

Article

Effect of Austempering on Mechanical Properties of Nb/V Microalloyed Bainitic Bearing Steel

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Abstract: In this study, a multi-element microalloyed GCr15Si1MoNbV bainitic bearing steel was designed by adding trace amounts of Nb and V. The bainite/martensite/retained austenite/undissolved carbide multiphase bearing steel with a short heat-treated cycle and remarkable comprehensive mechanical properties was prepared via low-temperature austempering, which was compared with a traditional quenched and tempered martensitic microstructure. The results showed that the comprehensive mechanical properties of the low-temperature bainitic microstructure of the Nb/V microalloyed bearing steel were better than those of a traditional tempered martensitic microstructure. In addition, as the isothermal temperature increased, the impact toughness of experimental steel was significantly improved, and the hardness decreased slightly. The tensile strength was improved with the increase in isothermal time. As the isothermal temperature increased, the bainite content of the experimental steel rose, and the stability of the retained austenite enhanced due to more carbon atoms being partitioned into it. The research work in this paper made a preliminary exploration for the application of Nb/V microalloyed bainitic bearing steel in the bearing field and provided some basic data for reference.

Keywords: low-temperature bainite; austempering; bearing steel; Nb/V microalloying; mechanical properties



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1. Introduction

Bearing steel plays a vital role especially in large machinery such as vehicles, high-speed railways, and wind power generation equipment. With the vigorous development of modern industry, the requirements for the performances of bearing steel are rising. There is also a wide range of indicators for evaluating the performance of bearing steel, such as hardness, impact toughness, strength, plasticity, wear resistance, and fatigue [1]. With very complicated working conditions, bearings always need to bear large axial and radial loads at the same time. Therefore, the higher impact toughness and strength could greatly ensure the stable operation of bearings while meeting the requirement of hardness [2].

At present, most of the worldwide bearing steels are developed by adjusting the content of Mn, Si, Cr, Mo, and other elements on the basis of GCr15 (equivalent to 100Cr6 in Germany; 52100 in the US; SUJ2 in Japan; En31, NF100C6, ShKh15, Rul 1, 1C-1.5Cr,

etc., in other countries). The bainite and martensite composite structures can be obtained via different heat treatment processes. The retained austenite in steel can greatly improve the plasticity and toughness. Zhou et al. [3] found that the retained austenite became more stable by adding Cr element and the volume fraction of retained austenite had a nonnegligible influence on the strength and plasticity of bainitic steel. The most common microalloying elements in steels are Nb, Ti, and V. They can be used alone or in combination with each other [4]. Foder et al. [5] measured the grain size and mechanical properties of the boron-type S1100QL steel, and the mechanical properties of adding V and Nb together were found to be better than those of adding Nb separately. The heat treatment process has a notable effect on the comprehensive performance of bearing steel. The heat treatment process used in traditional bearing steel is quenching plus tempering to obtain tempered martensite. The high-carbon martensite has high hardness but low impact toughness. The degree of undercooling is so large that even after tempering, it still has a large residual tensile stress, which greatly increases the brittleness [6]. Tang et al. used a two-step austempering process to conduct a series of studies on microalloyed experimental steel and found that the grains could be refined after the addition of Nb element, and the stability of retained austenite was greatly improved due to the high carbon content in it [7]. The excellent combination of toughness and strength is one of the advantages of bainitic steel. Dikic et al. [8] found that as an effective nucleation matrix, V nitride particles could increase the nucleation rate of bainite in a medium-carbon steel. The amount and morphology of retained austenite are of great importance in the application of rolling bearings [9], especially in improving plasticity and toughness. Zhang's research group developed a process that can prepare nano-bainite on the surface and low-carbon martensite with good plasticity and toughness in the core of carburized bearing steel [10]. This represents a significant improvement over martensitic bearing steel, which not only improves the toughness of the core but also improves the wear and fatigue resistance of the surface layer. However, the preparation period of nano-bainite is very long, resulting in a high production cost. In short, the mechanical properties and microstructures of Nb/V microalloyed bainitic bearing steels are still to be studied. It is essential to systematically study the relationship between heat treatment and mechanical properties of Nb/V microalloyed bainitic bearing steels.

This paper studies the addition of Nb and V to microalloyed traditional bearing steels and explores the effects of different isothermal temperatures and times on the mechanical properties of the Nb/V microalloyed bainitic bearing steels. The bainite/martensite/retained austenite/undissolved carbide multiphase bearing steel with a short heat-treated cycle and outstanding comprehensive mechanical property was optimized and prepared.

2. Materials and Methods

2.1. Material and Heat Treatment Process

Alloying elements Nb and V have the effect of increasing the recrystallization temperature, expanding the scope of the austenite recrystallization zone, and inhibiting the recrystallization of austenite. Therefore, adding an appropriate amount of Nb and V in steel can achieve the purpose of grain refinement and precipitation strengthening. Based on the bearing steel GCr15Si1Mo previously studied by our research group, this paper designed an Nb/V microalloyed bearing steel by adding trace amounts of Nb and V (see Table 1).

