Recrystallization–precipitation interaction of a Ti microalloyed steel with controlled rolling process

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Abstract: The interaction between austenite static recrystallization and strain induced precipitation of a Ti microalloyed steel deformed in the non-recrystallization region was investigated by two-stage interrupted compression method and transmission electron microscopy (TEM). The softening rate curves of austenite were obtained by the true stress-strain curves of the two-pass of deformation. Results showed that the softening rates increased with increasing deformation temperatures. The softening rate curve exhibited a “S” shaped, and the static recrystallization was completed in 300 s when deformed at 1000°C, while deformed temperatures were lower than 950°C, the softening rates dropped sharply and increased very slowly as pass inter time increased. At 975°C or below, platforms appeared in softening curves, which implied strain-induced precipitates occurred during the isothermal process after the first deformation. The shortest incubation time of strain-induced precipitates was about 78 s when deformed at 925°C. Based on TEM results, strain-induced precipitates were found to be mainly distributed at dislocations and the diameters of the particles precipitated at dislocations were obviously larger than those of precipitated in other nucleation sites due to the effective route for the diffusion of Ti element. Evolution of austenite grains morphologies showed that the static recrystallization of deformed austenite was strongly inhibited by strain-induced precipitation and occurred again after the strain-induced precipitation finished.

1. Introduction
Combined with Thermomechanical Controlled Process (TMCP), addition of a small number of microalloying elements in steel can significantly improve the strength and maintain good toughness via grain refinement strength and precipitation strength[1]. A Ti microalloyed steel with the yield strength of 700 MPa was developed by controlling grains and nanoscale precipitates[2]. TiC precipitated in deformed austenite during the controlled rolling process pinned grain boundaries and inhibited austenite recrystallization[3]. In industrial production, the controlled rolling process usually includes rolling in
recrystallization region and the non-recrystallization region. Rolling in the recrystallization region, austenite grains are refined through repeated dynamic recrystallization. Rolling at the temperatures within the non-recrystallization region cause accumulation of stored strain energy due to no complete recrystallization, which results in the formation of elongated grains and deformation bands, and a large increase of dislocations and vacancies[4]. It is clear that these elongated grain boundaries, deformation bands, and defects can act as nucleation sites for γ→α transformation, and finally lead to smaller ferrite grains. Besides the recrystallization temperature is generally increased by the microalloying elements and carbonitrides of the microalloying elements by solute drag effect and pinning effect, respectively[5]. Also, the carbonitrides precipitated in austenite can also act as nucleation sites for γ→α transformation[6]. In order to avoid rolling in the mixed region and refine the transformation structures to improve the product properties, it is of great significance to study the interaction between recrystallization and strain-induced precipitation of austenite in a Ti microalloyed steel.

2. Materials and experimental procedure

The chemical composition of the Ti microalloyed steel is shown in Table 1. The two-stage interrupted compression experiments were performed to investigate the interaction between static recrystallization and strain-induced precipitation by using Gleeble 3800® Thermo-Mechanical Simulator, as shown in Fig. 1. The cylindrical samples for hot compression tests with size of φ10 mm × 15 mm were cut from as-received cast steel. After austenization at 1200℃ for 5 min, samples were cooled to 1000-900℃ at 20℃/s rate and then deformed by 30% reduction, respectively. After hot compression, samples were held at the same elevated temperature for 0-1000 s and then further deformed by another 30%, after which, water quenched to room temperature. In order to ensure the repeatability of the experiments, each group of experiments was done three times. Fig. 1(b) shows the method for determining the softening ratio of austenite, and the calculation formula of the softening rate X is as follows[7]:

\[
X = \frac{\sigma_m - \sigma_{1,2\%}}{\sigma_m - \sigma_{2,2\%}}
\]

Where, \(\sigma_m\) is the maximum flow stress of the first-pass compression, \(\sigma_{1,2\%}\) and \(\sigma_{2,2\%}\) are the yield stress, i.e. the true stress at 2% true strain of the first and second pass deformation, respectively.

Prior austenite grain boundaries (process 1) were etched in saturated picric acid solution containing detergent for 3-15 min at 45℃ depending on different heat treatment conditions and the recrystallization volume fraction of austenite was measured using standard systematic point count method. The type, morphology and size distribution of precipitates (process 1) was analyzed using FEI Tecnai G2 F20 transmission electron microscope (TEM). The size of strain-induced precipitates was determined using Image J software.

