1. Introduction

Grain refinement for engineering materials has been studied for a long time to increase strength without deteriorating ductility and/or toughness. In recent reports concerning ultra-fine grained materials, the shape of stress–strain curve has been found to change with decreasing of grain size not only in steel but also in aluminum, as is illustrated in Fig. 1. As seen, the yield strength increases while elongation decreases with decreasing of grain size. The uniform elongation disappears for a single phase alloy with grain size of less than 1.0 μm. In such a material, necking takes place during the propagation of Lüders band (Type II) or at the same time (Type I). This is ascribed to the loss of work-hardening in ultrafine grained materials. Hence, several challenges have been made to overcome this drawback. For example, a material with bimodal distribution of grain size was reported to exhibit a relatively good combination of strength and uniform elongation. Another method is to mix the hard phase particles to the ultrafine grained matrix. If the second phase particle is metastable austenite in steel, it would be more effective to obtain higher ductility due to TRIP phenomenon by deformation induced martensitic transformation. In this study, an ultrafine-grained ferrite–austenite low alloy steel was made by using thermomechanically controlled process and the tensile behavior was studied by means of in situ neutron diffraction.

2. Experimental Procedures

The chemical compositions of a steel (nano-TRIP) used in this study were 1.57Mn–1.41Si–1.94Ni–0.017Nb–0.391C–0.004P in mass%. The hot-rolled steel plate with 4.5 mm in thickness was cooled down to room temperature and the surface layer was mechanically removed to be 3.5 mm thick sheet. The sheet was heated to 773 K and rolled to 1mm thick sheet. Then the rolled sheet was annealed at 998 K for 120 s and quenched in a salt bath followed by holding at 673 K for 300 s. Finally the sheet was cooled to room temperature in air. The microstructure of the steel was observed by FESEM with Hitachi S-4300. Tensile specimens with a gauge portion of 1 mm thickness by 5 mm width by 60 mm length were prepared by spark cutting followed by mechanical polishing. In situ neutron diffraction during tensile test was performed using an engineering diffractometer, “TAKUMI” at MLF/J-PARC. The experimental view at TAKUMI was presented in Fig. 2, where the tensile tester was installed at 45 degrees with respect to the incident neutron beam. The information in the axial direction was obtained at south detector (90 degrees scattering) while the transverse at north detector (90 degree scattering). Tensile load was increased in a step by step manner and neutron diffraction profiles in the axial and transverse directions were collected at each step with a time-of-flight method by the two detectors simultaneously. The measuring time for each step was 120 s which was shorter in case of the previous study done at Sirius/...
KENS because of higher intensity of incident neutron beam (5KW at KENS versus 22KW at J-PARC in 2008 (the final target is 1MW)).

3. Experimental Results and Discussion

3.1. Microstructure and Tensile Properties

SEM microstructure was shown in Fig. 3. The averaged grain size of the nano-TRIP steel was 0.4 µm that was much finer than that of commercially available TRIP steels: for example 7 µm for 590 T and 3 µm 780 T. Austenite volume fraction determined by X-ray diffraction was 19.5%. In the present nano-TRIP steel, the microstructure was composed of ferrite and austenite similarly to the case of a high alloyed 17Ni–0.2C steel. This kind of ultrafine grained two-phase steel was firstly studied to realize an extremely excellent strength and ductility balance by Miller for high alloyed steel. The present steel is therefore characterized by low concentration of alloying elements. The carbon concentration would be reduced by employing more sophisticated TMCP and hence the present steel is a model material to understand the mechanism how higher uniform elongation can be obtained in such ultrafine grained duplex steel (change from Type I or II to III in Fig. 1).

The 0.2% proof stress of 771 MPa, ultimate tensile strength of 1326 MPa and total elongation of 23% were obtained for this nano-TRIP steel. Nominal stress–strain curve was exhibited in Fig. 4 in which the stresses kept for neutron diffraction measurements were indicated by arrows with numbers. As is observed in Fig. 4, the nano-TRIP steel showed higher yield and higher ultimate tensile strength than commercially available TRIP steel, 780 T. Although the uniform elongation is comparable between the two, the local elongation or reduction in area was smaller in the nano-TRIP steel.

