Decomposition characteristic of austenite retained in GCr15 bearing steel modified by addition of 1.3 wt.% silicon during tempering

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ABSTRACT

The decomposition characteristic of austenite retained in a GCr15 bearing steel modified by the addition of 1.3 wt.% silicon during tempering was investigated by microstructural observation, X-ray determination, and dilatometric experiment. The addition of 1.3 wt.% silicon in the modified GCr15 bearing steel significantly increases the amount of remaining austenite. After tempering at 300 °C for 96 h, 18 vol.% of austenite with 1.6 wt.% carbon remained. Austenite decomposition during the tempering is a bainitic transformation, and occurs via the displacive mechanism, following by carbon partitioning into the remaining austenite. The bainite transformation becomes slower as the carbon enrichment in austenite improves. In contrast, carbide precipitation accelerates the bainite transformation kinetics. However, the carbon enrichment in austenite associated with carbon partitioning and the precipitation of carbides are competitive processes, with their relative rates depending on temperature. Consequently, the improvement in the thermal stability of austenite is ascribed to the combined effects of the partitioning of carbon into austenite and the suppression of carbide precipitation.

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1. Introduction

Early experiments on M2 high-speed steels concluded that the presence of retained austenite (RA) is detrimental to the fatigue performance of ball bearings [1], because the volume expansion accompanying this transformation introduces localized stresses [2,3]. An opposite effect is reported for 52100 (GCr15) bearing steel, where the RA enhances the contact fatigue performance of steel balls when a bearing operates in contaminated lubrication [4,5]. Indeed, it has been demonstrated that the RA leads to cyclic hardening through transformation and enhances the development of a mean compressive stress [6,7]. The effect is said to scale with the amount of austenite, especially when the steel in question is intrinsically brittle.
RA is an important metastable phase in common bearing steels based on a predominantly martensitic microstructure. This instability of RA will significantly affect both the mechanical properties and dimensional stability of bearing steels. Unfortunately, high mechanical properties and good dimensional stability are achieved by the opposing variations of the tempering conditions. Therefore, depending on each application, a compromise must be reached. In general, tempering leads to a dramatic reduction in the amount of RA, where it is observed that cementite can precipitate and austenite decomposes into ferrite and cementite. The rate of this transformation is controlled by the carbon diffusion in austenite [8]. However, Jimenez-Melero et al. [9] concluded that the decomposition kinetics of RA is controlled by the long-range diffusion of carbon atoms into the austenite ahead of the moving austenite/ferrite interface.

So far, the strengthening mechanism of rolling contact fatigue (RCF) performance by a certain amount of stabilized RA has been summarized as due to three effects [10,11]: (i) The blocking crack propagation (BCP) effect. The RA blocks crack propagation, i.e., the cracks either grow continuously in the austenite or trace a zigzag path through the interlocking martensite laths, requiring an increased energy for fracture. (ii) The stress-induced transformation (SITR) effect, which can relieve the stress within untransformed RA and its adjacent martensite. (iii) The dislocation absorption of RA (DARA) effect. The RA can continuously absorb the dislocations from martensite, resulting in the stress concentration in martensite decreased. This will require an increased energy for crack initiation and propagation in martensite. Accordingly, the results conclude that the more RA gives rise to stronger BCP, SITR and DARA effects as well as higher RCF performance.

The stability of RA is dependent on chemical composition, especially carbon content [12,13], morphology [14–16], grain size of the austenite [17,18], dislocation density in the austenite grains and surrounding microstructure [14,19]. However, the carbon content can have opposite effects on the thermal stability of RA during heating and cooling. On the one hand, carbon enrichment of austenite is desired to stabilize this phase upon martensitic transformation during quenching and partitioning [20,21]. On the other hand, the driving force for carbide precipitation increases with the increasing carbon content of austenite, which can result in a higher instability of RA during tempering [22]. Hence, the heat treatment and chemical composition of bearing steels is engineered in terms of RA in order to use the advantageous effect of the stabilized RA.

