

DEFORMATION INDUCED FERRITE TRANSFORMATION AND GRAIN REFINEMENT IN LOW CARBON STEEL

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ABSTRACT

Ultra-fine grained microstructure is one of the most important features for future steels. It is proved that deformation induced ferrite transformation (DIFT) is a very effective method to produce ultra-fine grained ferrite. This method can be more easily commercially realized than other grain ultra-refinement methods partly because of its similarity to TMCP that is widely used in mass production line. This paper will present our research works on DIFT from three aspects, i.e. DIFT thermodynamics, microstructural evolution during DIFT and grain refinement of microalloyed steel by DIFT rolling.

KEYWORDS

Grain refinement, deformation induced ferrite transformation (DIFT), thermodynamics, microstructural evolution, microalloyed steel

INTRODUCTION

Grain refinement is regarded as the only method to improve both the strength and toughness of steels simultaneously. Since 1960's, more works have been devoted to refine ferrite grain in HSLA steels. An effective grain refinement method, TMCP, has been developed and widely used in industrial production facilities^[1]. However, the minimum grain size of ferrite achieved by TMCP is limited to 10~20 μm for plain low carbon steels, and 4~5 μm for microalloyed steels. Since 1990's, some ultra-fine grained approaches, such as severely plastic deformation and ultimate use of TMCP, have been developed and intensively studied in the laboratory^[2,3]. The ferrite grain has been refined to an order of submicron or nanometer and thus improved the mechanical properties significantly. However, there is still a gap to be overcome between laboratory trials and low cost, high efficient industrial scale production of ultra-fine grained steels at present.

In 1981, R.Priestner et.al indicated that $\gamma \rightarrow \alpha$ transformation could occur in roll gap during rolling and called it "Strain Induced Transformation to Ferrite"^[4]. In 1987, Y.Matsumura and H.Yada reported that the ferrite grain of plain C-Mn steel could be refined to 1-3 μm through multi-pass deformation at 1073K, in which the grain refinement mechanisms were considered as a combination of both "Deformation Induced Transformation" and ferrite dynamic recrystallization^[5]. Hereafter, P.D.Hodgson et.al^[6], S. Lee et.al^[8] and W.Y.Choo et.al^[7] reported the transformation analogous to aforementioned ones in succession, and they named it "Strain Induced Transformation" and "Strain Induced Dynamic Transformation" respectively. Although the names that different researchers used are different, this kind of transformation possesses two common characteristics. First, the $\gamma \rightarrow \alpha$ transformation occurs during deformation rather than during cooling after deformation. Second, ultra-fine ferrite grains could be obtained through the transformation. The work of ours shows that the transformation is induced through the increase of both austenite free energy and nucleation site density caused by deformation applied, so each of deformation variables, as well as strain, strain rate, temperature or deformation manner could have an effect on the transformation. Thus, we named this kind of transformation as "**Deformation** Induced Ferrite Transformation" (abbreviated as DIFT)^[9, 10]. DIFT was applied by the authors to refine ferrite grains to 3 μm in plain low carbon steels and 1 μm in microalloyed steels, with yield strength to be increased to the level of 400MPa and 800MPa respectively^[9-11].

The advantage of DIFT over other grain ultra-refinement methods is that DIFT could be realized in

the existing industrial production facilities or in the modified ones. Therefore, DIFT Processing is expected to have an attractive future for application.

Some research results of ours will be presented in this paper, and three topics will be addressed: thermodynamics of DIFT, microstructural evolution during DIFT and grain refinement of microalloyed steel by DIFT rolling.

1. THERMODYNAMICS OF DIFT

The free energy change of $\gamma \rightarrow \alpha$ transformation without deformation is expressed by

$$\Delta G = -V\Delta G_{chem.} + A\sigma_{\gamma/\alpha} + V\Delta G_s \quad (1)$$

If austenite is deformed, the accumulative deformation energy should be taken into consideration. Equation (9) is written as

$$\Delta G = -V\Delta G_{chem.} + A\sigma_{\gamma/\alpha} + V\Delta G_s - V\Delta G_D = -V(\Delta G_{chem.} + \Delta G_D - \Delta G_s) + A\sigma_{\gamma/\alpha} \quad (2)$$

where $\Delta G_{chem.}$ is the chemical driving force, ΔG_D is the accumulative deformation energy, ΔG_s is the strain energy, and $\sigma_{\gamma/\alpha}$ is the γ/α boundary energy. It can be seen that the driving force for DIFT consists of two parts: one is the chemical driving force $\Delta G_{chem.}$, the other is the accumulative deformation energy ΔG_D . Therefore, it is important to obtain the value of accumulative deformation energy quantitatively and to study its influence on DIFT. The authors have made an attempt to study this accumulative deformation energy^[10]. The main results are presented as follows.

