Insights into effect of first-step austempering temperature on the microstructure and properties of austempered ductile iron

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Abstract

Austempered ductile iron (ADI) is a revolutionary material with high strength and hardness combined with excellent ductility and toughness. The discovery of a two-step austempering process has resulted in superior combination of all the mechanical properties. Therefore, ADI has many different applications in varying fields. One notable application of ADI is in transmission components for new energy vehicles. As the automotive continues to develop, ADI transmission components with higher damping properties are required, because the damping properties of ADI affect the vibration and noise of the transmission system. However, the mechanism behind the damping of ADI, as well as a full characterisation of the damping properties, has not been thoroughly investigated in the literature. Therefore, it is necessary to reveal the influencing factors and mechanism of the damping properties of ADI. In this paper, the effect of first-step austempering temperature on the microstructure, mechanical properties, and damping properties of ADI was investigated. The results revealed that the tensile strength, yield strength, and elongation of ADI all initially increased, until reaching a global maximum value (1320 MPa, 1230 MPa, and 13.2%, respectively) when the first-step austempering temperature was 280 °C, followed by a decrease in value. Interestingly, the impact energy of ADI initially decreased before reaching a global minimum and then increasing to a maximum value of 93.5 J, when the first-step austempering temperature reached 320 °C. With increasing strain amplitude, the internal friction values ($Q^{-1}$) of ADI treated with different first-step austempering temperatures all increased gradually, and the $Q^{-1}$ values of ADI with first-step austempering temperatures of 240 and 260 °C were higher than those of other samples. Moreover, the damping properties of ADI varied with frequency with obvious S-K peaks and Ge peaks.

1. Introduction

Austempered ductile iron (ADI) is a kind of ductile iron with acicular ferrite and austenite structure that is obtained by austempering [1]. At present, austempering process of ADI can be divided into two categories: single and two-step austempering processes [2]. In a single-step austempering process, fine acicular ferrites can be obtained under the condition of low temperature austempering, but the content of the retained austenite and its carbon content are low. Therefore, although the strength and hardness of the ADI are high, the plasticity and toughness are low [3]. Correspondingly, when the retained austenite and carbon content increase, the acicular ferrite structure becomes course. This leads to a notable decrease in its strength and hardness, while experiencing an increase in the plasticity and toughness of the material [4]. Therefore, it is difficult to improve both the strength and toughness simultaneously by single-step austempering process [5]. The two-step austempering process has been developed to address limitations of the single-step process, and thus, it is becoming increasingly popular as the research space gains more traction [6–12].
The mechanical properties (e.g. strength, wear resistance, fatigue resistance, and other comprehensive properties) of ADI are superior than those of other ductile cast irons \[13-15\]. Additionally, the strength to weight ratio of ADI is also high \[16\]. Therefore, ADI is widely used in the fields of transportation, energy, industrial equipment, etc \[17\]. In addition, ADI has the advantages of self-lubricity and low specific gravity in comparison with low-alloy steel, so it is gradually replacing low-alloy steel in transmission gears and other transmission devices \[18\], which broadens the application prospect of ADI in the transmission components of new energy vehicles \[19-21\]. However, the damping properties of ADI affects the vibration and noise of the transmission system. Therefore, with the development of the automobile industry, ADI transmission components with higher damping properties are required \[22\]. Further, it is of great theoretical and practical value to study the factors influencing the damping properties of ADI and elucidate its mechanism.

Currently, scientists have conducted many research studies on ADI, but the majority of these works focus on the influence of microstructures on mechanical properties, and there are limited studies on the damping properties of ADI \[23-30\]. In this study, the structures of the ADI matrix were tuned by adjusting the austempering temperature in the first step, and the mechanical and damping properties of the ADI were investigated. The main impacts of matrix structures on the mechanical and damping properties of ADI were studied by adjusting the first-step austempering temperature to establish the relationship among first-step austempering temperatures, microstructures, mechanical properties, and damping properties.

2. Experimental section

2.1. Experimental material

The raw materials used in this study were pig iron, carbon steel, alloy ferrosilicon, alloy ferromanganese, vermicular agent, and electrolytic copper, as listed in table 1. RE3Mg8 was used as the nodulariser and 75SiFe as the inoculant. A medium-frequency induction furnace (GWJ-0.35, Xi’an Sigma Equipment Co., Ltd China) was used for melting. After the material in the furnace was melted, the temperature was increased to 1550 °C ± 5 °C and the spheroidising agent, the inoculant, and the covering agent were poured into the ladle to inoculate it. When the temperature of the molten iron dropped to ~1350 °C, the inoculation treatment was poured into the sand type of the Y-shaped test block prepared in advance (according to ISO16112:2006). After cleaning the samples and cutting into standard dimensions, the austempering treatment was conducted by using an external heated salt bath furnace with 50% KNO₃ + 50% NaNO₃. The austempering process is shown in figure 1, and the parameters are listed in table 2. The samples with different first-step austempering temperatures were numbered separately, viz L1 (240 °C), L2 (260 °C), L3 (280 °C), L4 (300 °C), and L5 (320 °C).