However, if tempering of martensitic steels and associated RA amount changes has been studied in detail [8,23], not much has been published concerning the high-silicon and high-carbon bearing steels. Nevertheless, it has been demonstrated that high-silicon and high-carbon bearing steels can achieve a good high RCF performance and a higher wear life [4,5,24,25], which is related to a certain amount of stabilized RA. Therefore, it is very important to study the characteristic of RA decomposition during tempering in high-silicon and high-carbon bearing steels. The present work aims to systematically elucidate the decomposition characteristics of RA in a GCr15 bearing steel modified by the addition of 1.3 wt.% silicon during tempering.

| Table 1 – Chemical composition (in wt.%) of the GCr15 and modified GCr15 bearing steels. |
|---------------------------------|--------|--------|--------|--------|--------|--------|--------|
|                                | C      | Si     | Mn     | Cr     | Ni     | Mo     | Fe     |
| Modified GCr15                 | 1.0    | 1.30   | 0.45   | 1.50   | 0.20   | 0.28   | Bal.   |
| GCr15                          | 0.99   | 0.08   | 0.26   | 1.5    | 0.1    | 0.05   | Bal.   |

2. Materials and methods

The modified GCr15 bearing steel with the chemical composition listed in Table 1 was received as a forged ingot and then hot rolled into 60mm diameter bar. The as-received steel exhibits a ferrite matrix with spherical cementite particles. Samples (13 × 13 × 55 mm3) were austenitized at 900 °C for 30 min and then quenched to 50 °C in oil for 10 min followed by air cooling to room temperature.

The studied tempering temperature varied from 200 °C to 500 °C, which is higher than the starting temperature of RA decomposition during continuous tempering, with the tempering time ranging from 2 to 96 h. Dilatation was probed using a high-resolution Bähr DIL 805A/T dilatometer to record the length change in the process of tempering. Specimens with 4 mm diameter and 10 mm length were used for the dilatation measurement.

Microstructural observations were carried out by scanning electron microscopy (SEM). Specimens for SEM were prepared by mechanical polishing followed by etching in a 4 vol.% Nitral solution. SEM microstructural observation was carried out using a TSCAN LYRA3 microscope operating at 10 kV. The specimens for electron backscatter diffraction (EBSD) examination were prepared by mechanical grinding and then polished with a colloidal silica slurry. EBSD measurements were performed using Aztec software on a TSCAN LYRA3 microscope under the following conditions: acceleration voltage of 20 kV, working distance of 5 mm, tilt angle of 70 deg and step size of 0.1 μm. EBSD data were post-processed using the commercial CHANNEL 5 software provided by HKL Technology.

For transmission electron microscopy (TEM), thin disks with the diameter of 3 mm were mechanically thinned to 50 μm using 4000 grit SiC paper and then electropolished with a twin-jet electropolisher at −25 °C in a solution of 5% perchloric acid and 95% ethanol (by volume) at 50 kV until perforation occurred. A JEOL-2100F microscope operating at 200 kV was used to determine the types of phases and examine the microstructures at higher magnification.

The RA volume fraction as well as the RA carbon concentration was determined by X-ray diffraction (XRD) analysis using an Ultima IV (185 mm) X-ray diffractometer, in the 2θ range from 35° to 100°, with unfiltered Cu K-alpha radiation. The machine was operated at 40 kV and 30 mA. All specimens were metallographically polished and then etched in hydrochloric acid for 30 s in order to minimize the possible errors originating from the polishing process. MDI Jade6 software and the Rietveld method were used to quantify the RA volume fraction and lattice parameter (a, c) [8,26,27]. In the present study, the relationship between the austenite lattice parameter...
parameter and its chemical composition was used as given by [9,28–30]

\[ a_r = 3.556 + 0.045x_C + 0.00095x_{Mn} + 0.006x_{Cr} - 0.00057x_{Ni} \]

where \( a_r \) is in angstrom and \( x_{C}, x_{Mn}, x_{Cr} \) and \( x_{Ni} \) are in wt.%. The presence of silicon does not significantly influence the austenite lattice parameter within the experimental accuracy [26].

3. Results

3.1. Microstructural characteristics

Fig. 1 shows a typical SEM microstructure prior to the tempering of quenched specimen. It is clearly revealed that the quenched microstructure consists of martensite (M), retained austenite (RA) with block morphology, and a few spherical undissolved carbide particles. These spherical carbides are remained after austenitizing at 900 °C for 30 min. Retention of such spherical carbide particles provides resistance to abrasive wear. RA amount and its carbon content in the quenched specimen were determined to be 40 ± 2 vol.% and 0.82 ± 0.05 wt.%, respectively.

