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Effects of deep cryogenic treatment on microstructural evolution and alloy phases precipitation of a new low carbon martensitic stainless bearing steel during aging

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ABSTRACT

It is evident from controversial statements in the literature that the effects of deep cryogenic treatment on the microstructural evolution of low carbon martensitic steels are not fully understood yet. An investigation has been started to clarify this situation by analyzing the microstructural evolution and alloy precipitation after aging. The results show that the hardness is enhanced after deep cryogenic treatment and this is mainly contributed to the transformation of retained austenite to martensite at low temperature. By comparing with the samples subjected to quenching and deep cryogenic treatment, the hardness behaviors produced different variation tendency and characteristics during aging. According to obtained results and comparing with the characterization of carbides during aging, it showed that deep cryogenic treatment is an advantage for the formation and precipitation of alloy carbides during aging. The results indicated that deep cryogenic treatment increases the diffusion driving force of atoms (especially the carbon atoms) which promotes the formation of fine carbides in the process of aging.

1. Introduction

Bearings are widely used in the manufacturing fields like automotive, railway vehicles, industrial machinery and many kinds of special machinery. General-purpose bearings are usually treated through solution hardening and precipitation strengthening, the carbon concentration of the steels usually would be within the range of 0.8–1.1 wt% and an appropriate amount of alloying elements addition.

However, the conventional hardened bearing steels (such as AISI 52100, M50 and M50NiL etc.) are not suitable for use in advanced aerospace engines, wind turbines, and high-speed railways, etc. Because the above-mentioned bearings used for engine shafts were subjected to tolerate vibratory stresses, bending moments and high-speed rotation, elevated temperatures, static and/or thermal fatigue, wear resistance, dimensional stability, and even hot-oil & corrosion environment. These applications are required for the fatigue life of more than 30,000 h or 100 billion contact cycles [1] and subjected to a high-temperature environment. While the maximum service temperature of AISI 52100 steel can be used continuously is about 160 °C, whereas M50 is designed for service temperature as high as 310 °C. The excellent performance of M50 steel is relevant to the alloys and carbides in it [2]. It is obvious that the alloys and a large number of precipitated carbides provide secondary hardening effect for strengthening and stabilizing the microstructure and performance. The final microstructure of the bearing steels usually contains two types of carbides according to the carbides dimension, the small ones precipitated during tempering and the coarse carbides formed in the solidification process. As is well known, the morphology and distribution of carbides play an important role in the performance and service life of bearings. Authors [3] have pointed out that cluster of carbides is harmful to bearing steel than a single carbide due to the shape and size of carbides as well as the interactions between carbides that enhance the stress concentration in service. One of the effective approaches is to reduce the total content of carbides without compromising other properties [4] during materials design. In order to meet the requirements of high performance bearing in modern aerospace industry, a novel heat-resistant bearing steel has developed to appropriate the increasingly harsh requirements and conditions for reliability bearing applications basing on the approach of reducing the total content of carbides by Yang [5,6].

As for the study of bearing steels, the previous researches focused on mechanical properties and microstructural characterization after conventional heat treatment. It is shown that a number of retained

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austenite presented after quenching and tempering [5] due to the action of alloy elements addition. As is well known, the retained austenite is a soft and metastable phase at low temperature and tends to transform into brittle martensite during service under stress and/or high temperature. Thus the dimension will be changed while the retained austenite transforms into fresh martensite under service according to the theory of martensite phase transformation. The transformation of retained austenite to martensite causes volume expansion resulting in distortion and/or lock the bearings during service. In this case, the unpredicted accident would be happened and a heavy loss is inevitable. Deep cryogenic treatment (DCT) is applied to eliminate retained austenite to stabilize the dimension as a supplementary process to conventional heat treatment for industrial applications. It is usually used for tools and measuring implements in order to make the dimension stabilized. In addition, some good advantages and excellent performances have been shown in bearings after deep cryogenic treatment. Harish et al. [7] pointed out that En 31 bearing steel subjected to deep cryogenic treatment showed higher hardness than the conventional heat treatment. The wear resistance can be decreased by a maximum of 75% depending on the service conditions after cryogenic treatment [8]. Gunes et al. [9] have investigated the effects of deep cryogenic treatment on the wear resistance of AISI 52100 bearing steel and suggested that the optimized holding time is 36 h while the wear rate and friction coefficient were decreased. Moreover, Authors [10,11] have optimized DCT parameters for 100Cr6 bearing steel using Taguchi technique. The results show deep cryogenic treatment can improve the wear resistance of bearing steel with suitable parameters including the cooling rate, soaking temperature, soaking time, and tempering temperature.