A. Accumulative Deformation Energy

The accumulative deformation energy of austenite is thought to consist of mainly two parts: grain boundary energy and dislocation energy. The increase of grain boundary energy during deformation is dependent on two factors. First, deformation elongates austenite grains and increases the grain boundary areas. Second, deformation enhances the disorder of grain boundary structure, which leads to an increase in the grain boundary energy per unit area, $\sigma_{\gamma/\gamma}$ (increased by approximately 10%). Assuming the initial austenite grain is cubic with size of D_0 and the deformation mode is plane strain compression, the grain boundary energy per unit mole, ΔG_{gb} as a function of strain can be expressed by

$$\Delta G_{gb} = [\exp(\varepsilon) + \exp(-\varepsilon) + 1] \cdot \sigma_{\gamma/\gamma} \cdot V_\gamma / D_0 \quad (3)$$

where $\sigma_{\gamma/\gamma}$ is equal to 0.8J/m^2 , V_γ is the molecular volume of austenite ($=7.1 \times 10^{-6}\text{m}^3/\text{mol}$), ε is the strain.

According to the dislocation theory, the total energy of dislocation per unit length, E , which is a sum of elastic strain energy and dislocation core energy, is defined by

$$E = \frac{\mu b^2}{4\pi K} \ln \frac{R}{\lambda b} = k\mu b^2 \quad (4)$$

where μ is the shear elastic modulus of austenite, b is the Burgers vector, k is a constant ranging from 0.5 to 1.0 and takes 1.0 here. Then, the dislocation energy of austenite per unit mole ΔG_{dis} is obtained as

$$\Delta G_{dis.} = \mu \rho b^2 V_\gamma \quad (5)$$

where ρ is the dislocation density. The flow stress as a function of dislocation density is defined by

$$\sigma = M \alpha \mu b \rho^{1/2} \quad (6)$$

where σ is the flow stress, M is Taylor factor and α is a constant. Then, the dislocation energy per unit mole as a function of flow stress is obtained as

$$\Delta G_{dis} = \frac{\sigma^2 V_\gamma}{M^2 \alpha^2 \mu^2} \quad (7)$$

Fig.1 shows the grain boundary energy of austenite as a function of strain calculated from equation (3). Fig.2 shows the dislocation energy of austenite as a function of flow stress calculated from

equation (7), where $\mu=7.9 \times 10^{10} \text{N/m}^2$, $M=3.11$ for f.c.c metals and $\alpha=0.15$. In general, the flow stress during hot rolling of low carbon steels ranges from 150MPa to 300MPa, so the dislocation energy ranges from 10J/mol to 40J/mol. It can be seen from **Fig.1** that the grain boundary energy remains at a relatively low level until the strain is relatively large. Thus, in general, the grain boundary energy could be ignored.

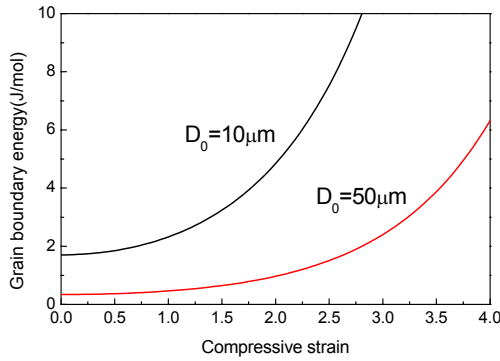


Fig.1 Austenite grain boundary energy as a function of compressive strain

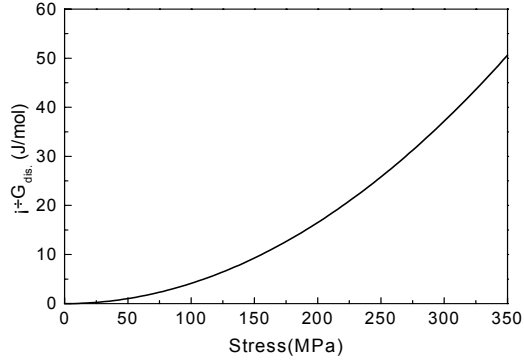


Fig.2 Dislocation energy as a function of flow stress

It is necessary to obtain the relationship between accumulative deformation energy and deformation variables. The method is described as follows. First, the relationship between flow stress and deformation variables can be described through the constitutive equation of hot deformation as follows

$$\dot{\varepsilon} = A \{ \sinh(\alpha \sigma) \}^n \exp(-Q / RT) \quad (8)$$

where the undetermined constants A , n and deformation activation energy Q can be obtained through experiments. Then, the relationship between accumulative deformation energy and deformation variables can be achieved through equation (7).