SEM microstructures of the tempered specimens are illustrated in Fig. 2. After tempering at 250 °C for 12 h and 350 °C for 5 h, as shown in Fig. 2(a, b), plate-shaped ferrite (PSF) is clearly observed by the SEM microstructures. However, long-rod carbides and block-type ferrite are observed in the specimen tempered at 400 °C for 5 h, as shown in Fig. 2(c). When the tempering temperature is increased to 450 °C, a large number of carbides are precipitated, as shown in Fig. 2(d).

Furthermore, the martensite in the quenched microstructure was tempered, seen from the precipitation of the fine structured carbides inside it. To investigate the carbides precipitated from the martensitic matrix at low temperature, TEM has been performed after tempering at 200 °C for 2 h, as shown in Fig. 3(a, b). Analysis of the SAD pattern results in the posi-
Fig. 3 – TEM microstructures highlighting ε-carbides and θ-carbides precipitated from martensitic matrix during tempering at 200 °C for 2 h (a, b) and 450 °C for 5 h (c, d), respectively, where (a) and (c) is the dark-field images, (b) and (c) is the corresponding SAD patterns (taken from the [-21-1]ε and [-2-1-1]θ reflection indicated by the arrow, respectively).

The changes in the RA amount and its carbon content in the specimens tempered at 300 °C as a function of tempering time, as determined from XRD analysis (such as in Fig. 4), are shown in Fig. 5. After tempering at 300 °C for 96 h, 18 vol.% RA remained and its carbon content was enriched to 1.6 wt.%. This indicates that the austenite decomposition during the tempering is accompanied by the carbon enrichment of the remaining austenite. Thus, the formation of carbon-enriched austenite is a manifestation of the incomplete austenite decomposition during the tempering, since the transformation stops before the phases achieve their equilibrium compositions [31,32]. However, it has been found that the RA in 52100 steel can be completely decomposed during tempering at 230 °C for 2 h [33]. This implies that the added silicon in modified GCr15 bearing steel not only decelerates the kinetics of RA decomposition but also increases the RA...
Photograph 5 – Changes in RA amount and its carbon content in the specimens tempered at 300 °C as a function of tempering time.

amount after the stasis of the RA decomposition. Below, these results are interpreted and the thermal stability of RA during tempering is discussed.

Fig. 6 shows the RA amount and its carbon content in the specimens tempered for 5 h at different temperatures. The RA volume fraction decreases with increasing tempering temperature, as shown in Fig. 6(a). This indicates that the RA is more inclined to decompose at temperatures higher than 350 °C, where the RA is determined to be completely decomposed during the tempering at 500 °C for 5 h. However, the carbon content in the RA exhibits a peak at 300 °C, as shown in Fig. 6(b). It clearly reveals that the RA carbon content in the tempered specimens is higher than that in the quenched specimen (0.82 wt.%).

3.2. Dilatation characteristics

Metallographic observations have already indicated that the microstructural evolution during tempering is due to both the tempering of martensite and the decomposition of RA. The former should lead to a contraction as the carbides precipitated from carbon supersaturated martensite, whereas the latter should be accompanied by an expansion. As a result, the temperature of the phase transformation occurring during the tempering can be determined by the dilatometric experiment.

Fig. 7 shows the dilatation curve in length during continuous tempering at the rate of 0.02 °C/s. It is clearly revealed that the cementite precipitation temperature in the modified GCr15 bearing steel is higher than 380 °C during continuous tempering, which is significantly higher than that in the GCr15 bearing steel (approximately 230 °C) [34,35]. This implies that the addition of 1.3 wt.% silicon can significantly suppress the precipitation of cementite. Fig. 8 shows the dilatation curves obtained during tempering at different temperatures. During tempering at 250 °C and 300 °C, the dilatation in length is gradually increased, implying that the decomposition of RA occurred. However, the dilatation in length during tempering at 350 °C first increased and then decreased, corresponding to the decomposition of RA and the precipitation of

Fig. 6 – RA amount (a) and its carbon content (b) in the specimens tempered for 5 h at different temperatures.

