Microstructural evolution of Ti-added interstitial free steel in high strain deformation by hot torsion

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Abstract. The dynamically evolved microstructure under high strain deformation condition does still have many debatable aspects, particularly in the case of easy-recovery metals like bcc-iron. In this research, microstructural evolution in high strain deformation by hot torsion of Ti-added interstitial free (IF) steel was systematically investigated. Torsion specimens were deformed up to an equivalent strain of ∼ 7 at different temperatures (650 ºC - 850 ºC) and strain rates (0.01 s⁻¹ - 1.0 s⁻¹), i.e., under various values of the Zener-Hollomon (Z) parameter. Immediately after the deformation, samples were water-quenched and microstructures were investigated by electron backscattering diffraction (EBSD) measurements and electron channelling contrast imaging (ECCI). Flow stress-strain curves of the IF steel under various deformation conditions showed typical flow curves of high stacking fault energy metals at low Z values, i.e., a peak stress followed by slight softening. On the other hand, under the high-Z deformation conditions, the specimens showed a larger stress drop after a certain amount of deformation. EBSD-based quantitative analysis was used to study the microstructural transition between high and low Z values. At low Z values, the occurrence of strain induced boundary migration (SIBM) as an initiation of dynamic recrystallization (DRX) was clearly observed. On the other hand, at high Z values, grain subdivision phenomena led to very fine and elongated structures.

1. Introduction
Dynamic recrystallization (DRX) is a type of microstructural evolution which leads to grain refinement of metals during hot deformation, especially those under low Zener-Hollomon (Z) parameter conditions. The Z parameter is expressed as $Z = \dot{\varepsilon} \exp(Q/RT)$, where $\dot{\varepsilon}$ is the strain rate, Q is the activation energy of deformation, R is the gas constant and T is the absolute temperature. Unlike metals with a low/medium stacking fault energy in which DRX easily happens, easy recovery metals with a high stacking fault energy including ferritic iron with a bcc structure do not show typical necklace-shape DRX morphologies along grain boundaries [1]. Although it is generally accepted that dynamic recovery (DRV) plays the main role in the dynamic restoration process of ferrite in this case, evidence of DRX phenomena happening in IF steel at low Z values has been also confirmed by some researchers [2,3].

On the other hand, surprisingly, it has been observed that grain refinement can also be achieved in medium and low temperature deformation or deformation under high Z condition by employing sufficient strains. The refined microstructure has been well explained by grain subdivision phenomena,
which is mostly the case in severe plastic deformation (SPD) processes. During straining, initial grains are subdivided by deformation-induced boundaries into smaller domains with different operative slip systems as well as other fine micro-volumes so-called cell blocks and cells. All deformation-induced boundaries between these fragmented areas can be categorized into two groups; i.e., incidental dislocation boundaries (IDBs) and geometrically necessary boundaries (GNBs). With further straining, the misorientation angle across dislocation boundaries (especially across GNBs) tends to increase but the boundary spacing decreases [4-6].

Since different deformation mechanisms within a wide range of Z values seem to lead to grain refinement, the aim of the current research is to systematically study the microstructural evolution of highly strained IF steel at different deformation temperatures and strain rates. To do so, hot torsion deformation was employed to realize continuous deformation under precisely controlled deformation conditions.

2. Experimental
A Ti-added IF steel with a chemical composition of 0.002 C, 0.01 Si, 0.12 Mn, 0.003 P, 0.003 S, 0.034 Ti, 0.028 Al, and 0.002 N (wt%) was used in this study. The material was received as square bars (15 mm × 15 mm) with 300 mm-length cut from a hot-rolled/air-cooled plate with a fully recrystallized structure. Torsion specimens with a 4 mm gauge length and 8 mm gauge diameter (figure 1) were cut off from the as-received bars in a way that the specimen axis was parallel to the rolling direction (RD).