Based on the method mentioned above, we have obtained the relationship between accumulative deformation energy and deformation variables for a low carbon microalloyed steel (0.11C-0.25Si-1.48Mn-0.048Nb).

$$\Delta G_D \text{ (J/mol)} = 77.5 - 13.5(\log Z) + 0.57(\log Z)^2 \quad (9)$$

where ΔG_D is the accumulative deformation energy and Z is the Zener-Hollomon parameter

$$Z = \dot{\varepsilon} \exp(Q / RT) \quad (10)$$

The accumulative deformation energy is plotted as a function of both temperature and strain rate, as shown in **Fig.3**. This figure shows that the accumulative deformation energy increases with decreasing temperature and increasing strain rate.

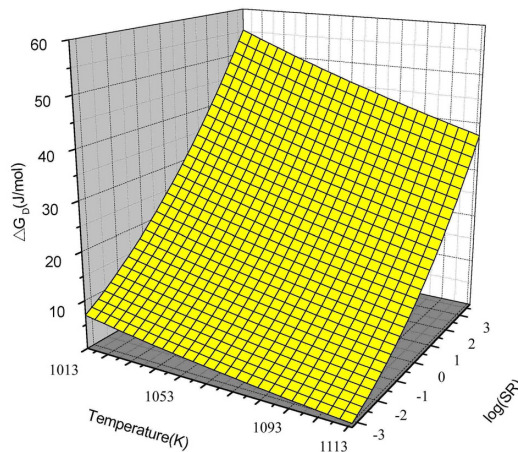


Fig.3 Accumulative deformation energy as a function of temperature and strain rate ($\varepsilon=0.6$)

B. Influence of Accumulative Deformation Energy on A_{d3}

Fig.4 shows the effect of accumulative deformation energy on the equilibrium $\gamma \rightarrow \alpha$ transformation temperature for 1.48Mn-0.25Si-0.048Nb steel, as calculated by Thermo-Calc. It is clear that the addition of accumulative deformation energy enlarges ($\gamma + \alpha$) two phase region and raises the equilibrium $\gamma \rightarrow \alpha$ transformation temperatures to higher ones. The new dynamic temperature is called the upper limit transformation temperature for DIFT at a certain deforming condition, A_{d3} .

Fig. 5 shows A_{d3} as a function of accumulative deformation energy for 0.11C-1.48Mn-0.25Si-0.048Nb steel. The data in **Fig.5** is linearly fitted and the following regression equation could be obtained

$$A_{d3}(K) = 1096.8 + 0.87 \Delta G_D \quad (11)$$

Then, the relationship between A_{d3} and deformation variables, such as deformation temperature and strain rate is obtained, as shown in **Fig.6**. It is shown that A_{d3} increases with the increase of strain rate or the decrease of deformation temperature.

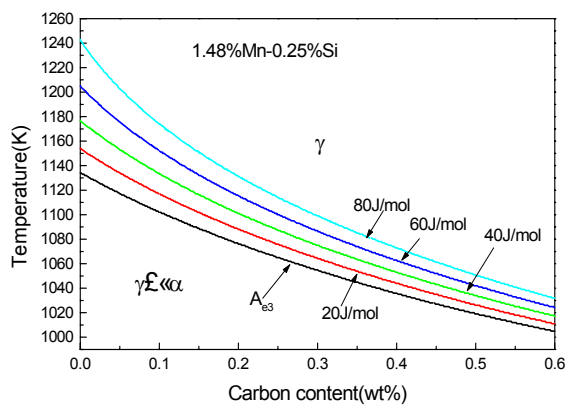


Fig.4 Influence of accumulative deformation energy on equilibrium $\gamma \rightarrow \alpha$ transformation temperature

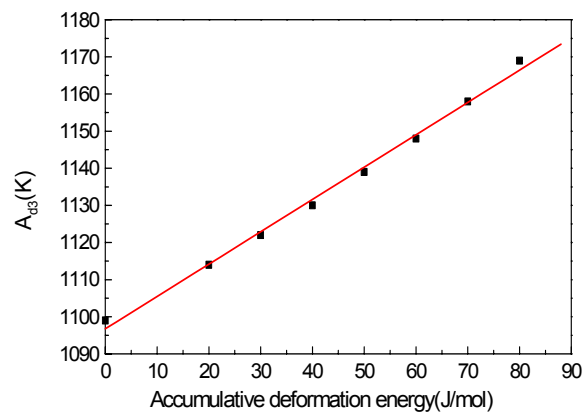


Fig.5 A_{d3} as a function of accumulative deformation energy

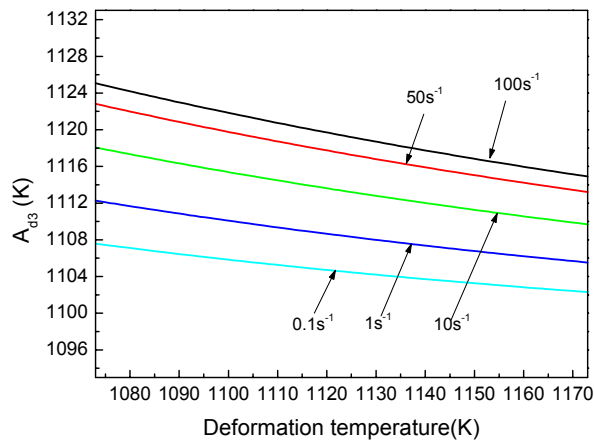


Fig.6 A_{d3} as a function of temperature at various strain rates ($\epsilon=0.6$)

