Obtaining ultrafine spheroidized carbides by combining warm deformation with divorced eutectoid transformation in GCr15 bearing steel

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Abstract

In this work, the ultrafine spheroidized carbides were obtained by combining warm deformation with the divorced eutectoid transformation (DET) in GCr15 bearing steel. The results show that the temperature range of DET is approximately 730 °C to 700 °C at a cooling rate of 0.05 °C s−1. It is also found that excellent spheroidized microstructure can be obtained and less deformation resistance can be generated when the deformation is carried out at 720 °C with a strain rate of 0.5 s−1. This result could be ascribed to the fact that deformation provides the driving force for transformation, dynamic recrystallization (DRX) and reduced dislocation density through the coordination of warm deformation and DET. This suggests that the deformation at the initial temperature is more favorable to the spheroidization than that at the intermediate temperature of DET. In addition, it is also proved that the warm rolling forming in the actual production line can shorten the spheroidization time from at least 10 h to 2 h and obtain ultrafine spheroidized carbides with the roundness close to 1. This research may provide a novel pathway to obtain good spheroidization effect with less energy conservation.

1. Introduction

GCr15 steel is a kind of traditional high carbon chromium bearing steel, which is widely used in railway components, die making, shipbuilding, aerospace and automotive applications. Due to the poor forming performance of GCr15 steel with the lamellar pearlite, it is difficult to produce complex components by cold forming, which limits the use of high carbon steel. For this reason, spheroidization annealing treatment (SAT) is used to obtain the spheroidizing microstructure in which not only helps to restore the ductility, but also helps to maintain the ferrite grain size and reduce deformation resistance. However, the traditional spheroidizing annealing process often needs more than 10 h at the expense of high energy consumption and oxidation of microstructure. Therefore, it is necessary to develop new spheroidization technology.

Recently, the combination of warm deformation with the divorced eutectoid transformation (DET) has been considered as a promising process for energy conservation, emission reduction and productivity improvement to achieve rapid forming and spheroidization. DET means that when the hypereutectoid steel is heated to Ac1 ~ Accm, the carbides fail to dissolve completely and exist on the austenite matrix, then the temperature drops below Ac1, these carbides grow as nucleus, completing the spheroidization. Some researchers have investigated that the heat treatment based on the principle of DET can obtain fine and evenly distributed spherical carbides in hypereutectoid steel. Silva and Asadi also mentioned that plastic deformation can make the lamellar pearlite broken into spherical carbides. Previous study has shown that the spheroidization of carbides can be achieved by warm rolling in a short time. When the matrix containing with undissolved carbides was cooled at a slower rate, it takes only 4 to 5 h to spheroidize. However, it is only a
group of warm rolling process to explore the spheroidization effects of warm rolling, hot rolling and isothermal spheroidal annealing, so the process parameters needs to be further optimized, and the spheroidization mechanism of warm rolling needs further analysis. In addition, some scholars [9, 18] also showed that the fine grains can be obtained by matching the plastic deformation and isothermal transformation. Therefore, the combination of DET with the warm deformation has profound research value.

Nevertheless, compared with the eutectoid transformation (ET) that forms lamellar pearlite, DET usually occurs in the two-phase austenite region at higher temperatures [16]. However, the temperature decreases continuously during the warm rolling process. Although the DET is initially generated by the presence of undissolved carbides in the matrix, the DET is converted to ET when the temperature falls outside the temperature range of DET. Therefore, there is a competitive relationship between DET and ET in the whole process of warm deformation, and the temperature at which the deformation takes place becomes crucial. According to the researches [17], no matter warm deformation occurs before or after γ→α transformation, annealing process can be accelerated. When warm deformation occurs before transformation, a very fine structure and grain may be obtained. Once the deformation occurs after transformation, the effect of phase transformation will also continue to the subsequent deformation process, so as to influence the deformation to a certain extent. However, the specific reasons for the spheroidization of carbide in a short period of time have not been carefully analyzed. Therefore, how to reasonably design the sequence of deformation and transformation needs to further explore.