Hot torsion tests were carried out at temperatures of 650 ºC, 750 ºC and 850 ºC and strain rates of 0.01 s⁻¹, 0.1 s⁻¹, and 1 s⁻¹ using a torsion machine equipped with a high-frequency induction heating system (Thermecmastor-TS). Under an Argon (Ar) atmosphere, the torsion specimens were heated up to a designed temperature below the bcc-to-fcc phase transformation at a heating rate of 2 ºCs⁻¹ using an induction heating system. Then the samples were held at those temperatures for 5 minutes for homogenizing and subsequently deformed up to an equivalent strain of ~ 7. Additionally, torsion-deformations at lower strain levels were carried out to study the microstructural evolution of IF steel during the hot deformation. It should be noted that torsion deformation provides different strains and strain rates even within one specimen, both increasing from the center to the surface of the torsion specimen. For both the equivalent strain and strain rate mentioned in this research, the specimen surface was taken as the reference position for calculation. Immediately after the torsion deformation, samples were water-quenched within less than 1 second to stop any microstructural change related to post-dynamic restoration.

The microstructure was investigated using electron backscattering diffraction (EBSD) measurements and electron channelling contrast imaging (ECCI) carried out in field emission scanning electron microscopes (FE/SEM- JEOL JSM 7100F and 7800F, respectively). The microstructure of the deformed gauge part was studied on a tangential section beneath the specimen surface (~ 0.9R; R is the radius of gauge part). For EBSD observations, electro-polishing in a 10%HClO₄-90%CH₃COOH solution was employed after mechanical grinding the surfaces. A different surface preparation method was used for
the channelling contrast imaging; i.e., mechanical grinding followed by mechanical polishing using 3 µm and 1 µm diamond paste and final polishing by a 0.04 µm colloidal silica suspension.

3. Results and discussion

3.1. Flow stress curves

Torque-rotation data obtained by the torsion test was converted into true shear stress-strain data by employing Fields and Backofen analysis [7] and subsequently von Mises criteria was used to attain equivalent stress-strain data [8]. Equations (1) and (2) show the relationships of equivalent strain (\(\varepsilon\))-rotation number (N) and equivalent stress (\(\sigma\))-torque (\(\Gamma\)), respectively, where R is the radius of the torsion specimen, L is the gauge length, n is the strain hardening coefficient (~ 0; near the peak and steady state condition) and m is the strain rate sensitivity (ranged from 0.1 at 650 ºC to 0.2 at 850 ºC in this study).

\[
\varepsilon = \frac{2\pi R}{\sqrt{3} L} N \tag{1}
\]

\[
\sigma = \frac{\sqrt{3} \Gamma}{2\pi R^3} (3 + n + m) \tag{2}
\]

Hot-torsion flow curves of the IF steel under different deformation conditions are depicted in figure 2(a). As is seen, they show typical flow curves of easy-recovery metals; i.e., a peak stress (\(\sigma_p\)) followed by slight softening before reaching a steady state stress. However large stress drops after the peak stress are observed for the deformation at 650 ºC. In addition, it seems that the highest flow stress curve associated with the highest Z value (650 ºC/1.0 s\(^{-1}\)) has not yet achieved a steady state condition even at a strain around 7. It is also noteworthy in hot torsion deformation of the IF steel in this study, that no matter under what condition a specimen was deformed, low stress levels (< 100 MPa) were recorded for all cases.

Based on the data derived from figure 2(a), peak stress and peak strain (\(\varepsilon_p\)) as characteristic parameters of the flow curves were measured. The dependence of these parameters on Z values is illustrated in logarithmic scales in figure 2(b). For calculating Z parameter, the activation energy of self-

![Figure 2](image-url)
diffusion in bcc-iron (\(\sim 250 \text{kJmol}^{-1}\)) was used as the activation energy (Q) of hot deformation. As is seen in figure 2(b), for both peak stress and peak strain, the dependence is well satisfied with power-law relationships on data. A power-law type relationship is generally the case of low flow stress levels in hot deformation [9].