2. MICROSTRUCTURAL EVOLUTION DURING DIFT

As mentioned above, DIFT is a more effective method for grain refinement than conventional TMCP. But the reason has not been made clear yet. In order to clarify it, it is necessary to investigate the microstructural evolution during DIFT and during conventional TMCP.

A. Experimental Procedures

The experimental materials were two Nb-containing steels, which are referred to as steel A and B

(Table 1). Steel A was used for the investigation on DIFT and Steel B for TMCP. Hot compression tests were performed using a Gleeble 2000 hot simulation testing machine. The samples with size of $\Phi 8 \times 12 \text{ mm}$ were firstly soaked at 1453K for 3 minutes, and then cooled the preset temperatures at a rate of 5K/s, followed by compression at a constant strain rate. The samples were water quenched immediately after deformation (Steel A) or cooled at a rate of 5K/s to different temperatures followed by water quenching (Steel B). The deformation temperatures, strains, strain rates and cooling modes used in the compression tests were listed in Table 2.

Table 1 Chemical compositions of the tested steels (wt%)

	Chemical compositions (wt.%)						
	C	Si	Mn	Nb	Ti	Cu	RE
Steel A	0.087	0.245	0.51	0.017	0.016	0.035	0.021
Steel B	0.1170	0.24	1.47	0.058	0.0144	\	\

Table 2 Hot deformation schedule for the tested steels

	Deformation temperature(K)	Strain rate(s^{-1})	Strain	Cooling mode after deformation
Steel A	1053	15	0.16, 0.25, 0.36, 0.69, 1.20	Water quenching
Steel B	1093	1	0.92	Water quenching or cooled at a rate of 5K/s to 973, 913 and 853K followed by water quenching

B. DIFT

Fig.7 shows the microstructures of the steel A obtained by deformation at 1053K and a strain rate of 15 s^{-1} to various strains. It can be seen that deformation induced ferrites (DIF) are firstly nucleated at prior-austenite grain boundaries (Fig.7(a)), and then occupy all the prior-austenite grain boundaries, forming a continuous net-like structure(Fig.7(b)). With the increase of strain, the nucleation sites of ferrite are changed into the interior of the prior-austenite grains, which leads to a rapid increase of ferrite amount (Fig.7(c)). It is observed that there are two types of nucleation site for intragranular nucleation of DIF. One is the deformation band (Fig.8(a)), and the other is γ/α interface (Fig.8(b)). DIF is nucleated at the γ/α interface repeatedly, which leads to the advance of DIF into the untransformed austenite.

The volume fraction of DIFT as a function of strain is shown in Fig.9. It can be seen that the transformation amount-strain curve exhibits a sigmoid shape, which means that the transformation proceeds relatively slowly at both lower and higher strain levels and proceeds most rapidly at medium strain level. Fig.9 also shows the relationship between DIF grain number per unit area and strain, which also exhibits a sigmoid shape. Thus, there are three stages for DIFT: the first stage with lower nucleation rate, the second stage with higher nucleation rate and the third stage with lower nucleation rate. As mentioned above, the prior-austenite grain boundaries are the main nucleation sites at the initial stage. Apparently, the nucleation rate is relatively low since the nucleation sites provided by the austenite grain boundaries are very limited. At the intermediate stage, however, the deformation bands are formed within the austenite grains, which provide abundant nucleation sites, and furthermore, the γ/α interfaces also become potential nucleation sites. These lead to high nucleation rate and transformation rate at this stage. At the final stage of transformation, the volume fraction of DIFT approaches the equilibrium state, leading to the decrease of the nucleation rate and transformation rate. Fig.10 schematically illustrates the three-stage microstructure evolution during DIFT.