Table 1. Element content of the designed steel (wt.%).

Element	C	Si	Mn	Cr	Mo	Nb	V	Fe
Content	0.985	1.424	0.338	1.433	0.376	0.086	0.146	balance

The steel was smelted in a vacuum induction furnace. After being removed from the furnace, it was forged and subjected to a four-cycle spheroidizing annealing. The

annealed experimental steel was heat-treated according to the process shown in Figure 1. The experimental steel was austempered at 210 °C, 230 °C, 250 °C, 270 °C, and 300 °C for 6 h, 12 h, and 24 h. The comparison process was directly quenching in oil to room temperature. Finally, the experimental steel was tempered in a box-type resistance furnace at 200 °C for 1 h.

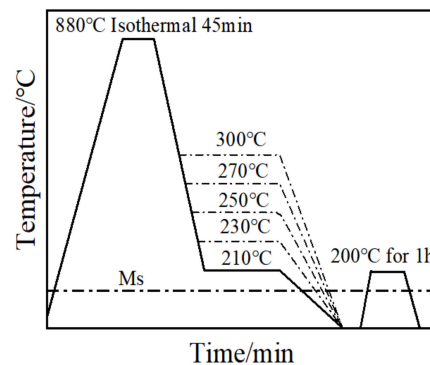


Figure 1. The figure of the heat treatment process.

The martensitic phase transition point (M_s) of the experimental steel was measured by a DIL-805 dilatometer (TA Instrument, New Castle, DE, USA). The result showed that the volume of the experimental steel expanded significantly when it was cooled to about 170 °C. The M_s temperature of the experimental steel was determined to be 175 °C by the tangent method. The austempering temperature used in this experiment was slightly higher than the M_s temperature, and the low-temperature bainite was intended to be obtained.

2.2. Mechanical Performance Experiment

The heat-treated experimental steel was prepared into 10 mm × 10 mm × 55 mm unnotched impact specimens and the tensile specimens were specified in the Chinese standard GB/T228-2002. The impact samples were polished smooth with sandpaper, one side was polished to the mirror surface, and the hardness value was measured with the BUEHLER Rockwell hardness tester. The hardness test took a total of seven points, and the measured values were averaged. The tensile test was carried out on a WDW-50GD (GuanTeng Limited, Changchun, China) electronic universal testing machine with a tensile rate of 0.6 mm/min.

2.3. Microstructure Experiment

The polished metallographic samples were first etched in 4% nitric acid alcohol solution and then observed by an ICX41M metallographic microscope (JingXi Instrument, Fuzhou, China). The D8 ADVANCE ECO diffractometer (BRUKER, Billerica, MA, USA) was used to perform X-ray diffraction (XRD) experiments. The sample was scanned using a step-by-step method. The working voltage and working current were, respectively, set to 40 kV and 40 mA. The scanning angle was set from 30° to 105°, 0.02° in each step, and held for 2 s. The content of retained austenite was calculated according to the measured diffraction peaks. The diffraction peaks of the (200), (220), and (311) of austenite phase (γ) and (200), (211), and (220) of ferrite phase (α) were substituted into the following equation (Formula (1)) to acquire the volume fraction of retained austenite (V_γ) [10].

$$V_\gamma = \frac{(1/n) \sum_{j=1}^n I_\gamma^j / R_\gamma^j}{(1/n) \sum_{j=1}^n I_\gamma^j / R_\gamma^j + (1/n) \sum_{j=1}^n I_\alpha^j / R_\alpha^j} (1 - V_C) \quad (1)$$

In Formula (1), n is the number of diffraction peaks, I represents the intensity of the diffraction peak, and V_c is the volume fraction of undissolved carbide. R represents the material constant, and its expression is as follows:

$$R = \frac{1}{V^2} F^2 P \frac{1 + \cos^2 2\theta}{\sin \theta \sin 2\theta} e^{-2M} \quad (2)$$

In Formula (2), V is the subunit volume, F is the structural parameter, P is a heterogeneous factor, e^{-2M} is the temperature parameter, and θ is the diffraction angle.

The SU-5000 scanning electron microscopy (SEM) model from Hitachi (Tokyo, Japan) was used to further observe the microstructure in detail under a working voltage of 25 kV and working current of 15 mA. The sizes of the observed sample were 10 mm × 10 mm × 10 mm. Undissolved carbide on the sample surface could be seen by SEM. The proportion and diameter distribution of carbides were calculated by Image-Pro Plus (IPP) software.

3. Results and Analysis

3.1. Microstructure Analysis

Figure 2 shows the light optical microstructure of the experimental steel austempered at different isothermal temperatures and times. It can be seen that as the isothermal temperature increases, the bright white parts of the light optical microstructure are gradually reduced. Compared with other phases, bainite is easily eroded by corrosive solution [11], so the dark black parts are bainitic areas. The microstructure of bainite is too fine to be observed clearly. The amount of bainite increases with the extension of isothermal time. As the temperature rises, the carbon atoms diffuse sufficiently and the phase transformation resistance is reduced, which is conducive to the formation of bainite. The amount of bainite increases as the austempering temperature increases [12,13]. Gu et al. [14] also found that raising the transformation temperature in TRIP steel can significantly promote the transformation of bainite, thereby increasing the amount of bainite. As the isothermal temperature continues to increase, the crystal grains gradually grow, resulting in a coarse microstructure at high isothermal temperatures.

