Influence of Tempering on the Mechanical Properties of Austempered Ductile Iron

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This paper aims to study the influence of tempering on the mechanical properties of austempered ductile iron. A modified austempering treatment was carried out to obtain a microstructure mixture of ausferrite and tempered martensite by stopping the austempering reaction before stage 1 reaction was completed, and resuming tempering at 473 K for 7.2 ks after cooling to room temperature. The change in the microstructure and the related changes in mechanical properties of ADI after tempering were investigated. The results show that tempering at 473 K after austempering decreased the sensitivity of mechanical properties to austempering conditions. Independent of austempering temperature, tempering at 473 K also led to significant improvement in mechanical properties with increasing martensite content. Strength and toughness could be improved to exceed the maximum values obtained without tempering, and these strengthening effects were particularly obvious at martensite content below 5%. With tempering at 473 K, the required austempering time for obtaining the maximum values of mechanical properties could be shortened from 10.8 ks without tempering to 1.8 ks with tempering, and the effective austempering time interval was also extended.

(Received February 12, 1999; In Final Form July 23, 1999)

Keywords: *tempered martensite, austempered ductile iron, mechanical properties, ausferrite, processing window*

I. Introduction

The mechanical properties of austempered ductile iron (ADI) are closely related to the selection of heat treatment parameters. During austempering, the austenite matrix of ductile iron undergoes a two-step reaction. In stage 1 reaction, austenite is transformed into ferrite and high carbon austenite, while in stage 2 reaction, the high carbon austenite decomposes into ferrite and carbide⁽¹⁾⁽²⁾. A microstructure of ADI and a schematic of the eutectic cell and intercellular boundary region are shown in Fig. 1. This microstructure is quite different from the case of steels. It consists of blocky unreacted austenite and a two-phase mixture of ferrite and high carbon austenite, named ausferrite. Details of the terminology and structure of ADI have been described by Kovacs⁽³⁾. The morphology of ferrite austempered at higher temperature is feather-shape. Hence, for convenience this mixture structure and this ductile iron are called upper ausferrite and upper ADI, respectively. Similarly, that austempered at lower temperature are named lower ausferrite and lower ADI. Before stage 1 reaction is finished, the unreacted austenite will transform into martensite if quenched to room temperature due to its low carbon content. Both martensite and carbide reduce the ductility and toughness of ADI. Therefore, a desired austempering treatment is to extend the isothermal holding time between the end of stage 1 reaction and the onset of stage 2 reaction in order to obtain the largest ductility and toughness and to satisfy the ADI standard (ASTM A897M-1990)⁽⁴⁾. This time period is called the proc-

essing window. However, the duration of the processing window is affected by variables including segregation of added alloys, austenitizing temperature and austempering temperature. When the austenitizing and/or austempering temperature increases, the processing window is reduced to a small range or, eventually vanishes⁽⁵⁾⁽⁶⁾.

The processing window is also reduced by segregation of alloy additions⁽⁶⁾, which causes the reaction rate to become faster in the eutectic cell and slower in the intercellular boundary⁽⁷⁾. From an engineering point of view, it is obvious that the mechanical properties of ADI are very sensitive to austempering conditions and alloy additions, thus increasing the difficulty for successful production of ADI.

A two-step heat treatment process was proposed to solve the narrowing of the processing window as a result of alloy additions. First, austempering was conducted at high temperature until stage 1 reaction took place in the intercellular boundary. Then, the austempering temperature was reduced. This increased the driving force for stage 1 reaction in the intercellular boundary and retarded that for stage 2 reaction in the eutectic cell. As a result, upper ausferrite was formed in the eutectic cell while lower ausferrite was formed in the intercellular boundary⁽⁸⁾⁽⁹⁾. This two-step treatment prevented effectively the formation of martensite and carbide, but it needed a longer isothermal holding time.