Table 1 Chemical composition of experimental steel (wt. %)

| C   | Si  | Mn  | P   | S   | N   | Ti  | Fe  |
|-----|-----|-----|-----|-----|-----|-----|-----|
| 0.05| 0.21| 1.04| 0.011| 0.0056| 0.0048| 0.11| balance |

Fig.1 Thermal simulation experiments scheme(a) and determination of softening ratio(b)
3. Results and discussion

The true stress-strain curves. Fig. 2 shows the true stress-strain curves of the tested steel deformed by two-stage interrupted compression. Regardless of the deformation temperatures, the true stress increased continuously with increasing strain during the first pass deformation, exhibiting a strong work-hardening behavior, which indicates that the steel did not occur its dynamic recrystallization. Besides, under the same deformation temperature and strain, the stress obtained by the second pass deformation was larger than that of the first pass deformation. However, increasing the holding time, the true stress-strain curves of the second deformation showed different trends. When the deformation temperature was 1000℃, the yield stress (2% offset stress) of the second pass deformation decreased with the increase of the holding time. Below 1000℃, the yield strength of the second pass was not always decreased with prolonging of holding time but showed the trend of decreasing-invariable-re-decreasing. The decrease of the yield strength of the second deformation indicated the static recovery and recrystallization occurred during isothermal heat treatment. With the holding time increased, more austenite residual strain energy was released due to more sufficient static recrystallization of austenite, which led to the decrease of yield stress of the second pass deformation.

![Fig. 2 The true stress-strain curves of specimens deformed by two-stage compression. Besides, The figure contain many important curves, not all of them are shown for clarity better identification. (a) 1000℃; (b) 950℃; (c) 925℃; (d) 900℃](image)

Softening rate curves. The static recrystallization process can be characterized by softening rates. Fig. 3(a) shows the softening rates calculated by Eq. (1) for five different deformation temperatures after deformed a 30% reduction with different holding time. It is clear that the deformation temperature has a significant effect on the softening rate of austenite. Regardless of the holding time, the softening rates increased with increase of the deformation temperatures. When the deformation temperature was 950℃ or higher, the softening rate of austenite increased rapidly with the holding time increased. Especially...
deformed at 1000℃ and the holding time prolonged to 300 s, the softening rate of austenite reached to 96.5%. That is to say, austenitic completed the static recrystallization process. However, the softening rates at 900℃ and 925℃ were obviously lower and increased very slowly with the holding time increased compared with higher deformation temperatures. It should be noted that platforms appeared on the softening rate curves with the increase of holding time when the deformation temperatures were 975℃, 950℃, 920℃ and 900℃, respectively. Wang indicated that the platform on the austenite softening rate curve means the occurrence of strain-induced precipitation[8]. The static recovery or recrystallization of austenite would be inhibited or even stopped by the pinning effect of precipitated particles. However, there was no plateau appeared on the softening rate curve during the isothermal process after deformation at 1000℃, which means that no strain-induced precipitates precipitated. It can be concluded austenite static recrystallization process and strain-induced precipitation interacted with each other and both are affected by temperatures.

Based on the softening rate curves, the recrystallization process of austenite deformed at 975℃ or lower, can be divided into three stages due to the strain-induced precipitation: (ⅰ) Before strain induced precipitates started, the volume fraction of static recrystallization increased rapidly with holding time increased; (ⅱ) The static recrystallization process was inhibited or even stopped due to the pinning effect by strain-induced precipitates; (ⅲ) Static recrystallization reoccurred, during which, precipitates coarsened with increasing the holding time, leading to weak pinning effect on grain boundaries.

![Fig. 3 The softening rate curves for different deformation temperatures after deformed by 30% reduction with different holding time](image)

**Strain-induced precipitates.** The start and finish times of the platforms that appeared on the softening curves marked with arrows are generally considered as the start (Ps) and finish (Pf) times of strain-induced precipitation. It is worth noting that the finish time does not indicate completion of the strain-induced precipitation, rather than the time, at which precipitation hardening reaches its maximum value[9]. By measuring the starting time of the platform, it can be found that the shortest starting time of strain-induced precipitates was about 78 s when deformed at 925℃. The deformation temperature with the shortest starting time is higher than that of 910℃ reported by Wang in a 0.05% C-0.10%Ti HSLA steel with a 20% deformation[8]. This result was agreement with Yong’s conclusion that deformation can slightly increase the nose temperature of strain-induced precipitation[10]. As compared with Wang’s work, the present work employed a more severe deformation condition. The relatively larger deformation was the main factor that leading to a high temperature. Large deformation brings higher dislocation densities and sub-grains, which facilitated the nucleation of precipitates as it provided a fast diffusion channel.

Fig. 4 shows the TEM morphology of precipitates observed in the sample deformed at 925℃ and isothermally treated for 600 s. Spherical and irregular shaped particles with size about 6-16 nm were distributed in the matrix. According to our previous research, these particles were identified as TiC with
a NaCl-type crystal structure[11]. It should be noted that a large number of particles precipitated on dislocations, and the diameters of the precipitates were obviously larger than that of precipitated in other nucleation sites. Hong et al showed that strain-induced precipitations preferentially nucleated at dislocations[12]. Okaguchi and Hashimoto reported that the maximum free energy change and critical embryo radius for carbides precipitation at dislocations both are less than those for nucleation at grain boundaries[13]. Based on the results in Figure 3, it can be seen that these precipitates cannot effectively pin the grain boundaries to prevent austenite recrystallization, because the softening rate curve shows an upward trend after deformed at 925°C by holding for 600 s.