The diffraction patterns measured by XRD are shown in Figure 3. It can be seen from the diffraction patterns that as the isothermal time increases, the intensity of the γ diffraction peak gradually weakens, and it can be inferred that V_γ is gradually decreasing. Substituting the relevant information of the diffraction patterns into Formulas (1) and (2), V_γ is calculated and shown in Table 2. The experimental data show that the percentage of retained austenite after different heat-treated processes hardly exceeds 19%, and with the extension of isothermal time, V_γ gradually decreases. With increasing isothermal time, the bainitic nucleation point and the transformation amount of bainite increase gradually, leading to V_γ reducing by degrees.

For the same isothermal time, V_γ decreases first and then increases with increasing isothermal temperature. The reason is that with the increase in isothermal temperature, the amount of bainite transformation increases, leading to the gradual decrease in retained austenite content. In addition, the low isothermal temperature results in a difficult diffusion of carbon atoms, which reduces the stability of retained austenite, and partial martensite will be formed in the final cooling process. When the isothermal temperature continues to rise to 270–300 °C, the content of retained austenite increases again. It is well known that there are two kinds of processes for carbon enrichment in austenite. One is that carbon is enriched into austenite during the formation of ferrite. The other is the partition of carbon from bainite to austenite during the isothermal transformation of bainite. When the isothermal temperature continues to rise, the bainite content greatly increases so that more carbon atoms are partitioned into the retained austenite. In addition, the carbon atoms could be fully solidly dissolved into the retained austenite at 270–300 °C, compared with 250 °C. Thus, the carbon content in retained austenite increases, which increases the stability of retained austenite. The calculation results of the carbon content in retained austenite are shown in Table 3.