2.2. Experimental method

The metallographic samples were ground, polished, and etched using a 4% nitric acid solution. The microstructure was observed by metallographic microscope (OM; Axio Vert. A1, Carl Zeiss, Germany). The
impact fracture was observed using a scanning electron microscope (SEM; S-4300, Hitachi Corporation, Japan). The substrate and interface were observed using a transmission electron microscope (TEM; JEM2100, Japan Electronics Co., Ltd). X-ray diffraction (XRD) analysis was used to calculate austenitic quantity and its carbon content. A D/max-2600/PC X-ray diffractometer (Rigaka, Japan) was employed with Cu Kα radiation at 40 kV and 150 mA, and a scattering rate of 3 °C·min⁻¹ in the 2θ range of 30°–100°. The specimen had dimensions of 10 mm × 10 mm × 1.5 mm. The volume fraction of the retained austenite was estimated using the following relationship [31]:

\[
\frac{I_{\gamma\{hkl\}i}}{I_{\alpha\{hkl\}j}} = \frac{R_{\gamma\{hkl\}X_\gamma}}{R_{\alpha\{hkl\}X_\alpha}}
\]

where \(I_{\gamma\{hkl\}i}\) and \(I_{\alpha\{hkl\}j}\) are the integrated intensities of a given \{hkl\} plane from the austenite and ferrite, respectively, \(X_\gamma\) and \(X_\alpha\) are the volume fraction of austenite and ferrite, respectively, and \(R_{\gamma\{hkl\}X_\gamma}/R_{\alpha\{hkl\}X_\alpha}\) was the ratio of intensity factor corresponding to the crystal plane of \{hkl\}, from austenite and \{hkl\}, from ferrite. The \{200\}, \{220\}, and \{311\} planes of austenite and \{200\} and \{211\} planes of ferrite were used to analyse the volume fraction of austenite. The volume fraction of austenite was calculated by \(L_{\gamma\{hkl\}}/L_{\alpha\{hkl\}}\), and the reported result was the average of five repeated measurements. The carbon content of austenite was determined by the equation:

\[
a_\gamma = 0.358 + 0.0044C_\gamma
\]

where \(a_\gamma\) was the lattice parameter of austenite (nm) and \(C_\gamma\) is the carbon content of austenite (wt.%).

The tensile test was carried out according to ISO 6892, using a universal tensile testing machine (WDW-E, Jiangsu Jinma Co., Ltd, China), and a tensile sample is schematically shown in figure 2. The stretching speed during the test was chosen as 4 mm·min⁻¹. To ensure the accuracy of the test data and minimise error during the test, the samples of each process were tested three times and the results averaged.

The unnotched Charpy impact test was performed according to Standard GB/T 229-2007 on a pendulum impact testing machine (NI300, The NCS Testing Technology Co., Ltd, China) at room temperature. The specimen had dimensions of 55 mm × 10 mm × 10 mm. To ensure accuracy of the test data and to minimise error during the test, the samples for each process were tested six times and the results averaged.

The damping performance test was carried out using a dynamic thermomechanical analyser (DMA; Q800, TA Instruments, America) with sample dimensions of 45 mm × 10 mm × 1 mm.

3. Results and discussion

3.1. Microstructure analysis

Figure 3 shows the metallographs of two-step ADI with different first-step austempering temperatures. For L1, the acicular ferrite can be clearly distinguished in the field of vision and the bright white-coloured retained
The austenite phase is uniformly distributed in the ferrite gap. As the austempering temperature increases to 280 °C (L3), acicular ferrite and retained austenite are uniformly distributed, but a small amount of austenite can still be observed in the structure. With further increase in the first-step austempering temperature, the distribution of the retained austenite phase in L4 and L5 tended to be uneven, and large bright white areas are present in the structure. Although acicular bainite and retained austenite are distributed in the bright white area, the content of acicular bainite is significantly reduced.

The metallographs (figure 3) show that with increasing first-step austempering temperature, the acicular structure of ferrite gradually grows and thickens. An obvious coarsening effect and an uneven distribution of the retained austenite phase is also seen. This is because under the same austenitisation and two-step austempering processes, lower first-step austempering temperatures can result in greater driving force for phase transformation, greater number of nucleation during isothermal transformation, and smaller critical grain size.
Meanwhile, during isothermal transformation, carbon atoms are rapidly discharged from the newly formed ferrite phase. Due to the short diffusion distance of fine carbon atoms in grains, the carbon content of residual austenite increases rapidly, and the content of stable retained austenite is also higher.

To analyse the influence of first-step austempering temperature on ADI microstructure more accurately, XRD characterisation was performed, the results of which are shown in figure 4 and the contents of the retained austenite phase after calculation are shown in table 3.

