PAPER

Analysis of flow stress and microstructure evolution of 9310 steel during the hot compression

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Abstract

Due to the low-cost and high-strength of carburizing 9310 steel, it has become widely used in the manufacturing industry especially gears and shafts manufacturing. Hence, this paper investigates the hot deformation behavior and microstructure evolution of the 9310 steel under hot compression conditions. With the help of a combination of standard compression testing, optical microstructure and electron backscatter diffraction (EBSD) observation experiment, the dynamic recrystallization (DRX) and dynamic recovery (DRV) mechanisms of the 9310 steel under compressive stress were determined and the constitutive equation model was also identified. It was found that the peak stress level, steady flow stress, dislocation density and number of substructures of the alloy increases with the decreasing of deformation temperature and the increasing of strain rate. Conversely, the high angle grain boundary area becomes larger, the grain boundary forms a serrated shape and the DRX in the alloy occurs. This comprehensive characterization of stress and phase transformation could enable a precise control of the microstructures of 9310 steel, and hence its properties.

1. Introduction

The increasing demand for gears drive in the fields of aircraft, high-speed rail, automobile and robot industries leads to more rigorous requirements of employed materials with respect to their surface integrity, geometric and physical performances, and fatigue life [1–4]. The 9310 steel is an early and representative type of carburized alloy structural steels that is applied in the aviation bearing gear, which has the characteristics of high strength, good plasticity and toughness, and relatively low price. Therefore, 9310 steel is a perfect type of aviation shaft material that mainly used in manufacturing the transmission structural components such as gears, gear shafts, and tail propeller shafts. At present, with the wide application of vacuum induction melting-vacuum arc remelting (VIM-VAR) double vacuum smelting technology on steel smelting, the existence of harmful elements in 9310 steel is greatly reduced, the non-metallic inclusions are effectively controlled, and the purity of steels is significantly improved. However, in the process of high temperature carburizing or deformation, the grain coarsening is easy to occur due to the high operating temperature and long processing period, which lead to material performance degradation. In high-strength steel, ultra-purity, ultra-refinement and high homogeneity have always been the goals pursued by researchers. While improving the purity of steel, making full use of the role of alloying elements can not only stabilize the strength of steels but also improve the toughness of steels [5, 6]. In recent years, a few achievements have been made in the research of 9310 steel. For instance, Li Yong [7, 8] investigated the transformation law of 9310 steel microstructure after austenitizing through the CCT curve. Zhang Xi [9] studied the influence of chemical composition fluctuation on the hardenability and mechanical properties of 9310 steel, and consequently determined the internal control range of its composition. Wu Qiuping [8] explored the mechanical properties variation and microstructure evolution of 9310 steel under different tempering temperatures.

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Table 1. Chemical composition of 9310 steel (wt. %).

| Element | C   | Cr  | Fe  | Mn  | Mo  | Ni  | P  | Si  | S  |
|---------|-----|-----|-----|-----|-----|-----|----|-----|----|
| C       | 0.08| 1.31| 94.3874 | 0.58 | 0.10 | 3.33 | 0.0026 | 0.20 | 0.010 |

Generally, the finite element method (FEM) analysis is an indispensable method, but the simulated results usually have a gap compared with the actual results. The predictability of the manufacturing simulation process highly depends on the accuracy of constitutive models to describe the mechanical behavior of materials [10, 11], which has guided the significance for the analysis and evaluation of forming characteristics and forming processes of metal materials [12–14]. A group of scholars have studied the material plastic flowability through compression, tensile, torsion and other analytical methods at elevated temperatures [15–20]. Furthermore, they have established the constitutive equations of materials based on the experimental data. By applying the material constitutive equation, the relationships between stress σ and strain ε, strain rate ˙ε and temperature T have been accurately predicted for various materials such as AZ31B magnesium alloy [21], 22MnB5 [22], TC4 alloy [23], 6063 aluminum alloy [24], X20Cr13 martensitic stainless steel [25], and Ti-Al alloy [26].