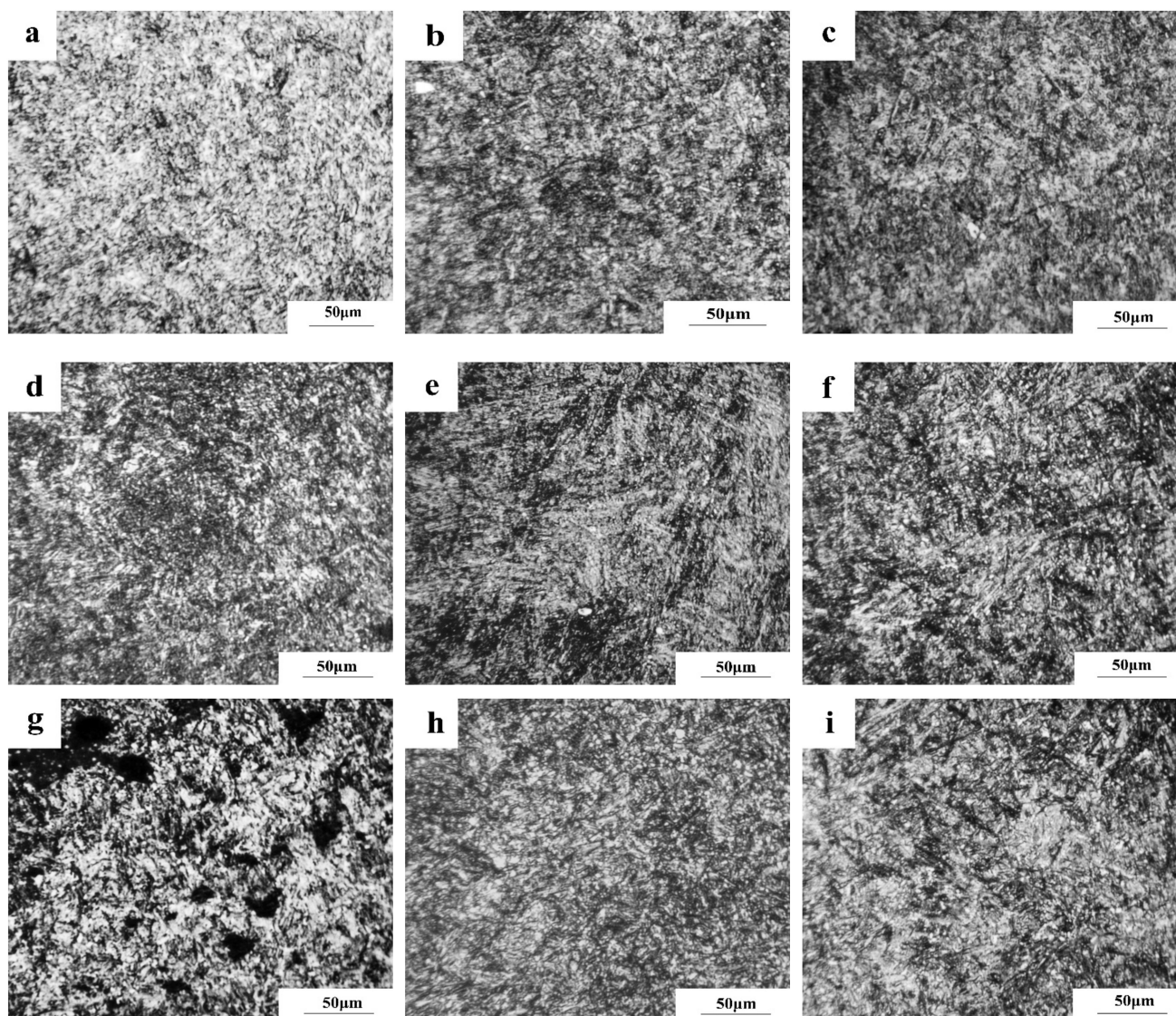


Figure 2. Light optical microstructures under different processes ((a–c) are at 230 °C for 6 h, 12 h, and 24 h; (d–f) are at 250 °C for 6 h, 12 h, and 24 h; (g–i) are at 270 °C for 6 h, 12 h, and 24 h, respectively).

Table 2. The V_{γ} under different processes (vol.%).

Temperature/°C	6 h	12 h	24 h
210	18.74	16.37	9.77
230	11.95	11.15	9.50
250	10.68	10.36	8.35
270	13.82	11.68	10.60
300	16.20	14.23	10.51
Oil quenching		14.70	

Table 3. Carbon content in retained austenite (wt.%).

Temperature/°C	6 h	12 h	24 h
210	0.989	1.294	1.824
230	1.220	1.448	1.835
250	1.555	1.408	1.645
270	1.562	2.000	1.654
300	1.703	1.645	1.725

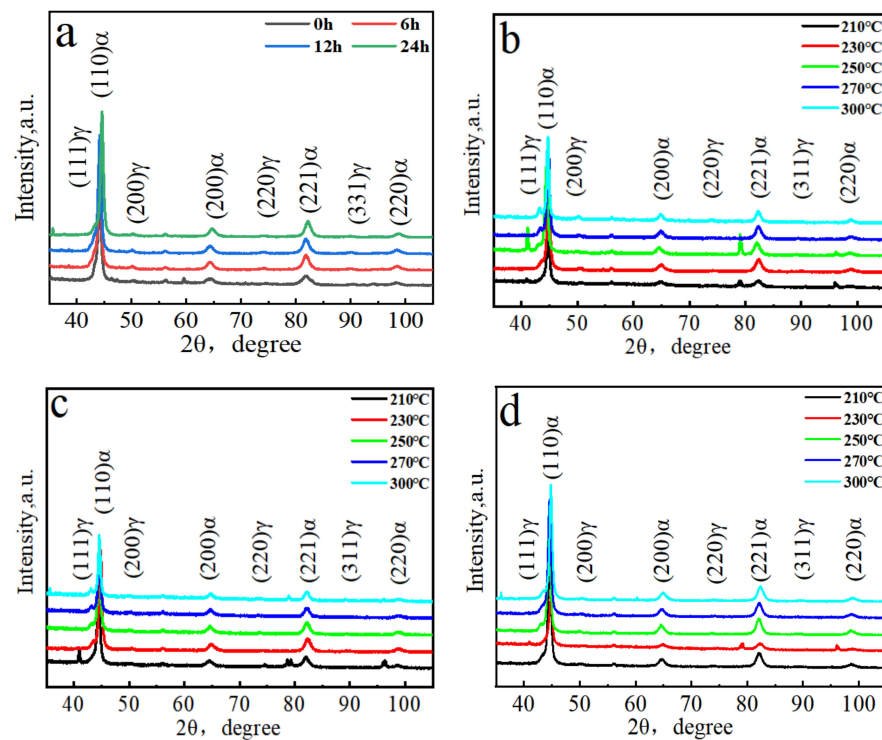


Figure 3. XRD patterns of different heat treatments processes ((a) is at isothermal temperature of 210 °C for 6/12/24 h; (b–d) are at different isothermal temperatures for 6/12/24 h).

Interestingly, the results show that the oil-quenched steel also has a high retained austenite content (14.7%), and this is because under the oil quenching process, a lot of super-cooled austenite has no time to transform into martensite, because of the great supercooling degree and the rapid reaction. In addition, the residual stress generated by this process is very high [1]. Even after tempering, the residual stress cannot be eliminated completely, and the carbon content in the retained austenite is low, resulting in the metastable retained austenite.