To be noted here is the deformation behavior in the beginning of deformation. A plateau region appeared in stress–strain curve similarly to many ultrafine grained materials. The aspect of surface of a specimen interruptedly unloaded during the Lüders deformation was shown in Fig. 5. It was found that stress-induced martensitic transformation took place in the Lüders band. This is very similar to the case of ausformed Ni–C austenite steel. TRIP steel was firstly invented by Zackey group. They studied TRIP phenomenon for ausformed steels and discontinuous yielding.
accompanying Lüders deformation was commonly observed. The similar tensile behavior with Lüders band was reported in ultrafine grained austenitic steel.\(^\text{15}\) Therefore, Lüders deformation accompanied by martensitic transformation appears in ultrafine grained or plastically deformed metastable austenite steels. Some fraction of Lüders strain must come from transformation strain. In such a high strength steel, the possibility of the onset of martensitic transformation must be equal throughout of the parallel portion of a tensile specimen, so that accidental transformation at a certain place may accelerate further transformation due to local stress concentration leading to the formation of Lüders band. In case of ultrafine grained materials, necking occurs after the onset of Lüders deformation resulting in little uniform elongation. However, metastable austenite transforms to martensite during Lüders deformation to prevent the occurrence of necking. Hence, it is speculated the intrusion of hard martensite contributes to higher work-hardening, but no direct evidence has been presented yet.

### 3.2. \textit{In situ} Neutron Diffraction During Tensile Deformation

Neutron diffraction profiles were obtained in the axial and transverse directions during tensile deformation. The overall profiles in the axial direction were summarized in Fig. 6. As seen, the intensity of austenite peaks decreases, and peak shifts towards wider spacing side are visible, with increasing the applied stress. The intensity of austenite peak is found to decrease particularly after the Lüders deformation. Figure 7 presents (211) reflection profile for ferrite and martensite obtained at the applied stress of 1185 MPa. As seen in inserted fitting curves for ferrite and martensite, a single-peak fitting was not adequate and hence a double-peak fitting was employed for the profiles obtained after the Lüders deformation. To do such double-peak fitting, firstly the mean carbon concentration of austenite \((X_c \text{ (mass\%)}\)) was estimated from the lattice constant of austenite \((a_c)\) and that of ferrite \((a_f)\) determined from neutron diffraction profile obtained before loading (no martensite phase in the beginning).\(^\text{16}\)

\[
X_c \text{ (mass\%)} = (a_f - 3.572 \times a_d / 2.8664) / 0.033 \quad \text{(1)}
\]

As results, the carbon concentration was estimated to be 1.125 mass\%. Then, the \(c/a\) of bct martensite was postulated from Eq. \(2\)\(^\text{15}\) to be 1.05, which was assumed not to change during tensile deformation.

\[
c/a = 1.000 + 0.045 \times X_c \text{ (mass\%)} \quad \text{.................(2)}
\]

Probably, carbon concentration must differ from grain to grain and martensitic transformation depends on the stability of austenite. Hence, as a first approximation, the obtained diffraction profiles were fitted using Gaussian function for austenite and two Gaussian curves with fixed \(c/a\) for martensite as demonstrated in Fig. 7. Similar separation technique for martensite and ferrite or ferrite and bainite diffraction profiles was successfully made for synchrotron X-ray diffraction of a dual phase steel by Cong \textit{et al.}\(^\text{18}\) and Jia \textit{et al.}\(^\text{19}\) respectively. They have revealed the stress partitioning behavior between the two constituent phases using such a profile analysis.