However, constitutive studies on the 9310 steel are still limited up to date. The aim of this paper is to get the Arrhenius constitutive equation of 9310 steel at high temperatures. Isothermal compression experiments of 9310 steel were conducted using the Gleeble-3180 thermal simulator under different temperatures and strain rates. Then experimentally obtained isothermal stress-strain curves were parametrically solved using the Arrhenius model. Subsequently, the effects of deformation temperature and strain rate on flow stress were analyzed. As controlling the microstructure to achieve a uniform and appropriate grain size is a challenging task, this paper investigates the grain size variation and microstructure evolution during the hot deformation process using optical microscopy and electron backscattering diffraction (EBSD) devises.

2. Experimental procedure

2.1. Material and equipment
The industrial grade 9310 aviation gear steel was chosen in this study, where its chemical composition is listed in table 1. For the compression tests, 18 small cylindrical samples were prepared with a diameter of 8 mm and length of 12 mm at a very slow speed to minimize the heat effects and shear stress, accordingly ensuring that the retained austenite was not inadvertently transformed as the samples were prepared. The experimental apparatus and samples before and after compression are shown in figure 1. Meanwhile, the oxide layer of the workpiece surface was removed to reduce the impact of impurities.

Gleeble 3180 thermal-mechanical test simulator can execute various simulation tests such as rolling and forging processes, continuous casting and smelting processes, welding process, metal heat treatment as well as mechanical thermal fatigue. During the testing process, it can control the temperature range and testing speed, and record the changes of temperature, force, strain, stress and other parameters.

2.2. Hot compression experiment process
The isothermal thermal compression simulation test was performed on the Gleeble 3180 thermal simulation test machine. Deformation conditions such as temperature, displacement and speed are automatically controlled by computer system programs. And true stress and true strain data are automatically collected correspondingly. Once the test is completed, the system automatically uses the Origin drawing software to plot the true stress-true strain curves under different deformation conditions according to the program.

The temperature monitoring and feedback control of the system are carried out by welding thermocouples at the middle and high points of sample’s surface. Tantalum and nickel-based lubricant are used for lubrication, where a graphite sheet is applied as the lubricant between the sample and the anvil to reduce friction.

As shown in figure 2, during the experiment, the sample was first heated to the temperatures of 900 °C, 1000 °C and 1150 °C, respectively, at a rate of 5 °C s⁻¹, and maintained for one minute to achieve uniform temperature distribution of the entire sample. The sample was compressed at predetermined strain rates of 0.01 s⁻¹, 0.1 s⁻¹ and 1 s⁻¹ with compression rate of 0.8 [27]. Once the predetermined strain is reached, the compressed specimen is water quenched to freeze its microstructure for the subsequent research. Finally, the test was repeat twice under similar conditions until the difference of peak stresses between the true stress-strain curves are within 5%–7%.
3. Results and discussion

3.1. Microstructure analysis

After the hot compression test, the plate and lath martensite and retained austenite structure of compressed specimens were observed by the optical microscopy images, as illustrated in figure 3. When the strain rate is \(0.01 \text{s}^{-1}\), the alloy’s high dislocation density and dislocation tangles are larger than the higher strain rate marked by the red arrows, as shown in figures 3(a) and (b). The possible reasons are that the precipitation of proeutectoid ferrite is more in high temperature area and the size of the precipitated phase is larger when the investigated material is a low carbon steel. When the strain rate is increased to \(0.1 \text{s}^{-1}\), the deformation in the high-temperature region is obvious, so more martensite nuclei may be produced in the medium and high temperature region. The more laminae energy is stored in low-temperature region, the more martensite is likely to be produced. The grains are refined owing to many defects, indicating that the main mechanism of softening during the initial stage of hot compression is a DRV [28]. However, since the disappearance of dislocation in DRV process is far lower than the newly generated dislocations during the hot compression, the work hardening plays a leading role in the steel compression process. Due to the small dislocation size, the recrystallization is incomplete DRX and the softening effect is correspondingly small [29, 30].