Fig. 7 – Dilatation curve in length during continuous tempering at the rate of 0.02 °C/s, where the change in length caused by thermal expansion coefficient of phase is ignored.
cementite, respectively. It demonstrates that the dilatation characteristics reflect the phase transformation kinetics. At low temperature, RA decomposition and transition carbides precipitation are the main phase transformations. However, the kinetics of cementite precipitation is significantly accelerated during tempering at 400 °C. Thus, it is suggested that the kinetics of phase transformation during tempering, such as carbide precipitation and RA decomposition, depends strongly on the tempering temperature.

4. Discussion

Austenite is susceptible of undergoing bainite transformation during tempering. Hence, the plate-shaped ferrite observed in Fig. 2, is identified as a product formed from the bainite transformation, referring to it as bainitic ferrite. The crystallographic orientation relationship (OR) between bainitic ferrite and its parent austenite was studied by EBSD analysis. Fig. 9 shows the OR map superimposed on the image quality map and phase color map of the modified GCr15 bearing steel subjected to bainite transformation at 300 °C for 2 h. The tolerance angle for the OR in this analysis was set as 3 deg. In this case, the OR of most interfaces between the bainitic ferrite and adjacent austenite is more inclined to an N-W OR [36,37]. There is strong evidence that suggest that in the modified GCr15 bearing steel austenite decomposition proceeds through bainitic transformation and occurs via a displacive mechanism.

As the bainitic ferrite formation progresses at 300 °C, Ms temperature determined by the dilatometric experiment from the untransformed austenite is gradually decreased, as shown in Fig. 10. The result implies that the bainitic ferrite formation accompanies the stabilization of untransformed austenite. After the transformation at 300 °C for 2 h, the carbon concentration in RA is enriched to 1.5 wt.%. Thus, it is suggested that carbon enrichment in austenite occurs during bainite transformation, arising from carbon partitioning into the

Fig. 8 – Dilatation curves obtained during tempering at different temperatures, where the change in length caused by thermal expansion coefficient of phase is ignored.

Fig. 10 – Change curve in Ms temperature determined by dilatometric experiment as a function of bainite transformation time at 300 °C.

Fig. 9 – Orientation relationship (OR) map superimposed on image quality map (a) and phase color map (b) of the modified GCr15 bearing steel subjected to bainite transformation at 300 °C for 2 h (red lines in (a) represent as N-W; black line in (b) represents as grain boundary of bcc phase (above 10°]).
surrounding austenite. However, the influence of the preferred transformation of low-carbon austenite on the carbon content of remaining austenite increased cannot be completely excluded.

The absence of carbide in the bainitic ferrite is interpreted in terms of carbon trapped at dislocations as described by the tempering theory of Kalish et al. [38]. They proposed that in the presence of dislocations, carbon atoms first concentrate at dislocations due to the stress/strain-induced ordering. It is energetically favorable for the carbon atoms to remain segregated at the dislocations relative to their precipitation as \(\varepsilon\)-carbides and cementite. Dislocation debris within bainitic ferrite and RA is shown evidently in Fig. 11. The relatively high dislocation density is mainly attributed to the fact that the shape deformation accompanying the displacive transformation is at least partially accommodated by plastic relaxation [39]. Hence, a substantial amount of carbon in the bainitic ferrite can be captured by the dislocation so that an increased amount of carbon is available for the carbon partitioning. This result concludes that the absence of carbides in the bainitic ferrite promotes the formation of carbon-enriched austenite. Furthermore, the impressive retardation of cementite precipitation in austenite, caused by the addition of 1.3 wt.% silicon, also contributes to the formation of carbon-enriched austenite, resulting from the effective carbon available for carbon enrichment in austenite increased. This is explained by the fact that, when the silicon is trapped inside the cementite lattice, the driving force for cementite precipitation from austenite is decreased significantly, making it impossible for the para-equilibrium cementite to precipitate from austenite [40].