3.2. Microstructures in high strain

Figure 3(a) and 3(b) show EBSD grain boundary maps of the deformed IF steel under two extreme conditions in the current study; i.e. the highest temperature/lowest strain rate (850 °C/0.01 s\(^{-1}\)) and the lowest temperature/highest strain rate (650 °C/1.0 s\(^{-1}\)), which correspond to the lowest and highest Z values, respectively. Between these two extremes, the refinement process was enhanced by decreasing the temperature and increasing the strain rate in a way that the range of mean linear intercept length for high angle boundaries (\(\theta > 15^\circ\)) changed from \(\sim 35 \mu \text{m}\) (at 850 °C/0.01 s\(^{-1}\)) to \(\sim 1.5 \mu \text{m}\) (at 650 °C/1.0 s\(^{-1}\)). In general, it was observed that the structural refinement of the IF steel did not occur homogeneously which was previously reported by Tsuji et al. [3]. In other words, the deformed microstructure typically contained some large elongated grains with many low angle boundary segments (\(2^\circ < \theta < 15^\circ\)) inside, as well as relatively smaller elongated grains and newly developed-equiaxed grains.

Moreover, the fraction of high angle boundaries (\(F_{HABs}\)) and misorientation angle (\(\theta\)) measured for all the deformation conditions are depicted versus the Z parameter in figure 4. According to this figure, at least two evolution regimes can be found for the deformation conditions in this study. Firstly, the specimens highly deformed at 850 °C show no Z-dependence of \(F_{HABs}\) and \(\theta\) by varying strain rates (0.01 s\(^{-1}\), 0.1 s\(^{-1}\), and 1.0 s\(^{-1}\)), although microstructural refinement still occurs with increasing strain rates. The values of \(F_{HABs}\) and \(\theta\) under these high temperature-deformation conditions are scattered around 0.4 and 19°, respectively. Furthermore, another trend is observed for the specimens highly deformed at 750 °C and 650 °C. Within this region, \(F_{HABs}\) and \(\theta\) clearly increase with increasing Z parameter or decreasing temperature/increasing strain rate; i.e., \(F_{HABs}/\theta\) increase from \(\sim 0.3/15^\circ\) (at 750 °C/0.01 s\(^{-1}\)) to \(\sim 0.7/30^\circ\) (at 650 °C/1.0 s\(^{-1}\)). This tendency was also found to be consistent with the microstructural observation. The microstructure of the specimen deformed under the lowest Z value condition in the region 2 of figure 4 (750 °C/0.01 s\(^{-1}\)) typically showed elongated grains with many subgrains inside. However, the specimen deformed under the highest Z value condition in this region (650 °C/1.0 s\(^{-1}\)) contained many fine grains with a smaller amount of detectable subgrains (\(2^\circ < \theta < 15^\circ\)) inside.

It is believed that the change in microstructural evolution in the region 2 suggests the occurrence of more effective grain subdivision phenomenon while in the region 1 with lower Z values, different deformation mechanism must be dominant. To prove this point, nevertheless, not only the highly

Figure 3. EBSD grain boundary maps of the hot torsion-deformed IF steel (a) at the lowest Z value (850 °C/0.01 s\(^{-1}\)) and (b) the highest Z value (650 °C/1.0 s\(^{-1}\)) in the current study. Red lines and black lines represent low angle boundaries (\(2^\circ < \theta < 15^\circ\)) and high angle boundaries (\(\theta > 15^\circ\)), respectively. Data with misorientation angles below \(2^\circ\) were eliminated to prevent possible errors caused by the limitation on angular resolution of EBSD measurements.
deformed microstructures under steady state conditions are necessary but also microstructural evolution at different strain levels (before reaching steady state condition) must be investigated. This topic is discussed in the section 3.3.

