Effect of deformation parameters on microstructural evolution during hot compression of Nb–V–Mo microalloyed steel

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Abstract

The flow behavior of Nb–V–Mo microalloyed steel at different deformation parameters was investigated using isothermal compression tests. The microstructures of the specimens after deformation were observed through the use of a metallographic microscope. The results indicated that the deformation temperature had a significant effect on the grain size of the deformed specimen and the average size of grains increases with corresponding increase in temperature in the range of 800 °C–1100 °C. The dislocations and substructures of the specimens after deformation were characterized using TEM. The bulged grain boundary was observed through TEM when the specimen was deformed at 1100 °C and 1 s⁻¹. The effect of the strain rate on the dynamic recrystallization behavior was investigated using electron backscatter diffraction (EBSD). The EBSD results indicated that the fraction of low-angle grain boundaries decreased with a corresponding decrease in strain rate, and the average sub-boundary misorientation increased with a decreasing strain rate. The continuous dynamic recrystallization mechanism was determined through EBSD analysis.

1. Introduction

High-strength low-alloy (HSLA) steel is an important structural material comprising a small amount of alloy elements (e.g. Ni, Ti, V). These elements enhance its strength and toughness by forming stable carbides, nitrides and carbonitrides, which provide greater atmospheric corrosion resistance than traditional carbon steel [1–4]. Due to their excellent properties, HSLA steels are utilized for many industrial applications, including use in marine structures, automobile manufacturing and transportation pipelines [5, 6]. HSLA steel with excellent properties can be obtained through developing a reasonable hot working process. In a thermomechanical control process (TMCP) such as rolling and forging, the material undergoes deformation at elevated temperatures. One of the most important microstructural changes that can occur during this process is known as dynamic recrystallization (DRX). DRX is also an important mechanism for controlling the evolution of the microstructure [7–9]. DRX grains were formed by the migration of high-angle grain boundaries (HAGBs) driven by deformation storage energy during hot deformation [10]. New grains that form by DRX are initially strain-free and will eventually grow into the existing deformed grains. However, the new grains themselves will increase in terms of stored energy with continued deformation. This may eventually lead to re-nucleation of new DRX grains. Often, the fine grain size obtained by TMCP is either directly or indirectly controlled by dynamic recrystallization. For example, the austenite grain size can be controlled by DRX behavior and then converted to fine ferrite during cooling. Many scholars have extensively investigated the DRX characteristics of HSLA steel through isothermal compressive experiments. For example, Wei et al. [11] studied the hot deformation behavior of a medium carbon microalloyed steel and developed the constitutive model and DRX kinetic model of the
steel. Liu et al. [12] investigated the hot deformation behavior of a medium carbon vanadium steel and indicated that the activation energy of hot working at low strain rate is very close to the activation energy of austenite lattice self-diffusion. Zhao et al. [13] researched the effect of deformation parameters on the DRX behavior of a low carbon V-Ni HSLA steel by hot compression tests, and established the DRX kinetic model and constitutive equation of the steel. Akbari et al. [14] proposed a simple constitutive model based on the Hollomon equation, which can accurately predict the flow stress of the medium-carbon microalloyed steel during hot compression. Lan et al. [15] investigated the flow behavior of a low carbon microalloyed steel by hot compression experiment, and the results indicated that DRX may occur even if the peak stress does not appear in the flow stress curves. Li et al. [16] investigated the flow behavior of an HSLA pipeline steel, and established the constitutive model and processing map of the steel.

However, most researches have focused on the constitutive and DRX kinetic models of HSLA steel, with few reporting on the DRX mechanism of HSLA steel under different deformation parameters. In this paper, the effects of deformation parameters on the microstructural evolution of Nb–V–Mo microalloyed HSLA steel during hot compression were studied. Dislocations and grain boundaries after deformation were observed and analyzed through TEM. In addition, to aid further exploration of the evolution mechanism of DRX in the HSLA steel, EBSD analysis was adopted.