In this work, the phase transformation temperature in the two-phase region was measured by the thermal expansion experiment. The interactional influence of warm deformation and phase transformation are both obtained by warm compression experiment at different deformation temperatures (T = 750 °C, 720 °C, 700 °C) and strain rates (\(\dot{\varepsilon} = 0.05 \text{ s}^{-1}, 0.5 \text{ s}^{-1}, 5 \text{ s}^{-1}\)). The mechanism of spheroidization was analyzed in detail through microstructures, grain sizes, texture, grain boundary angle, dislocation density and deformation resistance. Finally, the process was verified and adjusted by hardness test and quantitative analysis of microstructure on the industrial production line.

### 2. Material and experiments

#### 2.1. Material and thermal expansion test

The high carbon steel used in the present investigation is received in hot-forged material. The chemical composition is shown in Table 1. In order to analyze the temperature range of DET and ET in the process of warm deformation, thermal expansion test is necessary. According to the dilatometry result of heating the experimental material to 1000 °C at a rate of 1.0 °C s\(^{-1}\) on the thermal dilatometer (DIL), the Ac1 and Accm were determined as 775 °C and 860 °C, respectively. Based on the principle of DET, the selected temperature should be between 775 °C and 860 °C. Combined with the previous research, the temperature selected was 800 °C in this work. The specific process is as follows: The initial material was processed into a cylindrical sample with a diameter of 8 mm and a height of 12 mm, the sample was heated to 800 °C at a rate of 1 °C s\(^{-1}\) and held for 10 min to obtain partially austenitized microstructure, then slowly cooled to 650 °C in furnace at a cooling rate of 0.05 °C s\(^{-1}\) by Gleeble-3500 thermal simulator. The diameter change was recorded during the furnace cooling stage to get the temperature range of DET and ET.

| Table 1. Chemical composition of GCr15 bearing steel (wt%). |
|-----------------|---|---|---|---|---|---|---|
| Element | C | Cr | Si | Mn | P | S | Fe |
| Wt% | 1.05 | 1.61 | 0.33 | 0.34 | 0.02 | 0.023 | Bal. |

#### 2.2. Warm compression experiments

According to the result of thermal expansion test, warm compression experiments were designed to investigate the effects of different deformation temperatures and strain rates on microstructure. The equipment was the Gleeble-3500 thermal simulator. According to the empirical formula of rapid spheroidization \(t = 0.75 \text{H (H is the height of sample, t is the holding time), the samples were firstly kept at 800 °C and held for 10 min, then cooled to T (T stands for the temperature of warm deformation, which is 750 °C, 720 °C, 700 °C) at a cooling rate of 1 °C s\(^{-1}\) and kept warm for 3 min, following the samples were compressed by 40% at strain rates of 0.05 s\(^{-1}\), 0.5 s\(^{-1}\) and 5 s\(^{-1}\), respectively. After the deformation were completed, they were cooled down to 650 °C at a cooling rate of 10 °C s\(^{-1}\), and finally air-cooled. The experimental process is shown in the figure 1.
2.3. Microstructure characterization

The microstructures of specimens were characterized by means of scanning electron microscope (Model-ZEISS MERLIN SEM) operating at 10 kV accelerating voltage after etching with 2% nital solution for 5 s. The diameter and volume fraction of spherical carbides were measured using Image-pro Plus software. Optical microscopy (Carl Zeiss) was employed to obtain the micrographs for measuring the grain size after quenching and tempering. The evolution of texture was measured by means of electron backscatter diffraction (EBSD), the samples were prepared by mechanical grinding and polishing using a colloidal suspension of 40 nm silica. The data from EBSD testing were analyzed using TSL OIM software to evaluate texture, grain size, grain boundary character distribution (GBCD) and the local misorientation, of which the High angle grain boundaries (HAGBs) are defined as those with misorientation $\theta > 15^\circ$ whereas low angle grain boundaries (LAGBs) with misorientation $2^\circ < \theta < 15^\circ$. In addition, the microhardness was tested using a microhardness tester (HUAYIN, HV-1000A) with a load of 0.5 kg and a dwell time of 10 s.