Figure 4 shows the SEM images of the samples austempered at 210 °C and 230 °C. Evidently, the undissolved carbides (the white particles in the red circles are the representative) are distributed uniformly as an approximate spherical shape in the steel. The microstructure of the sample austempered at 210 °C consists of bainitic ferrite, martensite, retained austenite, and undissolved carbide. The bainitic ferrite is distributed in the form of fine needles, while the martensite is coarser than bainitic ferrite and exists in lath. The fine acicular bainitic ferrite becomes denser with increasing isothermal time, which also suggests that the amount of bainite increases with increasing isothermal time. However, under the same isothermal time, the amount of bainitic ferrite increases significantly with the increase in the isothermal temperature. The bainite transformation is mainly controlled by the diffusion of carbon. Below the nasal tip temperature of the bainite transformation kinetics C-shaped-form curve, the higher the phase transformation temperature is, the faster the phase transformation speed is. Therefore, a greater amount of bainite is formed under the same transformation time at the higher transformation temperature. In addition, it is not difficult to find that with increasing isothermal temperature or isothermal time, the amount of coarse martensite decreases obviously. Figure 5 shows the SEM images of the steel after the oil quenching and tempering process. Clearly, the microstructure is composed of martensite, retained austenite, and undissolved carbides, as we have said for the normal quenching and low-temperature tempering microstructure [15].

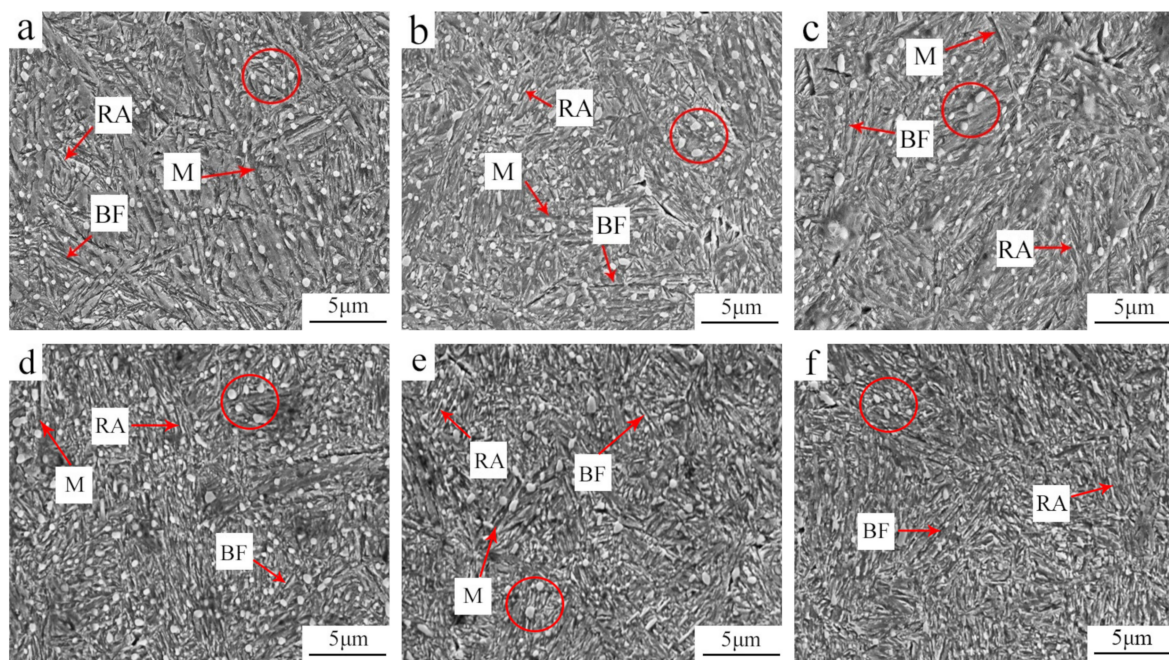


Figure 4. SEM images: (a–c) are at 210 °C for 6, 12, and 24 h; (d–f) are at 230 °C for 6, 12, and 24 h, respectively. Notes: BF—bainitic ferrite, M—martensite, and RA—retained austenite.

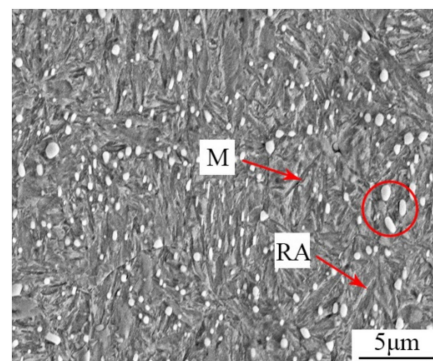


Figure 5. SEM images of the steel after oil quenching and tempering process. Notes: M—martensite, RA—retained austenite.

In order to study the compositions of these undissolved carbides, a local energy-dispersive spectrometer chemical analysis was performed. The results are shown in Figure 6. The compositions of carbide Spot 1 and carbide Spot 2 are very similar, and both have a relatively high content of Cr and V, both of which belong to the carbide-forming elements. Therefore, it can be inferred that the types of undissolved carbide are mainly Cr-carbide and V-carbide.

Excess undissolved carbides in the steel will increase the brittleness and damage the mechanical properties. However, the undissolved carbides can provide many hard points for the steel to enhance hardness and improve wear resistance [16–18]. Furthermore, the undissolved carbides can also pin the grain boundaries and inhibit the growth of grains. Thus, the grain can be refined and alloying elements can be absorbed if a small amount of carbides exist in the steel. The volume fraction of undissolved carbide ranging from 5% to 8% is appropriate for bearing steel. If the undissolved carbides are dispersed in the matrix with the fine and uniform sizes, the service life of bearing steel can be improved by suitable heat treatment.

