–2Si–1Ni–0.06C TWIP steel processed by HPT at room temperature [3]. In Ref. [11] it was shown that combination of repetitive cold rolling and annealing one could increase hardness of 0.3C–17Mn–1.5Al steel up to 5600 MPa ( $\approx 560$  Hv) due to decreasing twin spacing down to 20 nm, however, the yield stress measured was estimated to consist less than 1000 MPa. Authors of [12] obtained similar level of the yield stress thanks to partially recrystallized submicron-grained structure in Fe–31Mn–3Al–3Si TWIP steel as well as the yield stress measured in Ref. [13] for TWIP steel with ultrafine elongated grain structure. In the latter case the chemical composition of the investigated steel was very close to that used in the present study (Fe–17Mn–0.6C). Assuming that the HPT-produced steels in our case had equiaxed UFG structure one would estimate the yield stress  $\sigma_{0.2}$  corresponding to hardness Hv from the simplified Tabor's law as  $\sigma_{0.2} \approx Hv/3$ . The calculated  $\sigma_{0.2}$  value should at least exceed  $\sim 1700$  MPa for the hardness equal to 570–580 Hv. Thus, one may note that the UFG TWIP steels obtained here demonstrated the highest strength properties ever reported for this type of alloys.

High hardness of the 300 °C HPT steel comparable to room temperature one requires additional discussion. One would expect that smaller grain size estimated for 20 °C HPT steel would yield considerably higher strength values. However both states exhibit almost the same level of hardness. Earlier similar effect was shown for the UFG austenite stainless 316 steel [6] produced by HPT at different temperatures where it was explained by formation of grain boundary segregations at elevated HPT temperature. The segregations could suppress emission of dislocations from grain boundaries thus increasing stress required for the strain propagation [14]. Renk et al. [15] explained the effect of increasing strength on annealing of room temperature HPT-processed 316 steel to strengthening by grain boundaries which got straightened by recovery. In any case the observed phenomena might be connected to fine features of grain boundaries and will be a subject of further detailed investigation.

### 4. Summary

For the first time we report the results of microstructural and

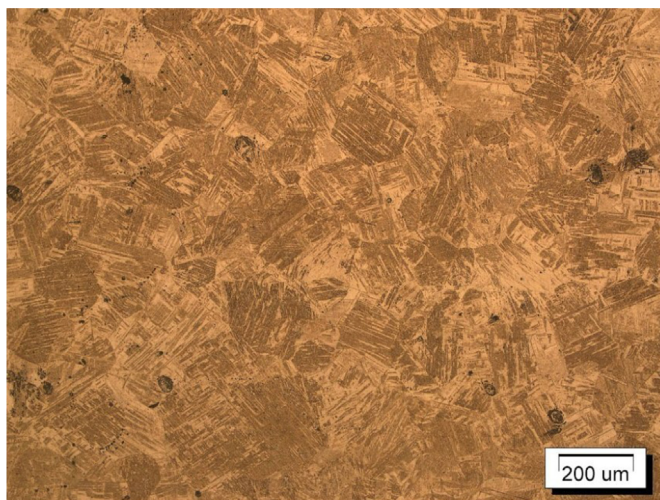


Fig. 1. Microstructure of an initial TWIP steel after quenching procedure as obtained by optical microscopy.

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