2.4. X-ray diffraction analysis

The specimens for x-ray diffraction (XRD) analysis were polished mechanically and electrolytically, and then the x-ray diffraction experiments were performed with a scanning speed of 1° min$^{-1}$ on a Rigaku D/MAX-RB diffraction analyser with Cu-K$\alpha$ radiation at 12 kW. The dislocation density was calculated using the WH method by the following equations (1) and (2) [18]:

$$\frac{\delta \cos \theta}{\lambda} = \frac{\alpha}{D} + 2\varepsilon \sin \theta$$  
$$\rho = 14.4 \left( \frac{\varepsilon}{b} \right)^2$$

where $\delta$ is the the full width at half maximum (FWHM) of the diffraction peak; $\lambda$ is the wavelength of x-ray, Cu radiation is 0.15405 nm; $\theta$ is the diffraction angle; $\varepsilon$ is the microscopic distortion; $\alpha = 0.9$; $b$ is the Burgers vector, equal to 0.248 nm; $D$ is the grain size of the ferrite.

3. Results and discussion

3.1. Thermal expansion test

Researchers believe [19] that the pearlite transformation during the heating process involves the dissolution of cementite and austenite transformation. Similarly, It also includes the precipitation of carbide and pearlite transformation during the cooling process. The pearlite transformation was expressed according to the Avrami equations (1) and (2) [20]:

$$x_p = 1 - \exp (-Kt^n)$$  
$$\Delta T = vt$$

where $x_p$ is the proportion of pearlite transformation, $K$ represents the speed and $n$ represents type of the transformation, corresponding to a nucleation growth mechanism. $V$ is the cooling rate, constant.

According to the obtained thermal expansion curve from 800 °C to 650 °C shown in figure 2(a), it can be seen that the expansion of sample radius mainly consists of three stages during the cooling process by lever method [21]. The first stage is the undissolved carbide is slowly precipitated, the second and the third is the
pearlite transformation. By making \( \ln t \) and \( \ln(-\ln(1-X_p)) \) line, \( K \) and \( n \) could be obtained. After fitting, the slope is found to be two values in two time periods shown in figures 2(b), (c). Two-stage slope is caused by the so-called position saturation, the nucleation site is not saturated in the prior stage, and the nuclear embryo is limited to the grain boundary, which is represented as \( k_1 \). In the latter stage, the nucleation position has become saturated, which is equivalent to zero. At this time, only the transformation product is considered to extend into the crystal, corresponding to \( k_2 \). Li [22] found that the curve is also two straight lines after the deformation at austenite stage. He believes that this is caused by the non-uniform nucleation. DET is also a non-uniform nucleation method. So when the temperature is high, the carbide grows on the undissolved core which is the DET. When the temperature is low, the carbide carbide precipitates from the grain boundary and grows into the crystal which is ET.

As shown in figures 2(b), (c), the black line is pearlite transformation, red line represents the DET transformation and the blue line represents the ET. The intersection of the red and blue line is the inflection point of the two transformation modes. By calculation and turning the argument time into temperature, the ratio of DET at the stage of pearlite transformation is 87.6%, the starting and ending temperature of the divorced eutectoid transformation (DET) is 731 °C and 701 °C, respectively. And the ending temperature of the eutectoid transformation (ET) is 682 °C.

In order to observe the microstructures of DET and ET, one sample was heated to 800 °C for 10 min and then cooled to room temperature, which had undergone the ET. The SEM micrographs is shown in figure 3(a). It can be found that the microstructure is composed of lamellar pearlite and spherical pearlite after the furnace cooling. This indicates that under this initial condition, the DET accounts for a larger proportion in the pearlite transformation which is consistent with the calculation. Another was heated to 800 °C for 10 min and cooled to 700 °C for 20 min and then cooled to room temperature, which the phase transformation is completed in DET range. The SEM micrographs is shown in figure 3(b). As can be seen from figure 3(b), there is essentially no product of ET in the material. It is indicated that under such conditions, the ET does not occur in the pearlite transformation. Due to the competition of the DET and ET in the cooling process, how to control the warm deformation is meaningful for spheroidization.