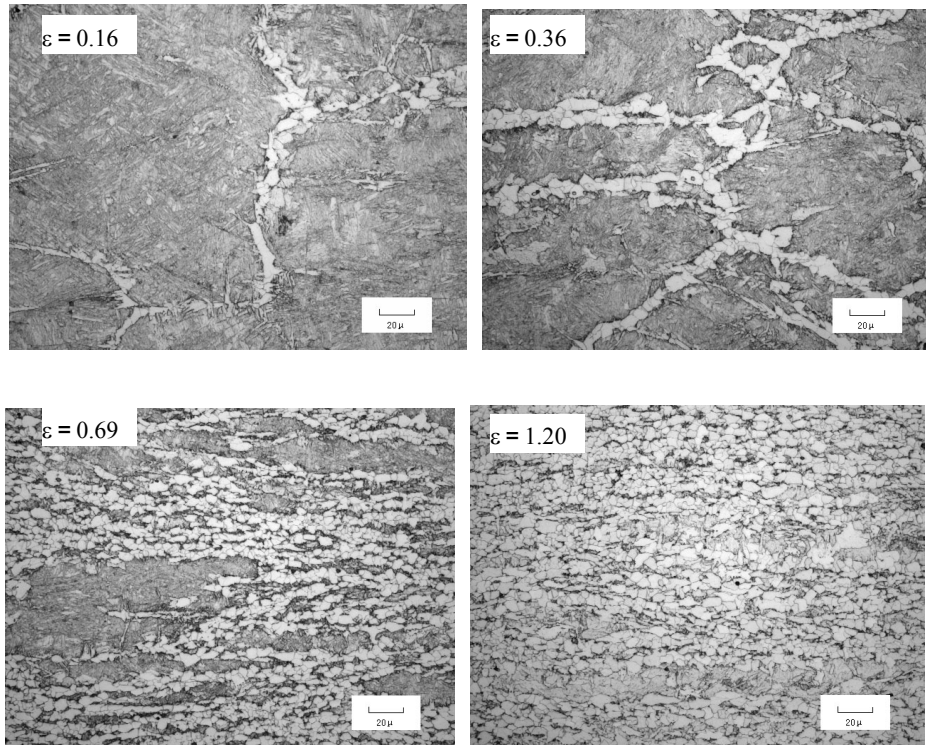


Fig.7 Microstructures obtained by deformation at 1053K and a strain rate of $15s^{-1}$ to various strains for steel A

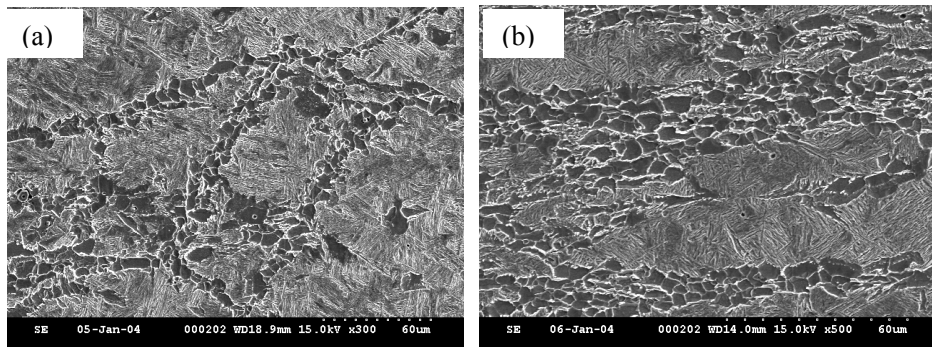


Fig.8 Intragranular nucleation of DIF at (a) deformation band and (b) γ/α interface

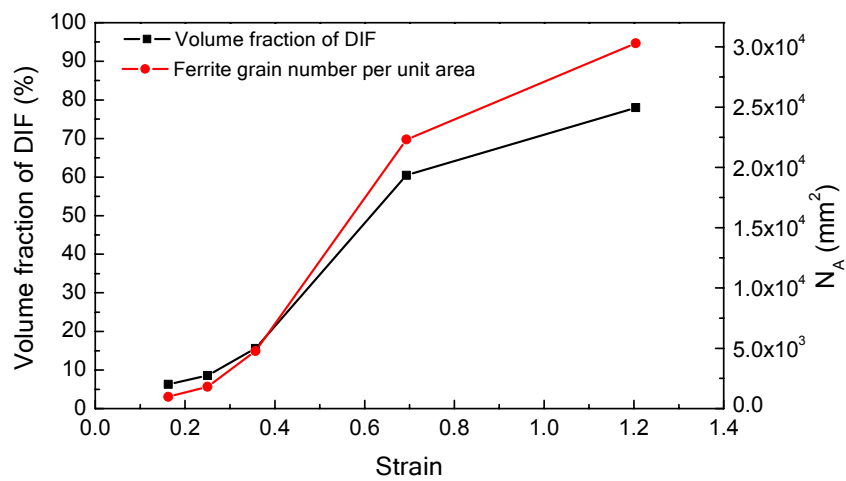


Fig.9 Volume fraction of DIF and ferrite grain numbers per unit area as a function of strain