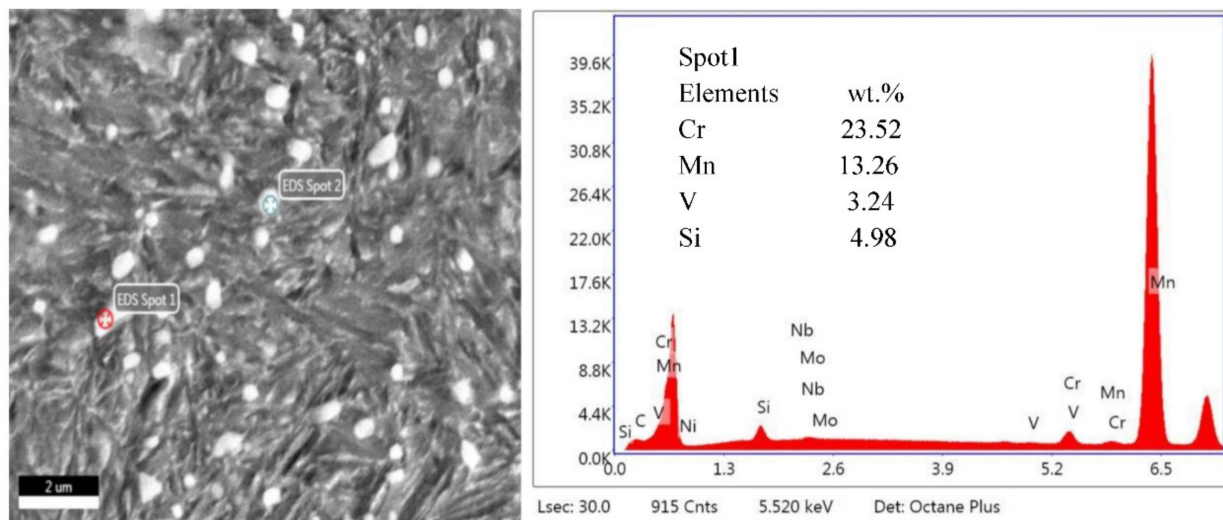


Figure 6. Energy-dispersive spectrometer analysis of undissolved carbides (210 °C × 24 h).

There were undissolved carbides in the experimental steel; however, no carbides were precipitated during bainite transformation. This is due to the large amount of silicon (1.4 wt.%) added to the steel, which prevents carbide precipitation. The microalloyed bearing steel designed in this paper obtained a fine microstructure after the isothermal heat-treatment process. The addition of V/Nb is beneficial to refine the grain. It is easy for V to form carbides in steel, and the carbides can pin the grain boundaries to inhibit grain growth and refine grains [5]. Nb not only has the same effect as V for grain refinement but can also form compounds with other elements in the steel. The Nb compounds can provide more nucleation sites for bainite transformation [19], thus accelerating the bainite transformation.

The equivalent diameters, size distribution, and percentages of the undissolved carbides were calculated based on a total of 10 SEM images by IPP software. The volume fractions of undissolved carbides under different isothermal processes are shown in Table 4, which suggests that the volume fractions of undissolved carbides are between 5% and 6%. The proper volume fraction of undissolved carbides is beneficial to improve the wear resistance and will not increase the brittleness of the steel seriously. We assume that all of the undissolved carbides are regular round shapes. Thus, we can calculate the equivalent diameter based on the area of the undissolved carbide particles under SEM. The equivalent diameter distributions of undissolved carbides of the sample's isothermal process for 6 h at 230 °C are shown in Figure 7. Apparently, the equivalent diameters of undissolved carbides are all between 0.1 and 0.8 μm and less than 1 μm . The fine and uniform undissolved carbides are able to improve the wear performance and extended the rolling contact fatigue life of bearing steel greatly.

Table 4. Volume fraction of undissolved carbides under different isothermal processes, vol.%.

Isothermal Temperature	0 h	6 h	12 h	24 h
210 °C	5.72	5.61	5.87	5.85
230 °C	5.72	5.66	5.76	5.79

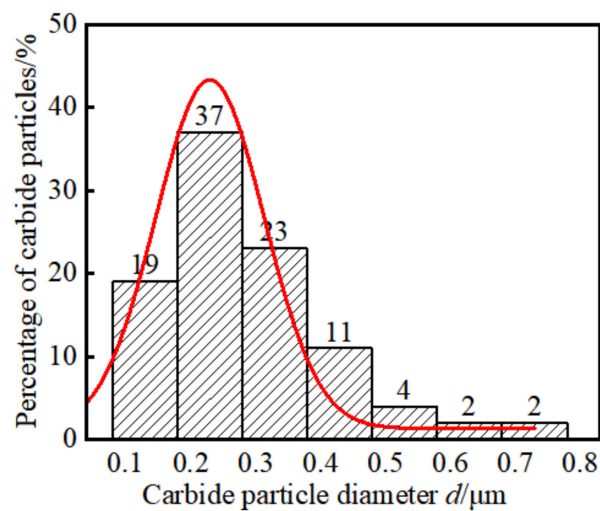


Figure 7. The undissolved carbide diameter distribution after isothermal process for 6 h at 230 °C.