**Figure 2.** (a) Thermal expansion curve and fitting results; (b) Kinetic parameters; (c) Conversion ratio of pearlite transformation.

**Figure 3.** SEM micrographs of the samples subjected to (a) ET and (b) DET.
3.2. Influence of deformation temperature on the microstructure and texture

Figures 4(a)–(d) show the microstructures of initial material and the specimens after deformed at 750 °C, 720 °C and 700 °C. When the samples are deformed at 750 °C, there are a large amount of lumpy and large-grained carbides in the microstructure. When the samples are deformed at 700 °C, it still contains a large amount of carbides stacked together to form large particle carbides with average diameter of more than 2 μm, although the finer carbides have been obtained. However, when the samples are deformed at 720 °C, the carbides have been completely spheroidized. And the distribution of carbides is more uniform. According to the image-pro software, the roundness of warm deformation of 750 °C, 720 °C, and 700 °C is 1.42, 1.34 and 1.16 and the average diameter is 0.38 μm, 0.34 μm and 0.23 μm. It indicates that when the temperature deformation is carried out at a higher temperature, carbides are larger but less circular in line with the standard of traditional spheroidizing annealing. Deformed at a lower temperature, the carbide are more round, but clustered together and the distribution is uneven. According to the thermal expansion results, 720 °C is within the DET, when the warm deformation in the two-phase region, it contributes to more spheroidization than deformation in the low-temperature austenite region [23]. It suggests that the dissolved carbide is more likely to precipitate from the undissolved core.

In order to observe the dynamic recrystallization of the warm deformation, the samples deformed at 720 °C and 700 °C were reheated to 860 °C for 15 min and subsequently quenched into oil (60 °C). After that, the as-quenched samples were finally tempered at 160 °C for 2 h in an air furnace. As can be seen from figures 5(a), (b), the average quenched grain size are 11.74 μm and 12.76 μm, respectively. Heavy warm deformation at 700 °C and 720 °C both contribute to dynamic recrystallization (DRX).

Studies [24, 25] have shown that the migration and growth of grain boundaries affect microstructure, and the essence of DET is a process of γ-α phase boundary migration. When the γ-α phase boundary passes through the undissolved nucleation core, the undissolved carbide begins to grow and form spheroidal carbides. In addition, grain size definitely affect DET. When it deforms at 720 °C, dynamic recrystallization occurs before the phase transformation. the grain refinement leads to an increase in grain boundary area, and carbides are more likely to precipitate. Since dynamic recrystallization occurs earlier, the grains are also very uniform during subsequent quenching and tempering shown in figures 5(a), (b). Uniform microstructure and grain size can be obtained deformed at 720 °C with good mechanical properties when used. The precipitated carbides are easy to dissolve again when deformed at 700 °C. Meanwhile, the finer the grain, the higher the hardness, the worse the formability. Liu et al [26] believe that 850 °C to 650 °C deformation is the most conducive to spheroidization in GCr15 steel, Chao [27] limits this temperature to 750 °C to 700 °C. However, based on the observed microstructure and grain uniformity, it can be assumed that the appropriate deformation temperature is located at the temperature range of 700 °C ~ 720 °C.

Figure 4. SEM micrographs of (a) initial material and warm deformation at: (b) 750 °C; (c) 720 °C; (d) 700 °C.
Furthermore, according to the equations (1), (2) and XRD analysis shown in figure 6, the dislocation density and the volume fraction of carbides in ferrites for the specimens deformed at different temperature can be determined. Normally, the dislocation density increases with the decrease of deformation temperature. It can be seen that the dislocation density of the samples deformed at 700 °C and 720 °C are determined to be $1.534 \times 10^{14}$ m$^{-2}$ and $1.065 \times 10^{13}$ m$^{-2}$, respectively, which means that the dislocation density of the samples deformed before DET is much lower than that deformed after DET. This may be due to dislocation annihilation caused by partial deformations due to DET when deformed at 720 °C, which DET cannot offset by subsequent dislocations when deformed at 700 °C. In addition, it is also seen in figure 6 that the peak deformed at 700 °C is slightly offset to the left relative to at 720 °C, indicating that the carbon content in the ferrite is higher deformed at 700 °C, that is, the content of carbides is relatively low, which is consistent with that shown in the SEM diagram.