2. Experimental procedure

The chemical composition of as-cast HSLA steel is given in table 1. Isothermal compression experiments were conducted on a thermal mechanical simulator (MMS-200) at a temperature range of 800 °C–1100 °C and a strain rate range of 0.1–10 s\(^{-1}\). The MMS-200 control system and compression test area are illustrated in figures 1(a) and (b). The hot compression test process of HSLA steel is illustrated in figure 2. Cylindrical specimens (28 mm × 15 mm) were prepared for the hot compression test. All specimens were heated to 1250 °C at a rate of 20 °C s\(^{-1}\) and held for 5 min to obtain uniform microstructure before compression, then cooled to test temperatures (800, 900, 1000 and 1100 °C) at a rate of 5 °C s\(^{-1}\) and held for 30 s at the deformation temperature to eliminate the temperature gradient of the specimens. Subsequently, all the specimens were deformed to a true strain of 0.65 with the strain rates being 0.1 s\(^{-1}\), 1 s\(^{-1}\) and 10 s\(^{-1}\), respectively. After deformation, in order to retain the high temperature microstructure, all the specimens were immediately cooled with water.

After deformation, the specimens were cut along the compressed axis. The area used for microstructure observation is shown in figure 3. Metallographic microstructures were observed through an Olympus PME-3 optical microscopy (OM). The cut surface to be used for OM observation was ground, polished and etched in a solution of 4% nital. The evolution of the dynamic recrystallization microstructure was studied by electron backscatter diffraction (EBSD) Technology. The EBSD specimens were firstly mechanically polished and then

| Table 1. Chemical composition of the tested steel (mass percent). |
|-----------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|
| C | Si | Ni | Mn | Mo | Cr | Cu | Al | Nb | Ti | V |
| 0.01 | 0.146 | 0.51 | 1.38 | 0.13 | 0.21 | 0.39 | 0.019 | 0.027 | 0.006 | 0.009 |

Figure 1. MMS-200 thermal simulation testing: (a) MMS-200 control system and (b) Compression test area.
vibration polishing for 3 h. The EBSD test was employed on a scanning electron microscope (Hitachi S3400) equipped with HKL Channel 5 software. The acceleration voltage was 20 kV, and the scanning step size was 1.2 μm. Dislocations and grain boundaries were characterized using a JEM-1200 transmission electron microscope (TEM) at an acceleration voltage of 200 kV. The TEM specimens were machined to thin foils with thickness of 500 μm, then mechanically ground to 40–50 μm thickness and punched into Φ3 mm disks. Thereafter, the disks were further thinned by using an ion-beam thinning method.

3. Results and discussion

3.1. Microstructure evolution during hot deformation

Figure 4 depicts the typical microstructure of the tested steel deformed to a true strain of 0.65 at temperatures of 800, 900, 1000 and 1100 °C and the strain rates of 0.1, 1 and 10 s⁻¹. The results indicate that the deformation temperature has an important effect on the grain size of the deformed specimen. In addition, low deformation temperature might lead to the strain-induced precipitation in microalloyed steels during hot thermomechanical processing [17]. The average grain size of deformed specimens at various deformation parameters was measured using the linear intercept method. As shown in figure 5, the average grain size of specimens increased with a corresponding increase of deformation temperature in the range of 800 °C–1100 °C. However, the effect of strain rate on the grain size of deformed specimen is not obvious. When the specimens were deformed at 1000 °C and 1100 °C, the average grain size decreased slightly with the increase in the strain rate. It can be seen from figure 4 that the specimens have undergone DRX for all deformation conditions. Furthermore, the size of the
DRX grain is largest when the specimen was deformed at 1100 °C and 0.1 s\(^{-1}\). Due to the fact that the moving rate of grain boundary increases with increasing temperature, the DRX is more likely to occur at higher deformation temperatures. In addition, the DRX grains have sufficient time for nucleation and grain growth at a low strain rate. Hence, DRX can be carried out more completely and the grain size of DRX is larger when the specimens are deformed at higher temperatures and lower strain rates\(^{[18–20]}\).