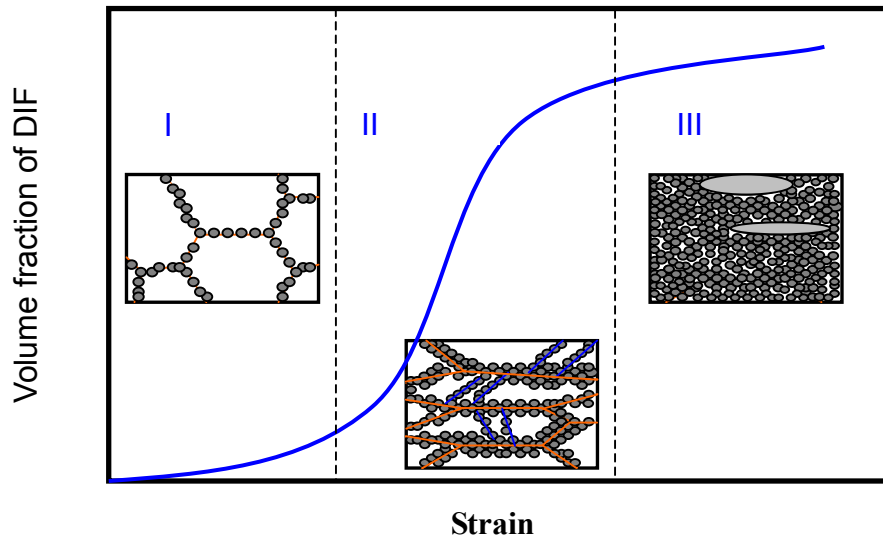


Fig.10 Schematic illustration of three-stage microstructural evolution for DIFT

TEM observation was also carried out to reveal the substructure of the deformation induced ferrite (DIF), as shown in Fig.11. It is found that the dislocation density of DIF is low, and some dislocations are being rearranged to form a low angle grain boundary. This indicates that dynamic recovery of DIF is occurring during deformation. Moreover, it is found that there are a large amount of Fe_3C precipitates with less than 20nm in diameter within DIF. This is probably because that carbon in DIF is initially supersaturated, which results in a rapid precipitation of Fe_3C after deformation.

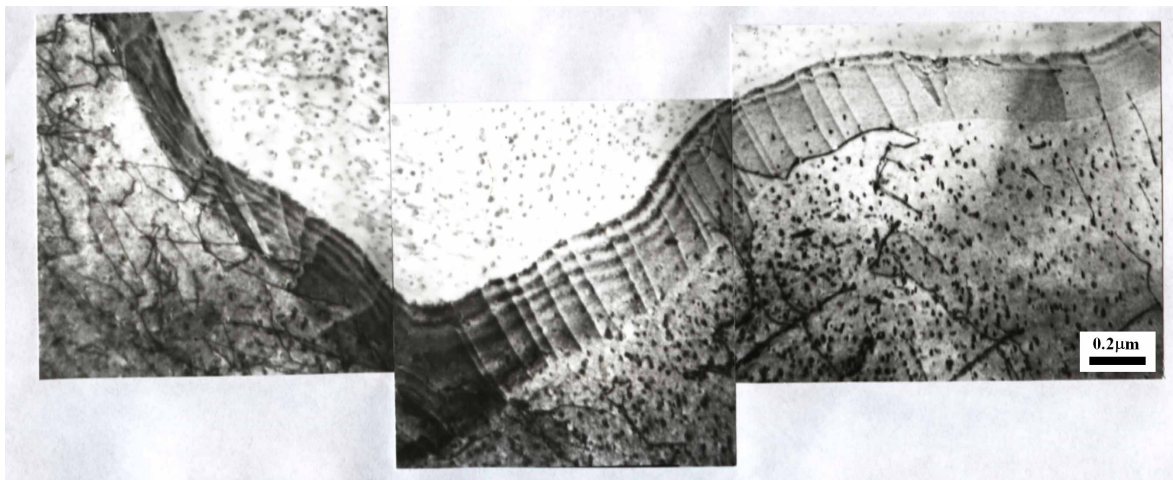


Fig. 11 TEM image of DIF

C. TMCP

Fig.12 shows the microstructures of steel B obtained by deformation at 1093K to the strain of 0.92 followed by water quenching (Fig.12(a)) or by cooling at a rate of 5K/s to various temperatures and then water quenching (Fig.12 (b-c)). It can be seen from Fig.12(a) that the microstructure is a mixture of bainite and martensite, which is the transformation product of deformed austenite during water quenching, indicating that DIFT didn't occur during high temperature deformation. During cooling after deformation, ferrites are nucleated at both austenite grain boundaries and deformation bands and subsequently grow up. Table 3 gives the volume fraction, mean grain size and grain number of ferrite at different temperature.

