1. Introduction

In the last decade a lot of efforts have been made to realize grain-ultrarefinement down to 1 μm in steels because it is only possible way to achieve high strength steels without sacrificing toughness. To date, some reduction in ferrite grain size has been commercially achieved through thermomechanical controlled processing (TMCP) which involves controlled rolling and subsequent accelerated cooling. However, conventional TMCP soon encounters a ferrite grain size limit of approximately 5 μm, regardless of the level of retained strain introduced into the austenite. Recently, though, a number of thermomechanical processes have been developed at the laboratory scale to produce ferrite grain sizes of 1–3 μm or less; ranging from extreme thermal and deformation cycles to more typical thermomechanical practices. Dynamic strain-induced transformation is one example that has received significant attention. Dynamic transformation includes transformation of austenite to ferrite during, rather than after, deformation (Fig. 1). This requires large strains, typically 2–5, being applied to the austenite in a critical temperature range. The critical deformation temperature is usually at, or just above, the Ar3 (i.e. the empirical austenite to ferrite transformation temperature). It was also demonstrated that dynamic transformation can occur if deformation is applied in the austenite metastable temperature range, i.e. between Ar3 and Ae3 (i.e. the equilibrium austenite to ferrite transformation temperature). More recently, it was shown that warm deformation at 570°C of extremely supercooled austenite (i.e. well below the Ar3) can form ultrafine ferrite as small as 1 μm. This is a temperature that bainite would usually form in the absence of deformation. The process appears to require a large supercooling, warm deformation and, most probably, dynamic transformation, although the underlying mechanism of such ultrarefinement is still under debate.

Some research groups employed a 70Ni–Fe (mass%) austenitic alloy*1 to investigate the deformed austenite structure. They showed relatively highly misoriented and dense dislocation substructures, so-called microbands, formed into austenite when the deformation was applied at a temperature close to Ar3 (Fig. 1). It was also reported that dislocation substructures were changed from dislocation cells to microbands with decreasing the deformation temperature in the unrecrystallized austenite field. The misorientation of the microbands was found to be averagely higher than the dislocation cells. The high potential and dense microbands work as effective nucleation sites for
austenite-to-ferrite transformation, so that ferrite nucleation rate increases with eventual formation of ultrafine grains. This explanation appeared to be enough to explain the underlying mechanism of grain-ultrarefinement through dynamic transformation. However, this correlation between dislocation substructure in austenite and final ferrite grain size explains only a static aspect of dynamic transformation mechanism and physical meaning of ‘dynamic’ transformation has not been clear yet.

Figure 2 is a schematic illustration to explain the difference between (a) static transformation and (b) dynamic transformation. In a case of static transformation, transformation starts after completion of deformation. Therefore, transformed product undergoes only pre-transformation deformation. In contrast, in a case of dynamic transformation, transformation takes place during, rather than after, deformation. Thus transformed products undergo not only pre-transformation deformation, but also post-transformation deformation. To understand the feature of dynamic transformation the effect of the post-transformation deformation and external stress on microstructural development should be, therefore, examined in detail. In other words, the effect of deformation in austenite/ferrite two-phase region on both kinetics and crystallography of transformed ferrite must be studied extensively.

Here, it is not clear yet whether which phase, austenite or ferrite, is more preferentially deformed plastically in high temperature austenite/ferrite two-phase region. If ferrite is always softer than austenite, ferrite would be more preferentially plastically deformed than austenite in the two-phase region and eventually might be dynamically recrystallized. Meanwhile, if austenite is more preferentially deformed, (dynamic) transformation would be likely enhanced at the lattice defects introduced into the austenite. Up to now ferrite is believed to be always softer than austenite, if they are individual. However, it is unclear yet that if ferrite is embedded in austenite, how they are plastically deformed at elevated temperature.

With particular attention to an effect of post-transformation deformation on kinetics and crystallography of transformed ferrite, this paper aims to deepen insight into the mechanism of dynamic strain-induced ferrite transformation. An advanced in-situ neutron diffraction experiment will be demonstrated to understand microstructural evolution in high temperature two-phase region.