3.2.3.2 Observation of dislocation structure

Figure 6 shows the dislocations and substructures of the deformed specimens at a strain rate of 0.1 s\(^{-1}\) and a temperature of 1000 °C and 1100 °C. As illustrated in figure 6(a), when the steel was deformed at 1000 °C/0.1 s\(^{-1}\), the dislocation density increased extremely and the formation of a complex dislocation structure exhibiting some dark region. Dislocation lines were tangled including some subgrains which can be found in a region. Sun et al\(^{[21]}\) reported that the subgrain size increased with a corresponding increase of deformation temperature and decreased with the increase of strain rate. At higher deformation temperatures, the mobility of atoms is increased which means that the elimination and recombination of the dislocations are more complete,
and the subgrains will grow [21]. It is noted that the subgrain near the grain boundary being to be DRX nucleus [18]. When the steel was deformed at 1100 °C/0.1 s⁻¹, the evolution of dislocations during hot deformation can be seen in figure 6(b). It was found that dislocation lines were entangled and dislocation walls were formed in grains. In addition, a matrix with high dislocation density was also observed in some areas.

When the steel deformed at 1100 °C/1 s⁻¹, the dislocations and substructures in the deformed specimen are displayed in figure 7. Figure 7(a) depicts that the microstructure is composed of dislocation tangles and the matrix contains high dislocation density regions (dark region in figure 7(a)). The high dislocation density is associated with the work hardening during the early stages of deformation and the DRX nucleus is in the incubation period of microstructure evolution. Besides, the bulged grain boundary can be observed in figure 7(b). The DRX nucleus was formed by the prior grain boundary locally bulging out and the bulged grain boundary can be regarded as the nuclei of the DRX grain. This nucleation mechanism of DRX is called grain boundary bulging mechanism [22, 23].

Figure 6. TEM bright field images of deformed specimens at (a) 1000 °C, 0.1 s⁻¹, (b) 1100 °C, 0.1 s⁻¹.

Figure 7. TEM morphologies of the deformed specimen at 1100 °C and strain rate 1 s⁻¹ showing (a) staking faults and (b) grain boundary bulging feature.
3.3. EBSD analysis at different strain rates

In order to further investigate the effect of the strain rate on the DRX behavior of the tested steel during isothermal compression, EBSD experiments were carried out on the deformed specimens with deformation parameters of 1000 °C/0.1 s⁻¹ and 1000 °C/10 s⁻¹. The EBSD maps of the tested steel at the same temperature and different strain rates are presented in figure 8. Figures 8(a) and (c) is the crystal orientation maps of the deformed specimens at 1000 °C/0.1 s⁻¹ and 1000 °C/10 s⁻¹, respectively. It can be seen that a large number of grains were elongated, and a few fine DRX grains were observed at the original grain boundaries. Figures 8(b) and (d) is the grain boundary misorientation angle distribution maps corresponding to the crystal orientation maps. The results indicated that the fraction of low angle boundaries (LAGBs) gradually increased with the increase of strain rate. When the strain rate increased from 0.1 s⁻¹ to 10 s⁻¹, the corresponding fraction of LAGBs increased from 46.6% to 61.9%. At a higher strain rate, there was not enough time for the dislocation substructure to be annihilated and rearranged because the effective deformation time was reduced. Therefore, the fraction of LAGBs increased with the increase of strain rate. In contrast, at a low strain rate (0.1 s⁻¹), the dislocation and substructure was consumed gradually with the nucleation and grain growth of DRX grains. Therefore, the decrease in the strain rate also caused the fraction of LAGBs to be reduced. In addition, when the strain rate decreased from 10 s⁻¹ to 0.1 s⁻¹, the corresponding average subboundary misorientation (θAV) increased from 13.8° to 18.8°. According to the [23–25], the increase in θAV during hot deformation is an essential feature of continuous dynamic recrystallization (CDRX). Metals with high stacking fault energy, such as ferritic steel, β-titanium alloy and aluminum, etc, usually undergo CDRX during high temperature deformation [26]. CDRX is featured for nuclei growth by the transformation of LAGBs into HAGBs [27]. At a low strain rate (0.1 s⁻¹), the LAGBs continuously absorbed dislocations and gradually transformed into HAGBs, which was consistent with an increase in the orientation angle fraction in the range of 50°–60°, as shown in figure 8(b). The low strain rate was favorable for the nucleation and grain growth of DRX. Therefore, θAV increases with decreasing strain rate.