It can be seen from Table 3 that the volume fraction of ferrite increase from 973K to 913K, but it

changes very little from 913K to 853K. This indicates that the $\gamma \rightarrow \alpha$ transformation is nearly finished at 913K. The ferrite grain size increases continuously during the whole transformation process. The increase of grain size from 913K to 853K is evidently resulted from grain coarsening because the transformation is nearly finished at 913K. It should be noted that the ferrite grain number per unit area decreases with $\gamma \rightarrow \alpha$ transformation proceeding. This indicates that the nucleation of ferrite conforms to the site saturation mechanism^[1]. In other words, the nucleation process is finished at the initial stage of transformation, and during the subsequent transformation, grain growth and coarsening are dominant. Similar results were also obtained in the study of continuous cooling transformation of C-Mn-Nb steel in the literature^[12].

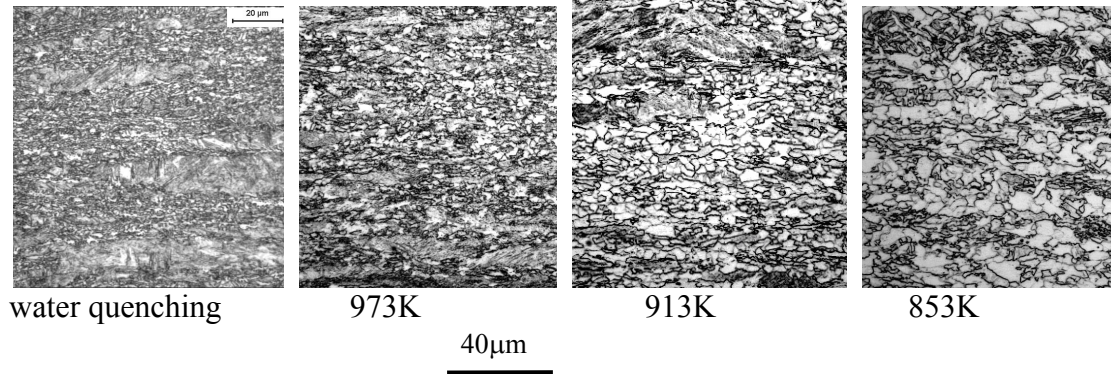


Fig.12 Microstructures of steel B obtained through deformation at 1093K to a strain of 0.92 followed by cooling at 5K/s to various temperatures and then water quenching

Table 3 Ferrite volume fraction (f_α), mean grain size (d_α) and ferrite grain number per unit area (N_A) of steel B deformed at 1093K to a strain of 0.92 followed by cooling at the rate of 5K/s to various temperatures

Temperature (K)	f_α (%)	d_α (μm)	N_A (mm^{-2})
973	23.1	2.88	20401
913	61.5	5.7	16925
853	63.0	7.5	15651

D. Comparison between DIFT and TMCP

Fig.13 compares the ferrite grain number per unit area as a function of ferrite volume fraction of DIFT and TMCP. It is clear that the grain number increases during DIFT, but it decreases during TMCP. Therefore, DIFT is a nucleation controlling process, i.e, the transformation is mainly accomplished through the continuous nucleation process, whereas TMCP is a grain growth and coarsening dominant process. The growth of ferrite grain is inhibited to a great extent during DIFT due to the rapid and repeated nucleation of ferrite grain at γ/α interface, which results in a final finer grain size than that obtained by TMCP.

3. GRAIN REFINEMENT OF MICROALLOYED STEEL BY DIFT ROLLING

A lot of rolling tests were carried out in a laboratory rolling mill to investigate the effectiveness of grain refinement by DIFT. An example is shown as follows. The experimental material was a commercial Nb-V-N microalloyed steel with the compositions 0.094%C- 0.47%Si- 1.38%Mn- 0.1%V- 0.04%Nb- 0.02%Al -0.018%N. The steel plate with thickness 30mm was firstly soaked at 1473K for 30min, followed by five pass rolling to a final thickness 3mm, then cooled at 20K/s to 823K and finally cooled slowly in a furnace to room temperature. The rolling schedule is shown in Fig.14. The role of the first pass rolling at 1273K is to refine austenite grains by recrystallization considering that fine austenite grains will be beneficial to DIFT. The role of the second pass rolling at 1223K is to promote the precipitation of (Nb,V)(C,N), which was found to help to accelerate

DIFT kinetics^[10]. The final three pass rolling at 1093K is considered to be DIFT rolling according to the microstructural observation of water quenched samples^[9]. The final microstructure of the experimental steel is shown in Fig15. It can be seen that ultra-fine ferrite grains with 1.5 μm in size is obtained. The grain size is much smaller than that produced by conventional TMCP (4-5 μm).