3.2. Mechanical Performance Analysis

The experimental steel undergoes different heat treatments to obtain different microstructures, resulting in different mechanical properties. Table 5 shows the surface hardness of the tested steel. The oil quenching produces a large amount of martensite, which leads to the maximum value of hardness for the test steel. For the austempering processes, the isothermal time has little effect on the hardness. The hardness of the samples under the same isothermal temperature with different times changes little. However, the hardness decreases with increasing isothermal temperature. Table 5 suggests that when the isothermal temperature is 250 °C, the hardness value appears to be less than 60 Rockwell C hardness (HRC), and when the isothermal temperature is 300 °C, the hardness value is lower than 55 HRC, which does not meet the standard of bearing steel. With the isothermal temperature increasing from 250 °C to 300 °C, the contents of retained austenite and bainite increase gradually for the same isothermal time. Retained austenite and bainite exhibit better toughness and lower hardness than those of martensite [20], resulting in the decrease in hardness value.

Table 5. Hardness values of different isothermal processes/HRC.

Temperature/°C	6 h	12 h	24 h
210	62.4 ± 0.3	61.3 ± 0.1	61.4 ± 0.3
230	60.4 ± 0.3	60.4 ± 0.2	60.5 ± 0.1
250	58.2 ± 0.2	59.6 ± 0.3	60.4 ± 0.2
270	57.2 ± 0.2	58.4 ± 0.2	59.1 ± 0.3
300	54.1 ± 0.1	52.9 ± 0.3	54.8 ± 0.2
Oil quenching	65.3 ± 0.4		

Figure 8 reveals the functional form between impact toughness and isothermal process. The impact toughness of the isothermal process for 6–24 h shows a trend of decreasing first and then increasing. The reason is that the retained austenite reaches its maximum volume fraction when the isothermal time is 6 h. When the isothermal time reaches 12 h, as the supercooled austenite transforms into bainite gradually, the amount of retained austenite decreases but the bainite content does not reach the maximum, resulting in a slight decrease in impact toughness. When the isothermal time continues to extend to 24 h, the retained austenite continues to decrease, while the bainite content increases greatly, which improves the impact toughness with a slight increase.

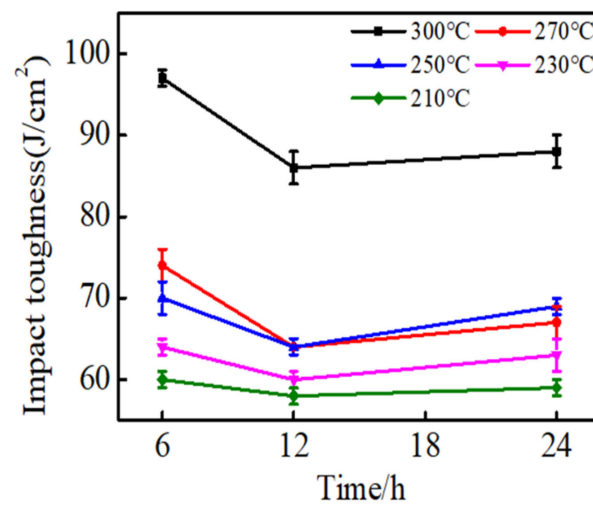


Figure 8. Impact toughness under different isothermal processes.

Figure 8 also indicates that the impact toughness increases as the isothermal temperature increases. With the increase in isothermal temperature, the amount of bainite increases. Bainite exhibits great plastic deformation resistance and can effectively hinder the initiation and propagation of cracks, thus improving the impact toughness [21]. The impact toughness of the experimental steel obtained by the oil quenching process is only 43 J/cm². Comparing the two processes, the impact toughness of these austempering samples is much higher than that of the oil quenching sample. After oil quenching, a large residual stress will be generated, which makes the steel easily produce cracks. After being subjected to a small impact, the unstable retained austenite will transform into untempered fresh martensite [22,23]. The fresh martensite makes the crack expand rapidly and greatly reduces the plasticity and toughness. However, for these austempering samples, the quenching stresses are less [24] and extremely fine bainite is produced in the microstructure, which are responsible for the excellent impact toughness.

The tensile strengths of the experimental steel are shown in Table 6. Obviously, the tensile strength of the austempering process is better than that of the oil-quenching process. Although the hardness of the oil-quenching process is highest, its tensile strength is not high enough. These experimental results indicate that tensile strength is not simply positively correlated with hardness, but closely related to the microstructure.

Table 6. Tensile strength of different isothermal processes/MPa.