2. Principle of Grain Refinement through Transformation

The essence of grain refinement through transformation is to satisfy the following four points: (1) increase of nucleation sites, (2) promoting potential of nucleation sites, (3) increasing the transformation driving force and (4) suppression of grain growth. Possible ways to increase nucleation sites are by refining initial austenite grains, introducing intragranular lattice defects and/or using precipitates/inclusions as nucleation sites. To increase potential of nucleation sites, low angle grain boundaries should be transformed to high angle grain boundaries and/or misorientation of dislocation substructures should be increased. Then supercooling is required to activate these nucleation sites. Finally, grain growth should be suppressed as much as possible even though nucleation rate increases by isothermally holding specimens at low temperature, using pinning effect by a second phase and/or using soft/hard impingement. These points can be expressed in the following equation:

\[ d_a = 0.91 \left( \frac{V}{I} \right)^{1/4} \]

where \( d_a \), \( V \) and \( I \) mean ferrite grain size, growth rate of
ferrite grain and nucleation rate of ferrite grain, respectively. This equation was deduced by assuming homogeneous nucleation. According to this equation, the ratio of $V/I$ must be as small as possible to refine ferrite grain size. Here $I$ is given as

$$I = \beta^* N_v Z \exp(-\Delta g*/kT)$$

where $\beta^*$ is frequency factor as expressed as $N_0 \exp(-Q_D/kT)$. $N_0$ is related to the number of atoms at embryo surface and lattice vibration. $Q_D$ is diffusion coefficient. $N_v$ is the number of nucleation site and $\Delta g^*$ is driving force. Based on this equation, to enhance nucleation rate of ferrite it is necessary to increase the number of nucleation sites in austenite and promote driving force, namely supercooling degree.

Because dynamic transformation is introduced by deformation in supercooled austenite, both nucleation sites and driving force increase simultaneously, which eventually allow the formation of ultrafine ferrite grains in general.

3. Dynamic Transformation

3.1. Effect of Deformation in Two Phase Region

Neutron diffraction spectrums during thermomechanical treatment processing were measured using ENGIN-X at Rutherford Appleton Laboratory, ISIS. Here a 0.2C–2Mn–(0.03Nb) steel (mass%) was employed with an initial microstructure of martensite was austenitized at 900°C and subsequently held isothermally at 640 or 680°C to stimulate partial austenite-to-ferrite transformation (Figs. 4(a)–4(c)). Then, it was deformed at strain rate 0.1/s at a given temperature, followed by isothermal holding (Fig. 4(d)). Ferrite volume fraction just before deformation was controlled by changing the pre-deformation isothermal holding period. By comparing before and after deformation, it was found...
that austenite-to-ferrite transformation was promoted by the deformation (Figs. 4(c), 4(d)). More quantitative evaluation is demonstrated in Fig. 5 showing a change of peak integrated intensity with deformation temperature and ferrite volume fraction. As seen in Fig. 5(a), deformation in the two phase region caused a sudden drop of intensity of all austenite diffraction peaks measured in this study. On the other hand, as seen in Fig. 5(b) where measurement was carried out in the axial direction, the deformation resulted in an increase of two ferrite diffraction peaks of three peaks measured and a decrease of one ferrite peak. Meanwhile, in the transverse direction (Fig. 5(c)), the peak intensity of ferrite changes with deformation in a different way. This result suggests that deformation in the two phase region introduces plastic strain not only into austenite enhancing austenite-to-ferrite transformation, but also into ferrite where deformation-induced texture is developed. Furthermore, according to volume fraction analysis based on Fig. 5, ferrite volume fraction*2 increased more as deformed at 640°C than at 680°C (Fig. 6). This result suggests that the plastic strain was concentrated more into austenite at lower temperature even in the two phase region.

This tendency can also be observed in the corresponding strain–stress curve (Fig. 7). With increasing the pre-deformation isothermal holding period (i.e. increasing ferrite volume fraction), the flow stress decreases as deformed at 680°C, but it increases as deformed at 640°C. This result indicates that deformation at higher and lower temperature in the two phase region introduces plastic strain preferentially into ferrite and austenite, respectively, which is in good agreement with neutron diffraction results. When the plastic strain is introduced preferentially into ferrite, it is assumed that dynamic recrystallization eventually takes place and the final microstructure formed through dynamic recrystallization of ferrite. In contrast, when plastic strain is partitioned preferentially into austenite, the final microstructure should mainly be formed by transformation of ferrite.

