Hot Deformation Characteristic and Optimization of Processing Parameters for 0.1C–18Cr–1Al–1Si Ferritic Heat Resistant Stainless Steel

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The hot deformation behavior of 0.1C–18Cr–1Al–1Si ferritic heat resistant stainless steel has been investigated in the temperature range of 900–1100°C and the strain rate range of 0.01–1 s−1 employing compressive tests. The obtained results have shown that the volume fraction and average size of the AlN phases and (Cr,Fe)3C2 carbides decrease with increasing strain rate. The deformation activation energy is calculated as 332.306 kJ·mol−1. Based on the analysis of processing map and microstructure, the optimum processing domain for hot deformation is confirmed as the temperature range of 1045–1100°C and strain rate range of 0.06–1 s−1. [doi:10.2320/matertrans.MT-M2020034]

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1. Introduction

The ferritic heat resistant stainless steel (FHSS) have been increasingly used as high-temperature components of power plant applications, due to their excellent high-temperature resistance and high thermal conductivity together with low thermo-expansion coefficient.1–4 0.1C–18Cr–1Al–1Si FHSS is considered as a candidate for power plant, which is related to the formation of uniform Cr2O3 and Al2O3 layers on its surface to improve the oxidation resistances. Therefore it exhibits good high-temperature properties.5,6 Many studies with regard to 0.1C–18Cr–1Al–1Si FHSS has primarily focused on improving the oxidation resistances.7,8 However, due to 0.1C–18Cr–1Al–1Si FHSS is ferritic at all temperatures, if the processing parameters are not designed properly, the initial as-cast columnar grains may become severely elongated bands during hot deformation, which can lead to the ridging problem and exhibit poor formability.9–12 Furthermore, the investigation of hot deformation behavior of this steel has received much less attention and the deformation mechanism remains unclear, an in-depth comprehension of the relationship between the hot deformation behavior and the processing parameters is vital significant for controlling the microstructure of ferritic stainless steels and improving the final product quality.13,14

The hot processing map based on the dynamic materials model (DMM) which can evaluate the deformation mechanisms and optimize the hot working parameters at various deformation temperatures and strain rates.15–16 It has been successfully applied in different types of ferritic stainless steels. Manmath Kumar Dash et al.17 have identified hot working domains in oxide dispersion strengthened (ODS) 18Cr ferritic steel by using processing map and confirmed that the most favorable processing parameters have been optimized in the temperature range of 1350–1450 K with a strain rate of 0.01 s−1 and 1473 K with a strain rate 0.1 s−1. Zhao et al.18 have investigated hot deformation behavior of 430 ferritic steel by using processing map and their results provide a good reference for the selection of hot working parameters. Liu et al.19 have investigated the hot deformation behavior of a new reduced activation ferritic/martensitic steel named SIMP steel. Based on the processing map and microstructure evolution, they have determined the optimum processing condition for the SIMP steel. Unfortunately, the hot processing map of 0.1C–18Cr–1Al–1Si FHSS is rarely reported, the investigating of hot processing map to optimize the processing parameters and control the microstructures is therefore required.

In this work, the hot deformation behavior of 0.1C–18Cr–1Al–1Si FHSS is systematically investigated by using microstructure evolution analysis, precipitation behavior analysis and Zener-Hollomon parameter. On this basis, the hot processing map is further discussed. The purpose of this study is to clarify the hot rolling schedules, which might improve the final product quality and provide a theoretical guidance for the hot working of 0.1C–18Cr–1Al–1Si FHSS.

2. Experimental Material and Procedures

The 0.1C–18Cr–1Al–1Si FHSS with a chemical composition of 0.09C–1.01Si–0.89Mn–0.007P–0.005S–18.02Cr–0.98Al–0.0122N–Fe mass% was received as a 40 mm-thick plate. It was hot-rolled to a 12 mm thickness hot sheet at 1200°C. The hot compression samples having diameter 10 mm and height 15 mm were machined with their axes parallel to the rolling direction of the hot sheet. The hot compression test was conducted on a Gleeble3800 thermal-mechanical simulator at 900–1100°C and 0.01–1 s−1. As shown in Fig. 1, the samples were firstly heated to 1200°C at a heating rate of 20°C/s and kept for 5 min and then cooled to a setting deformation temperature at a cooling rate of 10°C/s. Secondly, the samples were kept at the setting deformation temperature for 20 s to get a uniform temperature distribution. Finally, all the samples were compressed to a true strain of 0.8 and were quenched immediately to retain its high
temperature deformation microstructure. For optical microscope (OM) analysis and scanning electron microscopy (SEM) analysis, the samples were polished in a standard way and then etched in a solution of 5 g FeCl₃ + 50 ml HCl + 100 ml H₂O at room temperature for 30 s. For TEM analysis, thin foils were firstly polished manually to 50 µm thickness and then polished using twin jet polishing in a solution of 5% perchloric acid and 95% acetic acid bath at about –20°C.