To further investigate the effect of deformation temperature, texture, LABs and the misorientation angle of sub-grain boundary are studied by EBSD. Figures 7(a)–(d) shows the inverse pole figure(IPF) maps and pole figure maps deformed at 720 °C and 700 °C, respectively. It can be seen in figures 7(a), (d) that the random grain orientation can be observed deformed at 720 °C, while many same elongated-grains deformed at 700 °C, and the grain sizes of the former is finer and more evenly distributed, which are 4.41 µm and 5.12 µm, respectively. In addition, figures 7(b), (e) and (c), (f) also shows that the texture at 700 °C is more at reference directions $\langle 001 \rangle$ and $\langle 111 \rangle$. This may be the dislocation slip caused by deformation leads to DRX, and the recrystallized crystal nuclei grow up to form the recrystallized texture, eliminating deformation texture during the subsequent furnace cooling deformed at 720 °C. However, the ferrite recovery continuously deformed at 700 °C eliminates the dislocation pile-up to make DRX incomplete and parts of deformation texture remain.

In addition, the phase and crystal Angle distribution, grain size and misorientation angle distribution deformed at 720 °C and 700 °C were also analyzed in figure 8. The results show that is a certain increstion in the fraction of small size grain deformed at 720 °C, while the fraction of low angle grain boundary decreases. It also
Figure 7. IPF maps and pole figure maps deformed at: (a)–(c) 720°C; (d)–(f) 700°C.

Figure 8. Phase and crystal Angle distribution: (a) 720°C; (b) 700°C and grain size and misorientation angle distribution: (c) Grain size; (d) misorientation angle.
indicates the previous analysis that DRX has grown completely at 720 °C, resulting in a larger orientation angle at 700 °C. EBSD experimental results also reveal that local recrystallization occurred and the newly generated recrystallized texture with a certain orientation cancelled out the cold deformed texture, causing the maximum texture strength deformed at 700 °C. The specific quantity statistics at different deformation temperatures are shown in table 2.

As is shown that the proportion of LABs are 3.2% and 36.9% and the content of carbide is 3.1% and 4.0%, respectively, which indicates that dynamic recrystallization hardly occurs at 700 °C, but has been basically completed at 720 °C. Besides, the KAM deformed at 700 °C is larger than at 720 °C. In general, KAM is applied to describe the situation of strain energy and local dislocation, and the higher KAM values in the grains correspond to higher local misorientation. In this work, the KAM of the samples deformed at 700 °C is larger than that deformed at 720 °C., which indicates that the movement and migration of the second phase also affect and eventually weaken texture, deformation induces the precipitation of massive second phase and spreads early to the locations of dislocation pile-ups, such as subgrains and grain boundaries.

### 3.3. Influence of deformation temperature on deformation resistance

In order to know how DET affects warm deformation, different strain rates are designed. The deformation temperature is still 720 °C and 700 °C respectively, with 0.05 s⁻¹, 0.5 s⁻¹ and 5 s⁻¹. 0.5 s⁻¹ is closest to the strain rate of the industrial rolling ring. The stress-strain curve is shown at figure 9.