![TEM images of strain-induced precipitates of specimens deformed at 925°C for 600s](image.png)

**Effect of strain-induced precipitation on softening behavior.** Static recrystallization and strain-induced precipitation occurred during the isothermal process. In fact, recovery or recrystallization, as well as strain-induced precipitation, compete for stored energy because of deformations[14]. Strain induced precipitation inhibited the recrystallization of austenite and delayed the completion time of recrystallization. Fig. 9 shows optical micrographs of prior austenite in Ti microalloyed steel held for different times after deformed at 925°C. At 925°C for 80 s (the starting time of strain-induced precipitation is about 78 s), almost all of the grains were elongated along the deformation direction with flat shape. When the holding time was increased to 300 s (which is close to the end time of strain-induced precipitation at 925°C), original austenite grains hardly changed in Fig.5 (b) compared with austenite grains in Fig.5 (a). Further increasing the pass inter time to 1000 s, some new recrystallized grains were observed (see Fig. 9 (c)). The flat and static recrystallization grains (SRG) were co-existed and the volume fraction of recrystallization is about 36%, which was close to the calculated result in Fig. 3. From Fig.5, the evolution of austenite grain morphologies correlates with strain-induced precipitation: static recrystallization of deformed austenite was strongly inhibited by strain-induced precipitation and occurred again after the strain-induced precipitation finished.
4. Conclusions
The softening rate increased with increase of holding time and deformation temperature. Due to the inhibition of strain-induced precipitates on austenite recrystallization process, plateaus appeared on the softening rate curves when the deformation temperatures were below 975°C.

The shortest incubation time of strain-induced precipitates was about 78 s after deformed at 925°C. Strain-induced precipitates were found to be mainly distributed at dislocations and the diameters of the particles precipitated at dislocations were obviously larger than that of precipitated at other nucleation sites due to the effective route for the diffusion of Ti element.

Recrystallization is related to competition of recrystallization driving force and pinning force of precipitates. Recrystallization was almost completely inhibited during the growth stage of precipitates and reoccurred during coarsening stage.

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Reference
[1] J.B. Seol, S.-H. Na, B. Gault, J.-E. Kim, J.-C. Han, C.-G. Park and D. Raabe, Core-shell nanoparticle arrays double the strength of steel, Scientific Reports, 7 (2017) 42547.
[2] Q.L. Chen, X.P. Mao and X.J. Sun, Ti (CN) Precipitation in Ultra-High Strength Ti Micro-Alloyed Steels with 700MPa Yield Strength on TSCR Process, Advanced Materials Research, Trans Tech Publ, 2014, pp. 61-69.
[3] C.Y. Chen, C.C. Chen and J.R. Yang, Microstructure characterization of nanometer carbides heterogeneous precipitation in Ti–Nb and Ti–Nb–Mo steel, Materials Characterization, 88 (2014) 69-79.
[4] J. Calvo, I.-H. Jung, A. Elwazri, D. Bai and S. Yue, Influence of the chemical composition on transformation behaviour of low carbon microalloyed steels, Materials Science and Engineering: A, 520 (2009) 90-96.
[5] C. Klinkenberg, K. Hulka and W. Bleck, Niobium carbide precipitation in microalloyed steel, steel research international, 75 (2004) 744-752.
[6] S. Liu and F.C. Liao, Precipitate stability in the heat affected zone of nitrogen-enhanced high strength low alloy steels, Materials Science & Engineering A, 244 (1998) 273-283.
[7] A. Fernández, B. López and J. Rodriguez-Ibabé, Relationship between the austenite recrystallized fraction and the softening measured from the interrupted torsion test technique, Scripta Materialia, 5 (1999) 543-549.
[8] Z.Q. Wang, X.P. Mao, Z.G. Yang, X.J. Sun, Q.L. Yong, Z.D. Li and Y.Q. Weng, Strain-induced precipitation in a Ti micro-alloyed HSLA steel, Materials Science and Engineering A, 529 (2011) 459-467.

[9] Z. Wang, H. Zhang, C. Guo, W. Liu, Z. Yang, X. Sun, Z. Zhang and F. Jiang, Effect of molybdenum addition on the precipitation of carbides in the austenite matrix of titanium micro-alloyed steels, Journal of Materials Science, 51 (2016) 4996-5007.

[10] Q.L. Yong, Secondary Phases in Iron Material, edited by Metallurgical Industry Press, Beijing, 2006.

[11] Z.W. Peng, L.J. Li, S.J. Chen, X.D. Huo and J.X. Gao, Isothermal precipitation kinetics of carbides in undercooled austenite and ferrite of a titanium microalloyed steel, Materials and Design, 108 (2016) 289-297.

[12] S.G. Hong, K.B. Kang and C.G. Park, Strain-induced precipitation of NbC in Nb and Nb–Ti microalloyed HSLA steels, Scripta Materialia, 46 (2002) 163-168.

[13] S. Okaguchi and T. Hashimoto, Computer Model for Prediction of Carbonitride Precipitation during Hot Working in Nb-Ti Bearing HSLA Steels, Transactions of the Iron & Steel Institute of Japan, 32 (1992) 283-290.

[14] Z.H. Zhang, Y.N. Liu, X.K. Liang and Y. She, The effect of Nb on recrystallization behavior of a Nb micro-alloyed steel, Materials Science & Engineering A, 474 (2008) 254-260.