3. Results and Discussion

3.1 The flow behavior during hot deformation

The flow curves recorded in compression tests at various temperatures and strain rates are presented in Fig. 2. It is clearly shown that the deformation temperature and strain rate have the significant effects on the flow stress. The flow stress decreases with increasing deformation temperature and decreasing strain rate. This behavior is because higher temperature can offer higher mobility of dislocation and longer compression time can promote the annihilation and rearrangement of dislocations, nucleation and growth of new grains. In addition, the shape of the flow curves can reflect the microstructure changes during hot deformation. As one can see that the flow curves exhibit pronounced peaks at 0.01 s⁻¹ and 0.1 s⁻¹ under different hot deformation temperatures, which are the typical feature of the dynamic recrystallization (DRX). At 1 s⁻¹/1100°C, the flow curve also exhibits DRX characteristics. While it noted that the flow curves show a slow upward trend at 900°C and 1000°C, which indicate that dynamic recovery (DRV) is the primary softening mechanism. However, there are some studies demonstrate that DRX may occur even if no clearly peaks are observed in the flow stress curves. Furthermore, it is worth noting that clear serration occurs at 1000°C and 1100°C at a strain rate of 1 s⁻¹. The reason may be due either to machine compliance or to a high rate of data acquisition.

3.2 Microstructure evolution and precipitation behavior

Figure 3 shows the microstructure before hot deformation. It can be seen that the distribution of ferrite grains are relatively uniform and the average grain size is 127.3 µm. Moreover, some precipitates can also be seen in the grain boundaries or within grains.

After hot deformation, the optical microstructure at various conditions are shown in Fig. 4. According to the observation of optical microstructure, the microstructures can be initially divided into three groups. Group 1 includes A and D, the deformed ferritic grains are subjected to axial compression, which are obviously elongated along the direction of vertical compression. It indicates that the DRV is the main softening mechanism. Group 2 includes B, C, E, G and H, many
newly formed DRXed grains (the red arrows as shown in B, C and E) and subgrains (the red arrows as shown in G and H) are observed near the ferritic grain boundaries. It indicates that the nucleation of dynamic recrystallization (DRX) is beginning, but the DRX is insufficient and incomplete in these conditions. The main softening mechanism in these conditions maybe partial DRX or DRV. In group 3#, which includes F and I, the complete DRX occurs and the DRXed grains have become coarser. The average grain size is 122.6 µm and 178.1 µm at 1100°C/0.1 s⁻¹ and 1100°C/0.01 s⁻¹, respectively.

Figure 5 shows the SEM micrographs at 1100°C with strain rates of 0.01 s⁻¹ and 0.1 s⁻¹. As can be seen from Fig. 5 that the volume fraction and average size of the precipitates at 0.01 s⁻¹ are larger than that of 0.1 s⁻¹. This is because there is more time available for the formation and growth of precipitates at a lower strain rate.²⁷ The type of precipitates have be testified by the TEM observation, as shown in Figure. The pentagonal precipitate (shows in Fig. 6(a)) is about 500 nm. The EDS from the polygonal precipitate indicated that it was rich in N and Al, and combine the analysis by selected area electron diffraction (SAED) patterns with Z = [100] zone axes suggested that it is AlN phase. The strip shape precipitate (shows in Fig. 6(b)) is riched in Cr, Mn, Fe, and C, and the analysis of SAED patters which is indexed to a complex FCC structure in [112] zone axes, so it can be determined that the strip shape precipitate is (Cr,Fe)₂₃C₆ carbide.