### 3.3. Strain Partitioning Behavior During Tensile Deformation

The lattice strains were calculated assuming \((hkl)\) spacing measured before loading as “stress free spacing” for austenite and ferrite. For martensite, the stress-free spacing was taken from literature\(^\text{16}\) taking carbon concentration into account. The change in lattice strain in the retained austenite was shown in Fig. 8. A drastic change was observed at the yield strength because of the occurrence of Lüders band. After the yield strength (LYS) in Fig. 8(b), family grains oriented to [111] and [220] with respect to the tensile direction show a decrease in the lattice strain while [200] continuously increases. In case of commercially available TRIP-DP steels like 490 T and 780 T with moderate grain size\(^\text{9,20,21}\) all the lattice strains in austenite has been verified to increase after the onset of plastic deformation (continuous yielding) by \textit{in situ} neutron diffraction during tensile deformation, indicating that carbon-enriched austenite is stronger than the ferrite matrix. Zaefferer \textit{et al.}\(^\text{22}\) studied heterogeneous plastic deformation behavior by means of EBSD showing detailed local information which is almost consistent with the neutron experimental results. Because neutron diffraction data present bulky averaged information, stress partitioning is much useful to discuss...
macroscopic stress–strain behavior. In particular, Tomota et al.\textsuperscript{16} have proposed three transition points in nominal stress–strain curve; P1, P2 and P3, for several TRIP-DP steels. At P1, ferrite phase starts to deform plastically but the retained austenite is still only elastic. Then, the onset of deformation induced martensitic transformation is determined by P2. Finally at P3, the retained austenite starts to deform plastically. The stresses of P1 to P3 are dependent on chemical compositions and microstructure. The results of the present ultra-fine grained steel were quite different from these relatively coarse grained TRIP steels.

The ferrite lattice strains were plotted as a function of the applied stress in Fig. 8(a). As seen, differently from the coarse grained TRIP steels, the lattice strains for ferrite do not show clear decrease after LYS. Because ferrite grains were extremely fine in this steel, the strength of ferrite seems to become comparable with that of carbon-enriched austenite. In Fig. 8, stress balance in a whole microstructure does not hold as long as taking ferrite and austenite into consideration; the role of martensite must be crucial.

It should also be noted that the absolute value of transverse strain of ferrite decreases drastically just after LYS (see Fig. 8(a)). During the Lüders deformation, plastic flow occurs in ferrite grains and martensitic transformation takes place in austenite grains. Hence, the compressive strain along the tensile direction and transverse strain along the transverse direction must be yielded in preferentially deformed ferrite grains, corresponding to the internal stresses caused by plastic misfit strains. The compressive strain related to the applied tensile stress (i.e., Poisson effect) and this compressive internal strain are speculated to be canceled out leading to the drastic change in the ferrite transverse strain observed in Fig. 8(a). On the other hand, martensitic transformation and plastic flow occur in austenite grains, and the resulting internal stress must be complex. It is therefore necessary to make a suitable modeling for the detailed interpretation of Fig. 8.

The lattice strains of martensite determined by using the double-peak fitting are presented in Fig. 9. It is evidently found that the lattice strain in martensite increases rapidly after the Lüders deformation indicating that martensite bears higher stress. The behavior of lattice strain is dependent on crystal orientation, i.e., [hkl] and hence intergranular stresses would be overlapped onto the phase stress. To make clear the stress partitioning among the constituent phases, the phase strain of each constituent was estimated by simply averaging three different (hkl) lattice strains, and the results are presented in Fig. 10. Here, martensite is revealed to be the strongest, the next is ferrite and austenite is weakest. Hence, the average lattice strain of austenite is dropped once around the given lower yielding point. This suggests that austenite is softer than ferrite. Therefore, it is concluded that the origin of high work-hardening after Lüders deformation is caused by load sharing mechanism, where strong martensite plays an important role. Differently from the coarse-grained TRIP-DP steels, the strength of the retained austenite is softer than ferrite and thereby the deformation induced martensite is really needed to obtain uniform elongation in the ultrafine grained TRIP-DP steel. Al-
though the Rietveld refinement is more appropriate to evaluate phase strains, it is not applicable at J-PARC yet (the software called Z-Rietveld will be used in near future).