At a higher deformation temperature and a lower strain rate, atom diffusion capacity, dislocation cross slip and grain boundary migration can be enhanced, which is conducive to the nucleation and grain boundary growth of DRX [31]. As shown in figure 3(c), after the specimen was compressed at 1000 °C and \(0.1 \text{s}^{-1}\), the original crystal becomes fine and long, followed with appearance of a thin equiaxial grains. The results indicate that the main deformation mechanism of 9310 steel under this condition is a DRV without complete DRX. As shown in figure 3(b), when the thermal compression was executed at the deformation temperature of 1150 °C and low strain rate of \(0.01 \text{s}^{-1}\), the original grain boundary presents a jagged shape and the grain boundary with large angle significantly increases. A large number of fine recrystallized grains can be observed near the grain boundary, which indicates that the complete DRX occurs under these conditions.
The EBSD observation result of 9310 steel hot compression sample is shown in figure 4. The grain size range is 0.1–33 μm, and the average grain size is 18.42 μm. Compared to the initial microstructure, a significant refinement occurs, which is consistent with the results obtained by optical microscope. The orientation imaging results indicate the existence of a strong texture in the sample, where the orientation of the majority of grains is \( \langle 101 \rangle \), and the orientations of a few grains are \( \langle 111 \rangle \) and \( \langle 001 \rangle \), respectively. Even if they are not \( \langle 100 \rangle \) or \( \langle 001 \rangle \) oriented grains, the grain orientation has also been shifted to the line between \( \langle 101 \rangle \) and \( \langle 111 \rangle \), and most of the grains in the orientation image are formed long strips with a large number of sub boundaries. It can be seen that in the process of hot forging, there must be enough large strain to achieve the purpose of grain refinement, and a strong texture will be produced, which leads to the anisotropy of gear performance.

The practice shows that the analysis of orientation difference distribution of polycrystalline can play an important role in microstructure analysis. In polycrystal, the conventional grain interface usually has no specific characteristic, but the magnitude of orientation difference is different. Therefore, different conventional grain boundaries can be simply distinguished by the size of misorientation angle \( \delta \). The \( \delta \) can be divided into two categories: When \( \delta \) is very small (usually less than 15°), it is called small angle grain boundary; when \( \delta \) exceeds 20°, it is called the large angle grain boundary.

The misorientation angle determines many basic microstructure parameters, such as grain size, grain shape and texture. Yield, creep, fracture and corrosion of materials are closely related to the misorientation angle [32]. From the EBSD orientation distribution of 9310 steel in figure 5, it can be seen that most of the grain boundaries observed in 9310 steel are small angle boundaries after compression at 900 °C–1150 °C. The proper combination of dislocations in the crystal can form a small angle grain boundary. The small angle grain boundary is composed of some edge dislocations which are parallel to each other along the longitudinal direction [33]. The arrangement of edge dislocations makes the grain boundary in a low energy stable state, which is not easy to result in the thermal activation and migration of the grain boundary. As a consequence, the existence of small angle grain boundary can improve the strength, hardness and toughness of gears, and stabilize its surface properties.

Figure 3. Optical microstructures of the hot deformed 9310 steel samples under different conditions: (a) 0.01 s⁻¹–1000 °C, (b) 0.01 s⁻¹–1150 °C, (c) 0.1 s⁻¹ – 1000 °C, and (d) 0.1 s⁻¹–1150 °C.
3.2. Flow stress curve