3.3 Establishment of constitutive equation

During hot plastic deformation, the flow stress in a wide range can be related to deformation temperature and stain rate in the form of an Arrhenius kinetic rate equation by Sellars and McEwen:²⁸

\[
\dot{\varepsilon} = A \sinh(\alpha\sigma) \exp\left(-\frac{Q}{RT}\right)
\]

Where \(\dot{\varepsilon}\) is the strain rate (s⁻¹), \(\sigma\) is the (peak or proof) stress (MPa), \(R\) is the gas content (8.314 J mol⁻¹ K⁻¹), \(T\) is
the deformation temperature (K) and \( Q \) is the activation energy (J mol\(^{-1}\)). \( A \) and \( \alpha \) are the material parameters (s\(^{-1}\) and MPa\(^{-1}\)), \( n \) is the stress exponent.

In this paper, the \( \dot{\varepsilon} \) selects the peak stress \( \sigma_p \). The relationship between \( \ln \dot{\varepsilon} - \ln \sigma_p \) and \( \ln \dot{\varepsilon} - \ln \sigma_p \) has been drawn at different deformation temperatures (as shown in Fig. 7). By performing linear regression on the same temperature stress peak and taking the average of the reciprocal of the slope at different temperatures, then get the value of constant \( \alpha \) is 0.0182 MPa\(^{-1}\)\(29\).

Taking the natural logarithm on both sides of the eq. (1), we get:

\[
\ln[\sinh(\alpha \sigma_p)] = \frac{1}{n} \ln \dot{\varepsilon} + \frac{1}{n} \frac{Q}{RT} - \frac{1}{n} \ln A \tag{2}
\]

According to eq. (2) and peak stress, it can be obtained a linear relationship between \( \ln \dot{\varepsilon} - \ln[\sinh(\alpha \sigma_p)] \) and \( \ln[\sinh(\alpha \sigma_p)] - (1/T) \) which shows in Fig. 7. The \( n \) value is the the average slope of liner fit which is found to be 4.39.

The activation energy \( Q \) during hot deformation is calculated by using the peak stress data at different temperatures and strain rates based on eq. (2),\(^{30}\) it reflects the difficulty during the softening process. Partial derivatives of the eq. (2) yield the activation energy \( Q \) which can be expressed as:\(^{31}\)

\[
Q = R \left[ \left. \frac{\partial \ln \dot{\varepsilon}}{\partial \ln[\sinh(\alpha \sigma_p)]} \right|_T \frac{\partial[\ln[\sinh(\alpha \sigma_p)]]}{\partial(1/T)} \right] \dot{\varepsilon} \tag{3}
\]

The \( Q \) value is calculated as the average slope of \( \ln[\sinh(\alpha \sigma_p)] \) against \( 1/T \) (Fig. 8) which is determined as 332.306 kJ·mol\(^{-1}\) in the present study. It is similar to 329–359 kJ·mol\(^{-1}\) measured by the previous study for the activation energy of stabilized 12–27 mass% Cr ferritic stainless steels,\(^{32}\) but lower than 451 kJ·mol\(^{-1}\) obtained by advanced 9Cr–Nb–V ferritic heat-resistant steel.\(^{33}\) The reason for this difference might be the presence of impurity and minor alloying constituents affect diffusivity.\(^{34}\)

The Zener-Hollomon parameter \( Z \) is the deformation rate after temperature correction, which is commonly used to

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**Fig. 5** SEM micrographs of samples deformed at (a) 1100°C and 0.01 s\(^{-1}\) and (b) 1100°C and 0.1 s\(^{-1}\).

**Fig. 6** TEM micrographs of precipitates deformed at 1100°C and 0.1 s\(^{-1}\) (a) AlN phase; (b) (Cr,Fe)\(_2\)C\(_6\) carbide; (c) EDS analysis of AlN phase; (d) EDS analysis of (Cr,Fe)\(_2\)C\(_6\) carbide.
comprehensively consider the influence of deformation conditions on the hot deformation process. 35,36) An expression for the Z parameter during deformation can be obtained:

\[ Z = \dot{\varepsilon} \exp \left( \frac{Q}{RT} \right) \]  

Substituting eq. (1) into eq. (4), and taking the natural logarithm of the obtained results on both sides:

\[ \ln Z = \ln A + n \ln \left[ \sinh(\alpha \sigma_p) \right] \]  

It can be seen from eq. (5), there are linear relationship between \( \ln Z \) and \( \ln \left[ \sinh(\alpha \sigma_p) \right] \). The experimental value basically falls near the regression line which can be seen from the \( \ln Z - \ln \left[ \sinh(\alpha \sigma_p) \right] \) graph (shows in Fig. 9), and the linear correlation coefficient is 0.974, indicating that the experimental data well reflects the meaning expressed by eq. (5). The average intercept of the fitted straight line is \( \ln A = 38.25 \), then get \( A = 1.5 \times 10^{13} \text{s}^{-1} \).