3.4. Analysis of Vickers hardness after hot deformation test

The effect of deformation temperature on Vickers hardness of the specimen is shown in figure 9. The error bars (in figure 9(a)) represent a standard deviation of the data points for each specimen. The results show that the Vickers hardness increases slightly with a corresponding increase of deformation temperature in the range of
800 °C–1100 °C. This is attributed to the increase in work hardening due to an increase in dislocation density in the specimens. A comparison with the deformed specimens at different deformation conditions would show that the highest hardness value was 174 HV while the lowest of this condition was 149 HV. Besides, the increase of strain rate takes a small effect on the hardness in all condition tests. A contour map showing the distribution of hardness values of test steel is given in figure 9(b).

4. Conclusions

In this work, the flow behaviors of Nb–V–Mo microalloyed steel were studied using isothermal compression tests at temperatures of 800 °C–1100 °C and strain rates of 0.1–10 s⁻¹, the main results are as following:

1. The deformation temperature has a significant effect on the grain size of the deformed specimen. The average grain size of deformed specimens increased with a corresponding increase in temperature in the range of 800 °C–1100 °C. When the specimens were deformed at 1000 °C and 1100 °C, the average grain size decreased slightly with the increase of strain rate.

2. For the tested steel, the bulged grain boundary was observed at 1100 °C/1 s⁻¹ by TEM. The fraction of LAGBs increased with a corresponding increase in strain rate and the θ_AV decreased with increasing strain rate. When the strain rate increased from 0.1 s⁻¹ to 10 s⁻¹, the fraction of LAGBs increased from 46.6% to 61.9%, and the θ_AV decreased from 18.8° to 13.8°. The CDRX mechanism was determined through EBSD analysis.

3. The Vickers hardness of the deformed specimens increased slightly with a corresponding increase of deformation temperatures in the range of 800 °C–1100 °C. A comparison with the deformed specimens at different deformation parameters would show that the highest hardness value was 174 HV, while the lowest was 149 HV.