In an actual dynamic transformation event both dynamic transformation and subsequent dynamic recrystallization might proceed concurrently. However, the present results imply that dynamic transformation becomes more dominant process with lowering deformation temperature in the two phase region rather than dynamic recrystallization.

**3.2. Effect of Deformation in Low Temperature Austenite Region**

To understand transformation behavior in more detail, when the plastic deformation is partitioned preferentially into austenite, in-situ neutron diffraction experiments (Fig. 8) were additionally carried out at the dedicated high-reso-

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*Volume fraction was deduced by Rietveld refinement using GSAS software taking all diffraction peaks measured into consideration.

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Fig. 6. Effect of deformation on ferrite transformation evolution for Nb-added steel at (a) 640°C and (b) 680°C.11

Fig. 7. Effects of temperature and ferrite volume fraction on flow stress of Nb-added low alloy steel: (a) 640°C; (b) 680°C; (c) flow stress comparison between two temperatures.

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The current results can be summarized as follows. When the deformation is applied at relatively low temperature in the two phase region, the dynamic transformation takes place and plastic strain is introduced into austenite preferentially and repeatedly. Then, ferrite transformation is eventually accelerated. In addition, transformed ferrite might be partially deformed and subsequently recrystallized dynamically during further deformation.

### 3.3. Crystallography

When neighboring ferrite nuclei have the same orientation they will be coalesced and coarsened (Fig. 10(a)) even though nucleation rate is high. Thus, it should be revealed how deformation in the two phase region (corresponding to post-transformation deformation) affects austenite/ferrite orientation relationship. To understand the features of dynamic austenite-to-ferrite transformation in steels, effects of post-transformation deformation on the orientation relationship between fcc-matrix and bcc-precipitates was examined in a Ni–43mass%Cr alloy by means of electron backscatter diffraction (EBSD) in a high-resolution field emission gun scanning electron microscope (JEOL JSM-7000F).

The deviation angles of intergranular precipitates from the Kurdjumov–Sachs orientation relationship (KS

![Fig. 8. Effect of prestrain on ferrite transformation kinetics.](image)

The open marks denote the measured ferrite volume fraction by neutron diffraction. The close marks represent the ferrite volume fraction obtained by microstructure observation, which is a direct proof to confirm the reliability of neutron diffraction method.

![Fig. 9. Effect of double hit deformation on ferrite transformation kinetics for Nb-free steel.](image)

![Fig. 10. Comparison of variant selection between (a) static and (b) dynamic transformation.](image)
O.R.) were compared among undeformed, pre-transformation deformed and post-transformation deformed specimens. Post-transformation deformation was found to increase the deviation angles up to 13 degrees (Fig. 11). This can be explained through the accumulation of strain in fcc-matrix at the fcc/bcc interphase boundaries causing local lattice rotation in fcc-matrix. The increased orientation distribution of austenite phase leads neighboring ferrite grains to have a different orientation, because newly formed ferrite grains attempt to have the KS O.R. with respect to already locally rotated adjacent matrix (Fig. 10(b)). This weakened variant selection results in suppression of grain coalescence in dynamic transformation.

3.4. Static Transformation

As described previously, the most important point for ferrite grain refinement is to densely introduce high potential lattice defects into austenite. Despite of advantages of dynamic transformation such as repeated introduction of strain into austenite and distribution of ferrite grain orientation, it does not seem meaningful for grain refinement whether transformation mode is dynamic or static. For example, two ‘static’ transformation routes enabling ultra-grain-refinement will be explained below.

The first static transformation route is that high quench hardenable steels were deformed well below the $\text{Ar}_3$ temperature without inducing transformation, followed by reheating to the ferrite transformation region.$^{14}$ The ultimate aim of this investigation was to provide insight into the relative contributions of deformation temperature, transformation temperature and other thermomechanical parameters to microstructure refinement through static transformation of ferrite. The steels used were 0.2C–2Mn–(0.03Nb) steels, which are characterized by high quench hardenability. This enables to deform austenite at low temperature (well below $\text{Ar}_3$) without any dynamic transformation of ferrite. The samples were austenitized at 1 000°C for 300 s, followed by cooling to the given deformation temperature at a cooling rate of 50°C/s and immediately subjected to compression of up to 70% reduction at a strain rate of $5 \text{s}^{-1}$ at 570°C. Deformed specimens were subsequently reheated to 750°C at a heating rate of 30°C/s and held for 3 s, followed by cooling at 0.1°C/s (Fig. 12). This reheating process can produce homogeneous 1.5 μm ferrite grains (Fig. 13). Because in this process ferrite transformation mainly takes place during final cooling, this result suggests that deformation in low temperature austenite region is the most important factor for obtaining ultrafine ferrite grains.