The relationship between peak stress and deformation parameters during hot deformation can be obtained by solving the hyperbolic sine function in eq. (5), as shown in the following equation:

\[ \sigma = \frac{1}{\alpha} \ln \left\{ \frac{Z^2}{A} + \left[ \frac{Z}{A} + 1 \right]^2 \right\} \]  

Substituting the obtained \( A, a, \) and \( n \) values into eq. (6), the function relationship between \( \sigma \) and \( Z \) parameter during hot deformation of 0.1C–18Cr–1Al–1Si FHSS can be obtained:

\[ \sigma = \frac{1}{0.0182} \ln \left\{ \left[ \frac{Z}{1.5 \times 10^{13}} \right]^3 + \left[ \left( \frac{Z}{1.5 \times 10^{13}} \right)^3 + 1 \right] \right\} \]  

and,

\[ Z = \dot{\varepsilon} \exp \left( \frac{332306}{8.314T} \right) \]  

The smaller the \( Z \) value, the stronger the dislocation and the movement ability of the grain boundaries, and the more
opportunities of DRX to occur during hot deformation.\(^{37}\) It can be found in this experiment that at a smaller Z value, such as deformed at 1100°C/0.1 s\(^{-1}\) and 1100°C/0.01 s\(^{-1}\) (shown in Fig. 4 F and I), a completed DRX process occurs in this steel. On the contrary, the larger the Z value, the slower the DRX driving force, and only DRV and partial DRX can occur. Therefore, at a larger Z value, such as deformed at 1100°C and 1000°C/1 s\(^{-1}\) (as shown in Fig. 4 A and B), the microstructure exhibit the DRV and partial DRX characteristics, respectively.

3.4 Processing map

The processing map based on the principle of dynamic material model (DMM) developed by Prasad and Gegel et al.,\(^ {38-40}\) which is used to optimize the processing parameters. It can reflect the internal microstructure changes under different deformation conditions, and intuitively show the rheological instability zone and reasonable processing zone during hot deformation process of the material. Therefore, it can provide a theoretical basis for formulating reasonable metal thermal deformation process parameters. The total power (\(P\)) expended in hot deformation consists of two parts of \(G\) and \(J\), the \(G\) content is representing the heat generated from plastic deformation and \(J\) content is a function of strain rate sensitivity parameter \(m\) and varies with deformation temperature and strain rate. When \(\xi(\dot{\varepsilon}) < 0\), an unstable flow process will occur which should be avoided for processing. The area marked as negative on the power dissipation map is called the instability map. The processing map is constructed by the superimposition of instability map on the dissipation map.

The processing map of the 0.1C–18Cr–1Al–1Si FHSS is shown in Fig. 10. The A to I region in Fig. 10 are the same deformation conditions as Fig. 7. It is noted here that there is no instability domain in the processing map, which indicating the strain rate does not reach the critical condition of complete instability at true strain of 0.8. In addition, it is generally believed that the higher values of the dissipation efficiencies are favorable for hot processing of materials.\(^ {44}\) According to Fig. 10, the processing map can be considered as four domains. Based on the processing map and the

\[
\eta = \frac{J}{J_{\text{max}}} = \frac{2m}{m + 1} \quad (13)
\]

where \(J_{\text{max}} = \xi = \frac{\dot{\varepsilon}}{\dot{\varepsilon}_0}\) when \(m = 1\).

The power dissipation map consists of the variation of \(\eta\) with temperature and strain rate which can be directly related to a particular microstructure mechanism at different domains. During hot deformation, the DRX, DRV and superplasticity are the safe softening mechanisms, while wedge cracks and void formation are the unsafe softening mechanism.\(^ {41,42}\) Further, based on the dynamic material model theory, Ziegler combines the maximal principle of irreversible thermodynamics with large strain plastic theory to obtain the rheological instability criterion under certain deformation conditions:\(^ {43}\)

\[
\xi(\dot{\varepsilon}) = \frac{\partial \ln \left( \frac{m}{m + 1} \right)}{\partial \ln (\dot{\varepsilon})} + m < 0 \quad (14)
\]
analysis of microstructure and precipitates, the optimization of processing parameters is discussed as follow.