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References

[1] Ouchi C 2001 Development of steel plates by intensive use of TMCP and direct quenching processes ISIJ Int. 41 542–53
[2] Beidokhti B, Koukabi A H and Dolati A 2009 Effect of titanium addition on the microstructure and inclusion formation in submerged arc welded HSLA pipeline steel J. Mater. Process. Technol. 209 4027–35
[3] Chen X and Huang Y 2015 Hot deformation behavior of HSLA steel Q690 and phase transformation during compression J. Alloy.
Compsd. 619 564–71
[4] Ledermann C, Pratelli H I, Webster R F, Eizad jou M, Ringer S P and Primig S 2020 Microalloying effects of Mo versus Cr in HSLA
steels with ultrafine-grained ferrite microstructures Mater. Des. 185 108278
[5] Qiao G Y, Xiao F R, Zhang X B, Cao Y B and Liao B 2009 Effects of contents of Nb and C on hot deformation behaviors of high Nb X80
pipeline steels Trans. Nonferrous Met. Soc. China 19 1395–9
[6] Xiao F, Zhao M, Shan Y, Liao B and Yang K 2004 Processing of ultralow carbon pipeline steels with acicular ferrite J. Mater. Sci. Technol.
20 779–81
[7] Jonas J J 1994 Dynamic recrystallization—scientific curiosity or industrial tool? Mater. Sci. Eng. A 184 155–65
[8] Stewart G R, Jonas J J and Montheliet F 2004 Kinetics and critical conditions for the initiation of dynamic recrystallization in 304
stainless steel ISIJ Int. 44 1581–9
[9] Mirzadeh H, Cabrera J M, Prado J M and Najafizadeh A 2011 Hot deformation behavior of a medium carbon microalloyed steel Mater.
Sci. Eng. A 528 3876–82
[10] Doherty R D, Hughes D A, Humphreys F I, Jonas J J, Jensen D J, Kassner M E, King W E, Mcneely T R, Mcqueen H J and Rollett A D
1997 Current issues in recrystallization: a review Mater. Sci. Eng. A 238 219–74
[11] Wei H L, Liu G Q, Xiao X and Zhang M H 2013 Dynamic recrystallization behavior of a medium carbon vanadium microalloyed steel
Mater. Sci. Eng. A 573 215–21
[12] Zhao H, Liu G and Xu L 2013 Rate–controlling mechanisms of hot deformation in a medium carbon vanadium microalloy steel Mater.
Sci. Eng. A 559 262–7
[13] Zhao B C, Zhao T, Li G Y and Lu Q 2014 The kinetics of dynamic recrystallization of a low carbon vanadium-nitride microalloyed steel
Mater. Sci. Eng. A 604 117–21
[14] Akbari Z, Mirzadeh H and Cabrera J M 2015 A simple constitutive model for predicting flow stress of medium carbon microalloyed
steel during hot deformation Mater. Des. 77 126–31
[15] Lan L Y, Zhou W and Misra R D K 2019 Effect of hot deformation parameters on flow stress and microstructure in a low carbon
microalloyed steel Mater. Sci. Eng. A 756 18–26
[16] Li N, Kingkam W, Han R, Huang Y, Li Y, Zhu Y, Bao Z, Zhang H and Zhao C 2019 Processing maps for hot working of HSLA pipeline
steel Mater. Res. Express 6 1265g2
[17] Nasriz, Ghaemifar S, Najafizadeh M and Mirzadeh H 2020 Thermal mechanisms of grain refinement in steels: a review Met. Mater.
Int. (https://doi.org/10.1007/s12540-020-00700-1)
[18] Chen X M, Lin Y C, Chen M S, Li H B, Wen D X, Zhang J L and He M 2015 Microstructural evolution of a nickel-based superalloy
during hot deformation Mater. Des. 77 41–9
[19] Jiang H, Zeng S, Zhao A, Ding X and Dong P 2016 Hot deformation behavior of β phase containing γ-TiAl alloy Mater. Sci. Eng. A 661
160–7
[20] Zhou M, Lin Y C, Deng J and Jiang Y Q 2014 Hot tensile deformation behaviors and constitutive model of an Al–Zn–Mg–Cu alloy
Mater. Des. 59 141–50
[21] Sun Z C, Wu H L, Cao J and Yin Z K 2018 Modeling of continuous dynamic recrystallization of Al–Zn–Cu–Mg alloy during hot
deformation based on the internal-state-variable (ISV) method Int. J. Plast. 106 73–87
[22] Kamei K, Maehara Y and Ohmori Y 1986 Effect of stacking fault precipitation on hot deformation of austenitic stainless steel ISIJ Int. 26
159–66
[23] Huang K and Loge R E 2015 A review of dynamic recrystallization phenomena in metallic materials Mater. Des. 111 548–74
[24] Li X, Xi W, Yan H, Chen J, Su B, Song M, Li Z and Li Y 2019 Dynamic recrystallization behaviors of high Mg alloyed Al-Mg alloy during
high strain rate rolling deformation Mater. Sci. Eng. A 753 59–69
[25] Sakai T, Belyakov A and Miura H 2008 Ultrafine grain formation in ferritic stainless steel during severe plastic deformation Metall.
Mater. Trans. A 39A 2206–14
[26] Gourdet S and Montheliet F 2003 A model of continuous dynamic recrystallization Acta Mater. 51 2685–99
[27] He G, Liu F, Huang L, Huang Z and Jiang L 2016 Microstructure evolutions and nucleation mechanisms of dynamic recrystallization of
a powder metallurgy Ni-based superalloy during hot compression Mater. Sci. Eng. A 677 496–504