As shown in the figure 9, when the strain rate is 5 s⁻¹, the deformation resistance is high. And the deformation resistance at 700 °C is larger than 720 °C, which is in line with the high temperature stress-strain relationship [28]. However, when the strain rate is 0.5 s⁻¹, the deformation resistance at 700 °C is much lower than the deformation resistance at 720 °C. This is due to the DET during the cooling process from 720 °C to 700 °C. Within this temperature range, not only the carbides precipitate from the matrix, but also the undissolved carbides could act as the nucleation core to further grow, which will lead to the soft of material. When the strain rate is 0.05 s⁻¹, the deformation process is so long at 720 °C. The phase transformation process has also been finished. So the deformation resistance at 720 °C and 700 °C is almost uniform and very low. The results show that the phase transformation is helpful to the large deformation in a short time. During warm rolling in industry, the temperature of the forging is reduced very quickly, which makes it difficult to finish the deformation in pearlite transformation. It is very meaningful to use the DET to reduce the material deformation resistance. In order for this process to be applied quickly on current hot rolling lines, the DET needs to be carried out before the warm deformation.

| Temp(°C) | Grain size(μm) | Carbides(%) | KAM(*) | LAGBs | HAGBs |
|----------|----------------|-------------|--------|-------|-------|
| 720      | 0.34           | 4.0         | 0.41   | 0.032 | 0.968 |
| 700      | 0.23           | 3.1         | 0.28   | 0.369 | 0.631 |

Figure 9. Stress-strain curves at different temperatures and strain rates: (a) deformation at 720 °C (b) deformation at 700 °C.
4. Experimental verification of warm rolling in the industry

According to the results of the above studies, the better spheroidized microstructure can be obtained when the deformation occurs before the DET. Therefore, in order to verify this conclusion, two kinds of practical warm rolling processes are designed on a hot ring rolling production line of TMB (Chengdu Tianma Bearing Co, Ltd), one is to conduct the deformation before DET (figure 10(a)), the other one is to conduct the deformation after DET (figure 10(b)). The material are pearlite-structured GCr15 bearing steels, which were processed into ring blanks with the inner diameter of 110 mm and thickness of 35 mm. After two types of warm rolling process, rolling ring were processed into the ring blanks with a inner diameter and thickness of 180 mm and 20 mm. In order to evaluate the spheroidization effect of these two processes, a group of traditional spheroidization annealing process was also carried out. Figures 11(a)–(f) shows the SEM images and the carbide size distribution of two warm rolling experiments and traditional spheroidization annealing (SAT).

As is shown in figure 11, compared with the DET after deformation (figure 11(b)) and traditional SAT (figure 11(b)), the carbides are higher roundness, smaller size and more uniform distribution (figure 11(a)). Besides, the roundness of carbides by warm rolling before the DET is approximately 1, which greatly exceeds the effect of the conventional spheroidizing annealing. The carbide particles are also greatly refined. Most importantly, the total spheroidizing annealing time has been reduced from more than 10 h to about 2 h and the optimized warm ring rolling can be achieved on a hot rolling ring production line by simply adding a furnace. In order to compare the three processes more intuitively, the corresponding quantitative statistics were performed with Image-pro software. The statistical results are shown in table 3.

As can be seen from table 3, both large particle carbides and long carbides are greatly reduced. The hardness is slightly increased, but it is also basically in line with industrial production requirements. The quenching and tempering grains and carbides both are uniform. Although both the deformation before and after the phase transformation contribute to the spheroidization greatly, but it is the best when the deformation before DET among the three schemes in roundness, long, large carbides and uniformity.

5. Conclusions

This work focuses on a rapid spheroidization method for obtaining ultrafine carbides combined warm deformation with DET on GCr15 bearing steel, the conclusions can be drawn as following:

(1) The analysis on thermal expansion shows that the temperature range of the divorced eutectoid transformation (DET) and the eutectoid transformation (ET) are approximately 730 °C~700 °C and 800 °C~650 °C, respectively.
When the warm deformation is carried out at 750 °C, 720 °C and 700 °C, the roundness of carbide are 1.42, 1.34, 1.16, and the average diameter of carbide are 0.38 μm, 0.34 μm, 0.23 μm, respectively. Meanwhile, when the warm deformation at 720 °C, the deformation resistance is lower, while the final excellent spheroidization microstructure can be obtained.

In the actual production line for warm rolling forming, the whole processing time is shortened from at least 10 h to 2 h. This work provides a potential application prospect for high carbon steel to realize rapid spheroidization by the combination of the warm rolling and DET.

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