Domain #1 situates at temperature range of 900–1020°C corresponding to the strain rates of 0.01–0.16 s⁻¹ and it has the power dissipation efficiency of 5–35%. The microstructure of the samples in this study are deformed at 900°C/0.1 s⁻¹, 900°C/0.01 s⁻¹ and 1000°C/0.01 s⁻¹, which are marked as D, G and H in Fig. 10, and the elongated grains or insufficient DRX grains can be observed.

Domain #2 situates at temperature range of 900–1015°C corresponding to the strain rates of 0.06–1 s⁻¹ and it has the power dissipation efficiency of 40–50%, which is higher than that of domain #1. According to the analysis of microstructure, the samples deformed at 900°C/1 s⁻¹ (marked A in Fig. 10) are the elongated grains, and insufficient DRX grains can be observed at 1000°C/1 s⁻¹ and 1000°C/0.1 s⁻¹ (marked B and E in Fig. 10), but the DRX is not completed and there still exists unDRX grains.

Domain #3 situates at the temperature range of 1025–1100°C and the strain rates of 0.01–0.05 s⁻¹, it has the power dissipation efficiency of 30–35%. From the microstructure observation, it can be found that DRX has occurred at 1100°C/0.01 s⁻¹ (marked I in Fig. 10) which average grain size is about 178.1 µm.

Domain #4 situates at the temperature range of 1045–1100°C and the strain rates of 0.06–1 s⁻¹. It is a preferred domain which has the highest peak power dissipation efficiency (50%). As mentioned in Fig. 4, many newly formed DRXed grains are observed at 1100°C/1 s⁻¹ (marked C in Fig. 10), the rate is 0.1 s⁻¹ (marked F in Fig. 10), complete DRX has occurred and the average grain size is about 122.6 µm. The grain size at 1100°C/0.1 s⁻¹ is finer than that of domain #3 with 1100°C/0.01 s⁻¹. Furthermore, according to the analysis of precipitates, the volume fraction and average size of the AlN phases and (Cr,Fe)₂₃C₆ carbides at 1100°C/0.01 s⁻¹ are larger than that of 1100°C/0.1 s⁻¹. The coarsening (Cr,Fe)₂₃C₆ carbides and AlN phases would lead to a poorly mechanical properties.⁵⁻⁷⁻⁴⁷⁻

Combining with the above analysis from the processing map, microstructure evolution and precipitation behavior, the preferred domain for hot deformation should be chosen in the temperature range of 1045–1100°C and strain rate of 0.06–1 s⁻¹, where the material will receive much more DRX grains and good mechanical properties.

4. Conclusion

The hot deformation behavior of 0.1C–18Cr–1Al–1Si ferritic heat resistant stainless steel has been studied by hot compressing in the temperature range of 900–1100°C and the strain rate range of 0.01–1 s⁻¹. The flow behavior, microstructure evolution and precipitation behavior are analyzed. The processing map based on the principle of dynamic material model (DMM) is developed to optimize the processing parameters. The following are the main results:

(1) The lower strain rate and higher deformation temperature are beneficial for the occurrence of DRX. The volume fraction and average size of the AlN phases and (Cr,Fe)₂₃C₆ carbides are decreased with the increase of strain rate.

(2) The deformation activation energy of 0.1C–18Cr–1Al–1Si FHSS is calculated as 332.306 kJ·mol⁻¹. The constitutive equation can be expressed as:

\[
\sigma = \frac{1}{0.0182} \ln \left( \frac{Z}{1.5 \times 10^{13}} \right)^{\frac{1}{\gamma}} + \left( \frac{Z}{1.5 \times 10^{13}} \right)^{\frac{1}{\gamma}} + 1 \right)^{\frac{1}{\gamma}}
\]

and \( Z = \dot{\varepsilon} \exp \left( \frac{332306}{8.3147} \right) \).

(3) There is no instability domain in the processing map of 0.1C–18Cr–1Al–1Si FHSS. The optimum processing domain for hot deformation is confirmed as the temperature range of 1045–1100°C and strain rate range of 0.06–1 s⁻¹.

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