3.3. Effect of strain on microstructural evolution

To study how the microstructure evolves from the initial microstructure during torsion-deformation, the specimens were gradually deformed at different strain levels especially within the region until the flow stress reached a steady state condition. For this purpose, three deformation conditions with a constant strain rate (0.01 s\(^{-1}\)) were chosen; i.e., 850 °C/0.01 s\(^{-1}\), 750 °C/0.01 s\(^{-1}\), and 650 °C/0.01 s\(^{-1}\) which hereafter are referred to as “low-Z”, “medium-Z” and “high-Z” conditions, respectively. The results are discussed in the following sections 3.3.1 and 3.3.2.

3.3.1. Low-Z condition (850 °C/0.01 s\(^{-1}\)). Figure 5(a) and 5(b) show an EBSD inverse pole figure map and channelling contrast image of the water-quenched specimen deformed up to strain \(\varepsilon \approx 0.8\). Since the peak strain measured in this deformation condition is about 0.4, the strain \(\varepsilon \approx 0.8\) belongs to the post-peak region and is within the softening stage of flow stress curve until steady-state. It is seen that the microstructure shows elongated grains with relatively well-developed equiaxed subgrains inside. Additionally there exist very few small equiaxed grains which can be considered as newly-formed ones. It is also considered that many of those elongated high angle boundaries belong to the pre-existing grain boundaries at this strain level.

Furthermore, strong evidence for the phenomenon of so-called strain induced boundary migration (SIBM) [10] was found during straining before and after the peak stress. SIBM or bulging of pre-existing boundaries into adjacent substructures, shown by black arrows in figure 5(a), is a sign of the initiation of dynamic recrystallization and it is believed that many of those pre-existing grain boundaries in figure 5(a) play an important role in the restoration process at this stage. It should be noted that the boundary migration was clearer at the earlier stages of deformation while at the higher strain levels (\(\varepsilon > 1\)), a larger number of new equiaxed grains were observed in the microstructures.

3.3.2. Medium-Z (750 °C/0.01 s\(^{-1}\)) and high-Z (650 °C/0.01 s\(^{-1}\)) conditions. Microstructural evolution under the medium-Z and high-Z deformation conditions was also examined by gradual straining in torsion. It was observed that the microstructures, especially under the high-Z condition, adapted a different evolution regime from that which occurred under the low-Z condition (850 °C/0.01 s\(^{-1}\)). Figures

![Figure 4. The dependence of high angle boundary fraction \(F_{\text{HABs}}\) and misorientation angle \(\theta\) on Zener-Hollomon parameter.](image)
5(c) and 5(e) display EBSD inverse pole figure maps of the samples deformed up to a strain ~ 1.1 under a medium-Z condition and that deformed to ~ 1.8 under a high-Z condition. Similar to the low-Z case, these strain levels also belong to a softening stage of the flow curve after the peak stress.

Figure 5. EBSD inverse pole figure maps (a, c, e) and channelling contrast images (b, d, f) of the IF steel deformed within softening stages; (a) and (b) low Z condition-850 °C/0.01 s⁻¹, (c) and (d) medium Z condition-750 °C/0.01 s⁻¹, (e) and (f) high Z condition-650 °C/0.01 s⁻¹. Black lines in the inverse pole figure maps show high angle boundaries (θ>15°).
Similar to the low-Z deformation condition, the microstructure after the peak of the medium-Z specimen in figure 5(c) shows elongated grains as well as a few small equiaxed grains (shown by white arrows). However, the substructures of the IF steel deformed under these two deformation conditions are completely different. The channelling contrast image of the medium-Z specimen in figure 5(d) shows the presence of dense subgrain structures resulting in a high fraction of low angle boundaries. This can be the reason why the specimen highly deformed at 750 °C/0.01 s\(^{-1}\) showed the lowest \(F_{\text{HABs}}\) (figure 4) among all the deformation conditions. Further microstructural observation at the early stages of deformation (before the peak stress) revealed that these fine subgrains originated from a type of substructures characterized by in-grain band patterns.