Figures 6(a)–(c) demonstrate the stress-strain curves obtained at 900 °C, 1000 °C and 1150 °C, respectively. It can be observed from figure 6(d) that the flow stress is proportional to the strain rate and inversely proportional to deformation temperature. These curves display two distinct characteristics: (1) in the initial deformation stage, the stress rises sharply due to the dominant role of work hardening; (2) with the increase of strain, a
The balance between work hardening and strain softening is caused by dislocation rearrangement and annihilation during the softening of DRV and DRX, leading the flow stress to be stabilized. The high-temperature deformation of metal is a thermal activation process. The rises of deformation temperature enhance the thermal activation process, which reduce the density of vacancies generated by the deformation [34]. At this point, the movement of dislocations is the cross slip of spiral dislocations and the climbing of edge dislocations. The temperature rise resulted by thermal activation can immediately cause a recovery phenomenon. During the recovery process, the flow stress macroscopically decreases. After undergoing a DRV phase, the metal enters recrystallization transformation. Thermal activation determines the process of nucleation and recrystallization, so small changes in temperature will greatly change the time required for completing recrystallization. With the increases of temperature, the nucleation rate and grain growth rate of DRX increase, whereas the dislocation density and flow stress decrease dynamically. During the compression process, the shape of the corresponding curves is similar. As the amount of deformation increases, the stress gradually rises, and then gradually decreases after reaching the peak stress until it reaches a stable state. In this experiment, the stress-strain curve is sensitive to the strain rate. Consequently, the compressive stress gradually increases as the strain rate becomes larger. However, under the conditions of 900 °C, 1150 °C and 1 s⁻¹, the stress after yielding still continue its upward trend until reaching the peak stress, as a consequence of the initial hardening effect after yielding being greater than softening effect. As the stress reaches its peak, the hardening and softening effects tend to be flat, and then maintain the steady-state flow form. At the temperature of 1150 °C and the strain rate of 0.01 s⁻¹, the curve after yielding has a slightly decreasing trend of the flow form, indicating that DRX has occurred at a high temperature and a low strain rate. Coupled with DRV, the softening effect of the material after the yield point begins to be greater than the hardening effect, and the flow curve shows an oblique downward migration on the macro.

4. Constitutive equation

During thermal deformation, the Arrhenius equation is widely used to describe the dependence of flow stress on temperature and strain rate [35, 36]. This equation can be written as:
\[ \dot{\varepsilon} = \frac{Q}{RT} \]

where \( A \) is constants, \( Q \) is the thermal deformation activation energy, \( R \) is the gas constant, \( T \) is the absolute temperature, and \( \sigma \) is the maximum flow stress of the curve.

At low stress \((\alpha \sigma > 1.2)\) condition, equation (1) can be expressed as

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

At high stress \((\alpha \sigma > 1.2)\) condition, equation (1) can be expressed as

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

The hyperbolic sine Arrhenius equation obtained by the above equation is applicable to various stress states [37]:

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

where \( \alpha \) is a constant independent of the deformation temperature, \( n \) and \( n_1 \) represent the strain rate sensitivity index related to the hardening and softening behavior of the material, respectively.

Taking the natural logarithm of both sides of equations (2) and (3), they can be expressed as:

\[ \ln \sigma = \frac{1}{n_1} \ln \dot{\varepsilon} - \frac{1}{n_1} \ln A + \frac{1}{n_1} * \frac{Q}{RT} \]

\[ \sigma = \frac{1}{\beta} \ln \dot{\varepsilon} - \frac{1}{\beta} \ln A - \frac{Q}{RT} \]

Obtained by linear fitting of flow stress according to figure 7, \( n_1 = 6.418348 \), \( \beta = 0.0677702 \), \( \alpha = \beta / n_1 = 0.010558824 \).

Finding the partial derivative of the strain rate on both sides of equation (4), we obtain:

\[ \ln [\sin h(\alpha \sigma)] = \frac{1}{n} \ln \dot{\varepsilon} - \frac{1}{n} \ln A + \frac{1}{n} * \frac{Q}{RT} \]

It is easy to get \( n = 4.754885645 \).