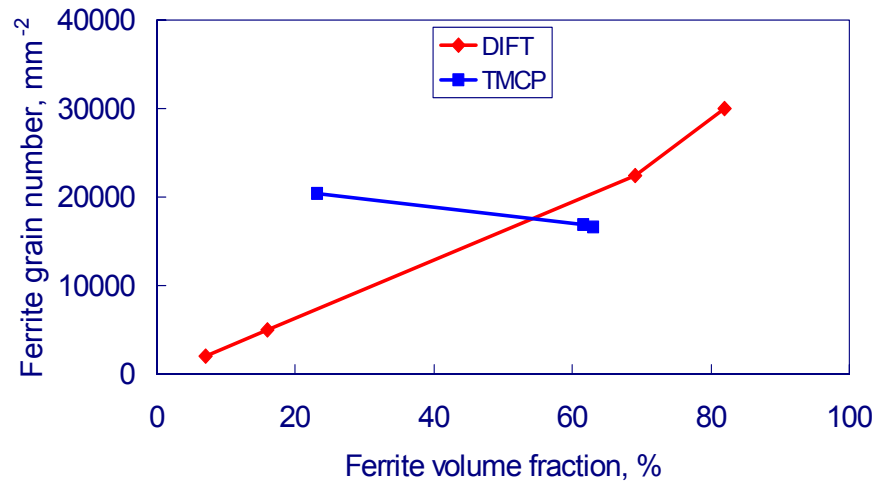


Fig. 13 Comparison of ferrite grain number between DIFT and TMCP

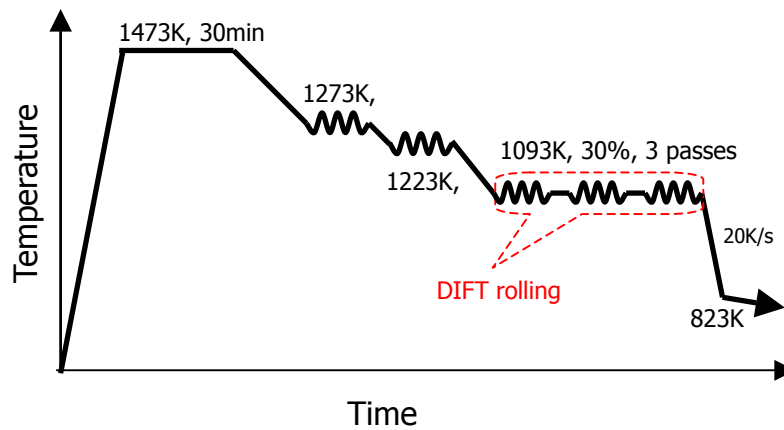


Fig.14 Deformation schedule for the laboratory rolling

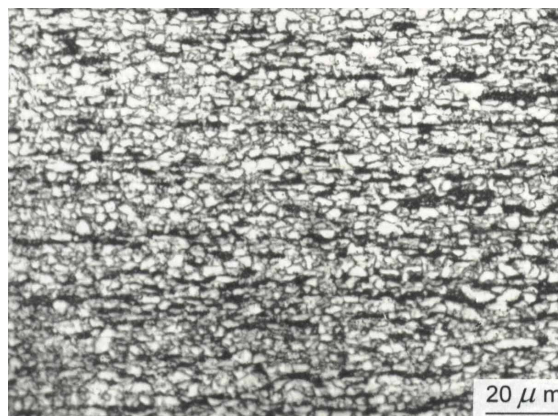


Fig. 15 Microstructure of ultra-fine grained V-Nb-N microalloyed steel

4. CONCLUSIONS

(1) In thermodynamics, a prominent feature of DIFT distinguished from static transformation without deformation is an addition of accumulative deformation energy (ADE) of austenite into the transformation driving force. The ADE mainly consists of two components: grain boundary energy

and dislocation energy. A method to calculate the ADE of austenite was proposed. It is shown that at certain strain, the lower the deformation temperature or the higher the strain rate, the higher the accumulative deformation energy. The addition of accumulative deformation energy raises $\gamma \rightarrow \alpha$ equilibrium transformation temperature to a higher one which is called A_{d3} .

(2) There are three stages for DIFT: the first stage with lower nucleation rate, the second stage with higher nucleation rate and the third stage with lower nucleation rate. DIFT is a nucleation dominant process, i.e. the $\gamma \rightarrow \alpha$ transformation is mainly accomplished by continuous nucleation, whereas TMCP is a grain growth dominant process. The growth of ferrite grain is inhibited to a great extent during DIFT due to the rapid and repeated nucleation of ferrite grain at γ/α interface, which results in a final finer grain size than that obtained by TMCP.

(3) Ultra-fine grained V-Nb-N microalloyed steel with a grain size of 1.5 μm was produced by DIFT rolling.

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