The second static transformation route, which realizes ferrite grain ultrarefinement has been recently reported by
Miyata et al. 22) This process is characterized by shortening the interval of multi-pass rolling as well as increasing post-rolling cooling rate to suppress the recovery of dislocation substructures in austenite. In this case only static transformation is expected to occur because deformation is applied above \( \Delta e_3 \). This super short interval multi-pass rolling led to the formation of about 1 \( \mu \text{m} \) ferrite grains in a 0.15C-0.75Mn steel (mass%). They also demonstrated that transformation is likely to proceed statically by comparing the transformed ferrite texture with the deformed austenite texture simulated using 70Ni–Fe alloy (mass%).

4. Effective Strain Introduction

Some efforts have been made to reduce the critical strain for obtaining ultrafine ferrite grains. 20,22–24) One is to refine initial austenite grain size through pining effect using unresolved precipitates and dynamic recrystallization. 23) The refinement of initial austenite grains is closely related to solidification process and a study of solidification microstructural refinement has been, therefore, in progress. The other is processing approach of multi-axial deformation 25) as well as super short interval multi-pass rolling. 22)

Nowadays commercial production rolling facility which enables the production of 2–3 \( \mu \text{m} \) ferrite grains operates where all knowledge for effective strain introduction such as short interval multi-pass rolling, post-rolling rapid cooling, interpass cooling and complicated strain path are reflected. 25)

5. Conclusive Remarks

With particular attention to mechanism of ferrite grain-ultrarefinement though dynamic transformation, related experimental results have been reviewed. Both dynamic and static transformation result in ferrite grain-ultrarefinement, if high potential lattice defects are densely introduced into austenite. However, the following two points are still open for question.

- Why the dynamically transformed ferrite is equiaxed and contains low dislocation density?
- Why the grain size obtained through dynamic transformation is always around 1 \( \mu \text{m} \)?

An idea has been proposed to answer these questions that dynamically transformed ferrite undergoes subsequently dynamical recrystallization and the ferrite grain size eventually reduces down to 1 \( \mu \text{m} \) corresponding to Zener–Hollomon parameter. 26) Another possible explanation for this is that when deformation is conducted in supercooled austenite region the spacing of introduced microbands is less than 1 \( \mu \text{m} \) and their misorientation is relatively large. Ferrite grains nucleated at the dense and high potential nucleation sites for short period cannot grow over 1 \( \mu \text{m} \) in size due to three-dimensional hard impingement. In particular more plastic strain is likely partitioned into austenite, rather than ferrite, with lowering deformation temperature, so that dislocation density in ferrite becomes less. Further study is required to answer these two mysteries. For this subject in-situ neutron diffraction with higher beam intensity such as Japan Proton Accelerator Research Complex (J-PARC) is promisingly useful to detect microstructural evolution in real time besides detailed microstructural examination.

Acknowledgement

Grateful acknowledgement is given to Prof. P. D. Hodgson and Dr. H. Beladi (Deakin University, Australia), Prof. J. W. Morris (University of California, Berkeley, United States), Prof. J. J. Jonas (McGill University, Canada), Super Metal Project members (NEDO/JRCM), Ultra-Steel Project members (National Institute for Materials Science), Proteus Project members (NEDO/JRCC) for the discussion on dynamic transformation. The authors would like to acknowledge to Dr. Y. Z. Bao (Central Iron and Steel Research Institute, China), Mr. M. S. Koo (Ibaraki University), Dr. E. C. Oliver (ISIS, Rutherford Appleton Laboratory, U.K.), Dr. P. Lukás and Mr. V. Davydov (Nuclear Physics Institute, Czech Republic) in particular for their considerable support in performing neutron diffraction experiment.

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