When the deformation temperature is constant, the heat activation energy for deformation is obtained as:

\[ Q = R \frac{\partial \ln \dot{\varepsilon}}{\partial \ln [\sin h(\alpha \sigma)]} \frac{\partial \ln [\sin h(\alpha \sigma)]}{\partial (1/T)} \]

Therefore, for a constant temperature, \( Q = 581.778 \text{ KJ mol}^{-1} \) is obtained by the slope of the curve \( \ln (\dot{\varepsilon}) - \ln [\sin h(\alpha \sigma)] \) multiply the slope \( \ln [\sin h(\alpha \sigma)] - 1/T \). For a constant strain rate and the gas constant \( R = 8.314 \text{ J mol}^{-1} \), so that the value of \( A \) is \( 1.069773 \times 10^{29} \).

Taking the above results into equation (4), the thermal deformation equation of 9310 steel is

\[ \dot{\varepsilon} = 1.069773 \times 10^{29} \times \sinh (0.010558824 \sigma)^{4.754885} \exp \left( \frac{581778}{8.314T} \right) \]

In order to verify the accuracy of the model, we substituted the obtained constitutive model into DEFORM for forging simulation. Figure 8 shows the comparisons between the experimental and simulated flow stress values. A line inclined at 45° from the horizontal is drawn in figure 8. Theoretically, all the points should lie on this line and the correlation coefficient is 0.97005. The values of MARE and RMSE are 1.295797% and 11.7668, respectively. In conclusion, the simulated and calculated results agree well with the experimental ones.

The \( Z \) parameter (the Zener-Hollomon factor) is widely used to represent the comprehensive effect of deformation temperature and strain rate on the deformation process. In the thermal deformation process, the relationship between the deformation temperature \( T \) with the deformation rate \( \dot{\varepsilon} \) and the \( Z \) parameter is described as follows [38]:

\[ Z = \dot{\varepsilon} \exp \left( \frac{Q}{RT} \right) = A \sin h(\alpha \sigma)^n \]
Based on the obtained activation energy $Q$ for thermal deformation, the parameter $Z$ of the test steel under different deformation conditions can be calculated. Figure 9 shows that with the rise of temperature and strain rate, the value of $\ln Z$ multiplies, and the peak stress of thermal deformation of the test steel multiplies accordingly.

5. Conclusions

The flow stress and microstructure evolution during the hot compression testing of 9310 steel were studied at a temperature range of 900 °C to 1150 °C and a strain rate range of 0.01 s$^{-1}$ to 1 s$^{-1}$ by using a combination of
standard compression testing and microstructure observation, where the following conclusions can be listed as follows:

1. Deformation temperature, strain and strain rate have a significant influence on DRX. The DRV of 9310 steel can be effectively induced by optimizing thermal deformation parameters, and the microstructure can be further improved. At a given strain rate, the flow stress reduces significantly with the rise of deformation temperature.

2. The thermal activation become higher due to the decrease of critical shear stress and the improvement of grain boundary mobility at higher deformation temperatures. Higher deformation temperature or higher strain could easily lead to DRX. At low temperature and high strain rate, DRV is the main softening mechanism. At deformation temperature of 1150 °C and low strain rate of 0.01 s⁻¹ (high ln Z), the 9310 steel has the best structural properties with the smallest grains.

3. In the process of hot forging, enough large strain is needed to achieve the purpose of grain refinement, then a strong texture will be produced, which leads to the anisotropy of material performance.

4. The high-temperature flow stress constitutive model of 9310 steel is established in the form of Arrhenius hyperbolic sine function including the deformation temperature and strain rate:

\[
\dot{\varepsilon} = 1.069773 \times 10^{29} \times \left[ \sinh \left( 0.010558 \sigma \right)^{4.754885} \exp \left( -\frac{581778}{8.314T} \right) \right]
\]

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**Data availability statement**

The data that support the findings of this study are available upon reasonable request from the authors.

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