Moreover, figure 5(e) exemplifies a water-quenched microstructure in the middle of the softening region of the specimen deformed under a high-Z condition. The microstructure shows a significant difference from that of the medium-Z condition although dense substructures also appear under the high-Z condition (figure 5(f)). Unlike the medium-Z case, it is seen (figure 5(e)) that many new high angle boundary segments start to appear within the softening stage of the high-Z specimen. This event is seemingly associated with the noticeable stress drop in the flow curves of the specimens deformed at 650 °C (figure 2(a)). By decreasing the deformation temperature to 650 °C, numerous amounts of new high angle boundary segments were observed within the matrix where strong banding patterns or so-called deformation bands existed. By further straining under this condition, it was found that deformation bands with in-grain lamellar structures were gradually replaced by newly-developed grains and completely disappeared nearly at the end of softening stage (\(\varepsilon \sim 5\)).

As a rule of thumb, boundaries with a higher misorientation angle can provide higher migration mobility [11]. Based on this fact, it is reasonable to suggest that many high angle boundary segments previously formed within deformation bands can migrate and bulge out into new grains. This is similar to the case for static recrystallization of cold rolled and annealed IF steel where nucleation at an early stage of recrystallization was reported to occur on two intersecting sets of shear bands [12]. The band intersections which can provide new boundary segments with higher misorientation angles were also observed in this study at the early deformation stages (\(\varepsilon \sim 0.6\)) of high-Z specimen and became more frequent by further straining. It should be noted that in the medium-Z specimen, straining did not lead to significant production of high angle boundary segments within the band patterns.

4. Conclusions
Hot torsion deformation was used to study the microstructural evolution in a Ti-added IF steel in high strain deformation (\(\varepsilon \sim 7\)) at different temperatures (650 °C, 750 °C and 850 °C) and strain rates (0.01 s\(^{-1}\), 0.1 s\(^{-1}\), and 1.0 s\(^{-1}\)). The results are summarized as follows;

Hot-torsion flow curves of the IF steel showed a peak stress followed by slight softening although a larger stress drop after the peak stress was observed under deformation conditions at 650 °C. The dependence of both peak stress and peak strain on the Z parameter followed power-law type relationships.

Decreasing the temperature and increasing the strain rate enhanced the microstructural refinement process. The mean linear intercept length for high angle boundaries (\(\theta > 15^\circ\)) ranged from \sim 35 \mu m at the lowest Z value (850 °C/0.01 s\(^{-1}\)) to \sim 1.5 \mu m at the highest Z value (650 °C/1.0 s\(^{-1}\)). In addition, the dependence of high angle boundary fraction (\(F_{\text{HABs}}\)) and misorientation angle (\(\theta\)) on the Z parameter suggested at least two types of deformation regimes.

In the first deformation regime which happened in highly deformed specimens at 850 °C, \(F_{\text{HABs}}\) and \(\theta\) did not show any strain rate dependence although microstructural refinement occurred with increasing strain rates. For the deformation condition at 850 °C/0.01 s\(^{-1}\), the microstructure after the peak stress and within the softening region showed clear evidence for strain induced boundary migration (SIBM) accompanied with the formation of a few equiaxed grains newly formed, especially at the later stages of deformation.

In the second regime happening in specimens highly deformed at 750 °C and 650 °C, \(F_{\text{HABs}}\) and \(\theta\) increased with decreasing temperature and increasing strain rate. It was suggested that this trend is
caused by more effective grain subdivision phenomenon. Microstructures of the specimens deformed within the softening region at 750 °C and 650 °C (constant strain rate: 0.01 s⁻¹) more or less revealed the presence of dense substructures within the initial grains. Additionally, at a lower temperature (650 °C), the more frequent occurrence of deformation bands led to the formation of numerous numbers of high angle boundary segments.

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