Fig. 12 shows the bainite transformation kinetics of GCr15 and modified GCr15 bearing steels. Examination of the figure clearly reveals that the addition of 1.3 wt.% silicon not only slows down the kinetics of bainite transformation but also increases the amount of remaining austenite after the stasis of the transformation. Generally, the carbon enrichment of austenite leads to the precipitation of carbides [22], implying that the effective carbon enrichment of austenite is negligible [41]. However, in the modified GCr15 bearing steel, the carbide precipitation is kinetically suppressed, leading to significant carbon enrichment during the formation of bainitic ferrite [40]. Thus, as it progresses, the bainite transformation slows down because the formation of carbon enrichment in austenite results in the increase of the effective activation energy and thus, the effective driving force for the transformation decreased. In addition, if the carbon concentration in the untransformed austenite approaches the value at which the free energy of ferrite and austenite phase of the same composition become identical, the bainite transformation ceases. This result is consistent with the data shown in Fig. 5. Consequently, the carbon enrichment in austenite heavily influences the eventual fraction of bainite. This result also confirms that the incomplete austenite decomposition is a consequence of the formation of carbon-enriched austenite associated with carbon partitioning.
Fig. 13 – TEM microstructure highlighting the cementite precipitation during bainite transformation at 400 °C for 5 h, where (a) is the dark-field image and (b) is the corresponding SAD pattern (taken from the [0–21]θ reflection indicated by the arrow).

However, the kinetics of bainitic transformation at 400 °C in modified GCr15 bearing steel is inconsistent with that in the GCr15 bearing steel, as shown in Fig. 12(b). After the transformation at 400 °C for 2 h, the RA amount and its carbon content are determined to be 40 vol.% and 1.25 wt.%, respectively. Therefore, it is inferred that the deceleration of bainite transformation at the primary stage, is also ascribed to the carbon enrichment in austenite associated with the carbon partitioning. However, cementite precipitation during the transformation at 400 °C for 5 h is demonstrated by the results obtained by SEM and TEM, as shown in Fig. 2(c) and Fig. 13, respectively. The result implies that carbide precipitation within austenite, following by the formation of carbon-depleted austenite, accelerates the kinetics of bainite transformation at the secondary stage. This is explained by the fact that the effective driving force for the bainitic ferrite formation increases because of the decrease of the effective amount of carbon available for the carbon enrichment in austenite [42]. These results conclude that austenite decomposition at 400 °C occurs in two successive processes. In the primary stage, a portion of austenite transforms into bainite, with simultaneous carbon partitioning into untransformed austenite, resulting in carbon enrichment in austenite. In the second stage, cementite can precipitate, and austenite decomposes into ferrite and cementite. Therefore, it is suggested that the improvement in the thermal stability of austenite is ascribed to the combined effect of the partitioning of carbon into austenite and the suppression of carbide precipitation.

(1) The addition of 1.3 wt.% silicon in modified GCr15 bearing steel significantly increases the amount of remaining austenite. After tempering at 300 °C for 96 h, 18 vol.% of austenite with 1.6 wt.% carbon remained. Austenite decomposition during tempering is a bainitic transformation, and occurs by displacive mechanism, following by carbon partitioning into remaining austenite.

(2) The impressive retardation of carbide precipitation in austenite caused by the addition of 1.3 wt.% silicon assists the formation of carbon-enriched austenite associated with carbon partitioning, due to the increase in the effective amount of carbon available for carbon enrichment in austenite. Thus, the bainite transformation slows down with the greater carbon enrichment in austenite. In contrast, carbide precipitation followed by the formation of carbon-depleted austenite accelerates the bainite transformation kinetics.

(3) However, the carbon enrichment in austenite associated with carbon partitioning and the precipitation of carbides are competitive processes, and their relative rates depend on the temperature. Consequently, the improvement in thermal stability of austenite is ascribed to the combined effects of the partitioning of carbon into austenite and the suppression of carbide precipitation.

Conflicts of interest

The authors declare no conflicts of interest.

Acknowledgments

This work was financially supported by the National Basic Research Program of China (973 Program, Grant No. 2011CB706604) and CNC machine tools and basic manufacturing equipment technology comments II (Grant No. 2012ZX04012011).

5. Conclusions

The decomposition characteristic of austenite retained in the GGr15 bearing steel modified by the addition of 1.3 wt.% silicon during tempering, has been investigated. The main conclusions drawn from the investigation are as follows:
REFERENCES


[37] Suikkanen PP, Cayron C, DeArdo AJ, Karjalainen LP. Crystallographic analysis of isothermally transformed
bainite in 0.2C–2.0Mn–1.5Si–0.6Cr Steel Using EBSD. J Mater Sci Technol 2013;29(4):359–66.