Table 3 shows that with the increase of first-step austempering temperature, the content of retained austenite in ADI matrix reduces significantly. When the first-step austempering temperature was higher than 280 °C, the retained austenite content increased slightly. Hence, the amount of retained austenite was larger at lower first-step austempering temperatures. This is because under the constant austenitising parameters, the lower first-step austempering temperature can result in a greater driving force for phase transformation, a greater amount of ferrite nucleation, and a smaller critical nucleation radius. After the same second-step austempering treatment, the ferrite phase is finer. In the first-step austempering treatment, the greater the amount of nucleation, the shorter the average diffusion distance of carbon atoms becomes. In the second-step austempering treatment, the high austempering temperature also accelerates the diffusion of carbon atoms, which rapidly increases the carbon content in austenite, forming stable retained austenite.

The TEM micrographs of ADI with different first-step austempering temperatures (figure 5) show that the ferrite bundle is composed of alternate ferrite lath and thin film austenite. With increasing first-step austempering temperature, the thickness of thin film austenite and the dislocation density in ferrite lath both increased greatly. When the first-step austempering temperature increased further, no significant change was
observed in the dislocation density in the matrix, but the dislocation density at the graphite-matrix interface increased greatly. A massive austenite structure was also observed in the matrix.

3.2. Fracture analysis
Figure 6 exhibits the fracture morphology of impact specimens. A characteristic cleavage fracture, consisting of cleavage plane and river pattern, is present in the fracture surface of L1. A small deformation can be observed in the matrix around the graphite nodules. With increasing first-step austempering temperature, a large number of dense and fine dimples are distributed around the graphite nodules, and many cleavage planes are observed between the graphite nodules, as shown in figure 6(c). With increasing one-step austempering temperature, a large number of crystal sugar morphologies appeared in the fracture surfaces of L3 and L4, while no deformation was found in the matrix around the graphite nodules. The fracture mechanism changed from cleavage to quasi-cleavage and then eventually transformed to brittle with increasing first-step austempering temperature.

3.3. Mechanical property analysis
Figure 7 shows the mechanical properties of two-step ADI with treated under different first-step austempering temperatures. As can be inferred from figure 7, when the first-step austempering temperature is 240 °C, the tensile strength and yield strength of ADI are 1190 MPa and 960 MPa, respectively. When the first-step austempering temperature rises, there is a gradual increase in the tensile and yield strengths of ADI, which reach a maximum (1320 MPa and 1230 MPa, respectively) at 280 °C. With continued rise in temperature, the tensile and yield strengths of ADI decrease rapidly, reaching 1080 MPa and 880 MPa at 320 °C. Furthermore, with increasing first-step austempering temperature, the elongation of ADI initially increases and then decreases from 10% at 240 °C to 13% at 280 °C, finally falling to 8.5% at 320 °C. Meanwhile, the reduction of area of ADI exhibits a gradually decreasing trend, reaching a minimum of 7.5% at 320 °C. The impact energy of ADI fluctuates with an increase in the first-step austempering temperature, which shows an initial decline and a subsequent rise. Moreover, when the first-step austempering temperature is higher than 280 °C, the impact energy exhibits negligible changes, reaching a maximum of 93.5 J at 320 °C.

3.4. Damping property analysis
Figure 8 shows the effect of strain on the damping property of ADI processed under different first-step austempering temperatures. The internal friction value ($Q^{-1}$) of all samples fluctuates in a small range when the strain is small, and as the strain amplitude increases, $Q^{-1}$ continues to increase. The internal friction values of L1 and L2 are significantly higher than those of other samples, mainly resulting from the retained austenite content in the matrix structure.
It is evident from figure 5 that the dislocation densities in the ferrite and at the matrix-graphite interface increase considerably with increasing first-step austempering temperature. Dislocations consume more energy when they get rid of pinning, which effectively enhances the vibration energy consumption and damping property. However, it is evident from figure 9 that the $Q^{-1}$ values of L1 and L2 with lower dislocation densities are significantly higher than those of other samples with higher dislocation densities. This is primarily attributed to the higher retained austenite content in L1 and L2 compared with other samples, resulting in higher plasticity and toughness. Therefore, the energy absorbed by L1 and L2 during vibration is significantly greater than that consumed by the increase of dislocation density and interface area.
4. Conclusions

The effect of first-step austempering temperature on the microstructure and the mechanical and damping properties of ADI was investigated. Some significant conclusions include the following:

1. As the first-step austempering temperature increases, the acicular ferrite structure of ADI sample coarsens gradually. When the temperature reaches 280 °C, the structure is most compact and uniform. When the temperature continues to increase, the coarsening effect of acicular ferrite is obvious, and the structure is noticeably uneven.

2. When first-step austempering temperature reaches 280 °C, ADI has the best comprehensive mechanical properties. The tensile strength, yield strength, and elongation of ADI all reach a maximum value (1320 MPa, 1230 MPa, and 13%, respectively).

3. With the increase of strain amplitude, Q⁻¹ of ADI of different first-step austempering temperature all increase. The higher the content of retained austenite, the greater the internal friction value obtained. Moreover, the internal friction value of ADI rises gradually with an increase in the frequency. There are two conspicuous damping peaks observed near 160 Hz (S-K peak) and 190 Hz (Ge peak).

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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