Isothermal Time	6 h	12 h	24 h
210 °C	1960 ± 10	2150 ± 9	2301 ± 9
230 °C	2126 ± 8	2283 ± 11	2320 ± 10
250 °C	2100 ± 9	2133 ± 8	2204 ± 8
270 °C	2072 ± 10	2107 ± 8	2122 ± 8
300 °C	1995 ± 9	1807 ± 9	1799 ± 10
Oil quenching	1900 ± 11		

Figure 9 shows the tensile strength versus isothermal temperature and time. With the extension of the isothermal time at 210–270 °C, the tensile strength gradually increases. The amount of bainite increases with increasing isothermal time. In addition, the V_{γ} decreases with increasing isothermal time (Table 2), resulting in the loss of plasticity and toughness but improvement of strength. Bhadeshia et al. [25] and Zhao et al. [26] have reported that the incredibly fine bainite would be obtained after austempering at low transformation temperature for a long time, which is responsible for the high strength.

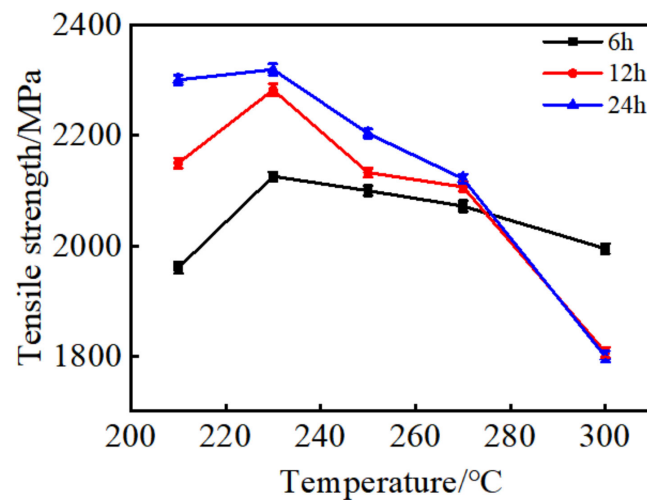


Figure 9. The curves of tensile strength versus isothermal temperature and time.

Figure 9 also suggests that the tensile strengths increase first and then decrease with increasing isothermal temperature. The volume fractions of bainite of the sample austempered at 210 °C for different times are less than those at 230 °C. In addition, the volume fractions of retained austenite of the sample austempered at 210 °C for different times are more than those at 230 °C. Therefore, the tensile strengths of the sample austempered at 210 °C are lower than those at 230 °C held for the same time. The tensile strength of the experimental steel achieves the best value when the sample is austempered at 230 °C. When the isothermal temperature continues to increase, the tensile strengths decrease significantly, especially when the isothermal temperature reaches 300 °C. The microstructure becomes coarser at higher austempering temperature [27], which explains the loss of strength of the steel at higher austempering temperature.

The mechanical properties are compared with the bearing steel with the composition of Fe-1.03C-1.50Cr-0.47Si-0.35Mo-0.23Mn, wt.% (without Nb and V) [28]. We find that not only is the hardness of Nb/V microalloyed bearing steel slightly higher but the impact toughness is also significantly improved, which is attributed to the addition of Nb and V.

The results suggest that the mechanical properties of Nb/V microalloyed bearing steel do not have linear relationships with isothermal temperature or isothermal time. The optimum comprehensive mechanical properties can be achieved by a reasonable heat treatment.

4. Conclusions

The conclusions of this study are as follows:

At the same isothermal temperature, $V\gamma$ gradually decreased with increasing isothermal time. As the isothermal temperature increased, the $V\gamma$ first decreased and then increased. This is because the rising isothermal temperature increases the amount of bainite transformation, resulting in a decrease in retained austenite. When the isothermal temperature continued to increase, the carbon atoms were able to fully diffuse into the retained austenite to increase its stability and $V\gamma$.

After spheroidizing annealing and austempering, the percentage of undissolved carbide was between 5% and 6%. The undissolved carbides were dispersed in the matrix and their average equivalent diameter was 0.31 μm ($<1\mu\text{m}$). The undissolved carbides can pin grain boundaries, which is conducive to the nucleation of bainite, resulting in a fine microstructure.

As the isothermal time increased, the hardness changed slightly. However, the hardness decreased as the isothermal temperature increased. The impact toughness was enhanced with rising isothermal temperature. The tensile strength increased with the isothermal time. The tensile strength achieved the best when austempered at 230 °C, and it

decreased when the isothermal temperature increased continuously. The remarkable comprehensive mechanical properties with a hardness of 60.4 ± 0.3 HRC, tensile strength of 2126 ± 8 MPa, and impact toughness of 63 ± 1 J/cm² were achieved when the experimental steel was austempered at 230 °C for 6 h.

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Abbreviations

bainitic ferrite	BF
Martensite	M
retained austenite	RA
martensitic phase transition point	Ms
X-ray diffraction	XRD
austenite phase	γ
ferrite phase	α
volume fraction of retained austenite	V γ
scanning electron microscopy	SEM
Image-Pro Plus	IPP
Rockwell C hardness	HRC

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