To confirm the load sharing mechanism, the residual strains after unloading were plotted in Fig. 11. As is observed, the compressive strain remains in ferrite and austenite, while tensile in martensite. If a component phase is softer, the compressive stress should be remained after tensile deformation. The strength order estimated from Fig. 11 is austenite, ferrite and martensite, being consistent with the above estimation.

Another evidence on stress partitioning among ferrite, austenite and martensite was reported by Jacques et al.\textsuperscript{23)} They obtained similar conclusion by using a digital image correction method on SEM micrograph, by which distribution of plastic strain on the surface of a tension specimen was depicted revealing heterogeneous plastic flow in the constituent phases.

3.4. Crystal Orientation Dependence of Transformation During Tensile Deformation

The change in $(hkl)$ peak intensity of austenite with deformation was computed and the results for $(111), (110)$ and $(200)$ were plotted in Fig. 12. It is confirmed from the decrease of diffraction intensities that martensite transformation really occurred during Lüders deformation. The trend of $(111)$ diffraction is different from that of $(200)$. As was discussed above, $[111]$ oriented grains are plastically softer while $[200]$ harder. Martensite transformation takes place obviously in $[200]$ oriented family grains, probably because of stress partitioning during tensile deformation. That is, $[200]$ oriented grains bear higher stresses with progress of plastic deformation and hence martensite was preferentially induced. Similar results were reported by Narui et al.\textsuperscript{10} for commercial TRIP-DP steels, 590 T and 780 T.\textsuperscript{10} Oliver et al. reported similar results for a metastable austenitic steel.\textsuperscript{24} Earlier transformation in $[200]$ oriented grains is estimated to be caused mainly by high intergranular stress and probably suitable transformation shear. Taking a closer look into Fig. 12, the $[111]$ diffraction intensity is found to increase a little after the Lüders deformation. On the other hand, the $[220]$ diffraction intensity decreases in spite of plastically softer grains. It must stem from deformation texture evolution in austenite with plastic flow.\textsuperscript{25,26} According to Fig. 10 of Ref. 26) for tensile deformation of single austenitic steels, the diffraction intensity of $[111]$ and $[200]$ in the tensile direction increases with increasing of tensile strain while that of $[220]$ decreases, due to crystal rotation caused by slip deformation. Hence, the austenite $[hkl]$ intensity in Fig. 12 is dependent on not only martensitic transformation but also deformation texture evolution in austenite.

A micromechanics modeling for TRIP steels was examined by Tsuchida and Tomota\textsuperscript{27)} and developed further by other investigators.\textsuperscript{28–30} To figure out a general overview of deformation mechanism and then to use such a modeling to steel design, flow stresses of constituent phases and transformation strain and kinetics have to be taken into consideration. Neutron diffraction experiments are very useful for verification of the established model and its development. The Lüders deformation appeared in the present steel has, however, not been challenged to estimate by modeling yet.

4. Conclusions

Tensile behavior of a nano-TRIP steels with submicron grain size was firstly studied under in situ neutron diffraction. The origin of high tensile strength with sufficient uniform elongation was discussed in this paper, leading to the following conclusions.

(1) Tensile strength of 1326 MPa and total elongation of 23% was achieved in the nano-TRIP steel with ultrafine grained structure that was made by thermo-mechanically controlled process.

(2) In spite of submicron sized structure, the nano-TRIP steel showed high strength and high work-hardening resulting in a sufficient amount of uniform elongation.

(3) In the beginning of tensile deformation, Lüders...
band appeared similarly to other ultrafine grained materials. The stress induced martensite was found to be generated in the band.

(4) The double-peak fitting method was applied to martensite plus ferrite overlapped peaks and thus the stress partitioning behavior among ferrite, martensite and the retained austenite could be tracked during tensile deformation. The obtained results reveal that the work-hardening after Lüders deformation in the nano-TRIP steel is caused by load sharing of deformation induced martensite.

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REFERENCES