Furthermore, researchers also made great efforts to figure out the mechanism of microstructural evolution and performance improvement of tool steels applied to deep cryogenic treatment [12–17]. Das et al. [18–20] established the relationships between microstructural parameters and properties of AISI D2 steel subjected to conventional heat treatment and different types of subzero treatments and optimized the duration of cryogenic processing to maximize wear resistance of AISI D2 steel. The results show the improvement of wear resistance is attributed to the retained austenite transforming into martensite and the increase of secondary carbides in the amount and population density [21]. Meng et al. [13] inferred that the improved wear resistance by cryo-treatment was due to the formation of η-carbide and suggested that the favorable distribution of carbides rather than by transformation of retained austenite to martensite. Most researchers believe that the advantage of deep cryogenic treatment is attributed to complete transformation of retained austenite into martensite [22–24] and the formation of fine carbides in martensite matrix [21,25–27]. Li et al. [28,29] established multi-physical field coupling numerical model to evaluate the cooling behavior by means of finite element method simulation and the predicted results were quite reasonable and show a good agreement with experimental results.

Available results in the literature as mentioned above focus on correlations of microstructural evolution and properties of high carbon alloy steels subjected to cryogenic treatment. However, the effects of deep cryogenic treatment on the microstructure and underlying mechanism of low carbon steel for achieving improved mechanical properties are not well crystallized. The purpose of this investigation focuses on the microstructural evolution and alloy phases precipitation during aging of a low carbon steel after deep cryogenic treatment.

2. Experimental procedure

2.1. Materials and heat treatments

The material used in this investigation is a new heat resistant low carbon bearing steel designed by Yang (Central Iron and Steel Research Institute, Beijing). The steel was produced by vacuum arc remelting (VAR) after vacuum induction melting (VIM) to obtain lower nonmetallic inclusion and suppress chemical segregations. The chemical composition of the tested steel is listed in Table 1.

Table 1
Chemical compositions of the tested steel (wt%).

C	Cr	Co	Mo	Ni	V	S	P	Fe
0.15	14.00	13.00	4.80	2.40	0.50	< 0.0001	< 0.0005	Bal.

The electroslag remelting ingot was hot forged into Φ100 mm bars after homogenization at 1250 °C for 10 h. The samples were prepared from the annealing bars along the longitudinal orientation. Heat treatment processes including austenitization were carried out at 1020–1050 °C for 1 h in a vacuum furnace before quenching into oil. The samples subjected to deep cryogenic treatment was soaked in liquid nitrogen directly for 10 h after oil quenching. All samples were tempered at 480–540 °C for 2 h under argon atmosphere. The aging process was carried out in a vacuum furnace and holding for 100 h at 510 °C.

2.2. Mechanical and microstructural characterization

The Rockwell hardness tester was used for all hardness testes. The major load is 150 kg and the duration time is 30 s respectively for all samples. Each effective hardness value of a sample was the average of seven measuring points. The impact toughness is determined for Charpy V-notched specimens with 10 mm × 10 mm × 55 mm in dimension. The phases in different samples were analyzed by X-ray diffraction instrument (DLMax-2550, Rigaku). The relative proportions of austenite and martensite from the X-ray diffractions after each treatment are calculated using the following equation:

$$V_\gamma = \left(\frac{I_\gamma^{hkl}}{R_\gamma^{hkl}}\right) / \left[\left(\frac{I_\gamma^{hkl}}{R_\gamma^{hkl}} + \frac{I_\alpha^{hkl}}{R_\alpha^{hkl}}\right)\right] \tag{1}$$

Here, V_γ is the volume percent of retained austenite, I_α and I_γ are the integrated intensity of the diffraction peak (*hkl*) in the α-phase and γ-phase respectively. R is proportional to the theoretical integrated intensity, depends upon interplanar spacing (*hkl*), the Bragg angle, crystal structure, and composition of the phase being measured. For numerous peaks, each ratio of measured integrated intensity to R-value can be summed. The microstructural evaluation was carried out by scanning electron microscopy (SEM) in backscattered electron mode (BSE). The more detailed and definitive microstructural characterizations were carried out by transmission electron microscopy (TEM). The elemental composition of the precipitates was analyzed by energy dispersive spectroscopy (EDS) attached to the SEM and/or TEM.

3. Experimental results and discussion

3.1. Mechanical and microstructural characterization after quenching and tempering

The optical microscope metallographic microstructure of the samples austenized at different temperatures is presented in Fig. 1. The microstructure is mainly composed of lath martensite and the original austenite grain size can be seen indistinctly. The grain size increased and the martensite lath beams become coarsening with the increase of austenized temperature. In addition, a little number of delta ferrite appears in microstructure when the austenizing temperature reached up to 1050 °C. The reason is the ferrite-stabilized elements such as Chromium and Molybdenum diffused and enriched at high temperature. It increased the Chromium equivalent (Cr_{eq}) and promoted the delta ferrite forming easily when austenized at a higher temperature [30]. As is known, the delta ferrite is a relatively soft phase in the martensite and it reduces the strength of the steel [31]. The morphology and orientation of δ-ferrite grains seriously affected the short-term