It is commonly held that tempering can increase the toughness of as-quenched martensitic microstructure⁽¹⁰⁾. The previous study indicated that tempering at 473 K could increase the ductility and toughness of ADI having

Fig. 1 Typical microstructure of ADI and schematic of eutectic cell and intercellular boundary region. [A: ausferrite, γ_u : blocky unreacted austenite]

a mixed structure of ausferrite and martensite austempered at 673 K⁽¹¹⁾. The same result was also observed by Rounds *et al.* in the study of ADI austempered at 644 K followed by tempering at 644 K⁽¹²⁾. The results mentioned above show that the ductility and toughness of upper ADI having a mixed structure of ausferrite and suitable amount of tempered martensite might be of the same level as those of ausferrite structure alone.

Only a few attempts have been made to examine the influence of tempered martensite on the mechanical properties of ADI. Tomita and Okabayashi have reported the relative variations in mechanical properties of steel having a mixed structure of various types of bainite and tempered martensite in the study of high strength steel⁽¹³⁾. Lower bainite mixed with tempered martensite significantly improved mechanical properties resulting in larger strength and ductility than those of lower bainitic structure alone. However, the mixture of upper bainite and tempered martensite possessed less ductility than that of upper bainitic structure only. In ADI, the matrix consists of ferrite, martensite, and a lot of high carbon austenite. This microstructure is different from that of steel, therefore, the influence of tempered martensite on the mechanical properties of ADI may be different.

In this paper, the isothermal holding of austempering was conducted at the various stages of reaction to obtain an ADI with different martensite contents. Then, the ductile iron was tempered at 473 K for 7.2 ks to obtain a mixed structure of ausferrite and tempered martensite. The mechanical properties of ADI after tempering were compared with those of ADI without tempering treatment to shed light on the influence of tempered martensite on the mechanical properties. The effect of tempering at 473 K on the increasing mechanical properties and the decreasing sensitivity to austempering conditions were discussed on the basis of the ADI standard⁽⁴⁾.

II. Experimental Procedure

The ductile iron used in this study was melted in a high frequency induction furnace in a commercial foundry,

then poured into the ladle in which nodularization was carried out by the sandwich method⁽¹⁴⁾. Rods with dimensions $\phi 25 \text{ mm} \times 250 \text{ mm}$ were cast in green sand molds. The chemical composition of ductile iron was 3.52% C, 2.63% Si, 0.18% Mn, 0.77% Cu, 0.50% Ni and 0.037% Mg in mass%.

Casting bars were homogenized at 1233 K for 86.4 ks and annealed at 973 K for 10.8 ks. Then, tensile specimens of $\phi 6 \text{ mm}$ diameter as shown in Fig. 2, 10 mm $\times$ 10 mm $\times$ 55 mm unnotched Charpy impact specimens, and $\phi 20 \text{ mm} \times 20 \text{ mm}$ metallurgical specimens were machined before austempering treatment. These specimens were austenitized in a salt bath at 1173 K for 3.6 ks, and were then rapidly quenched in another salt bath for austempering at 573, 598, 623 and 648 K. The isothermal holding times were kept in the range of 90 and $1.2 \times 10^6 \text{ s}$. Finally, the specimens were water quenched to room temperature. The tempering treatment was conducted at 473 K for 7.2 ks to obtain a mixed structure of ausferrite and tempered martensite. The isothermal holding times used in austempering were derived from a resistivity curve obtained in the previous study⁽¹⁵⁾. Chen and Lei had also recorded the full reaction progress, and defined clearly the stage 1 reaction, the processing window and the stage 2 reaction⁽¹⁵⁾.

Tensile specimens were tested on a hydraulic Shimadzu model 48-25 universal testing machine with a constant cross beam travel speed of 1.2 mm/min. Unnotched Charpy impact specimens were tested in Tinivis Olsen impact machine at room temperature. Metallurgical specimens were polished, etched and then heated in an electrical oven at 528 K for 21.6 ks for heat tinting. After heat tinting, quantitative measurements of the volume fraction of unreacted austenite, X_{uy} , were estimated by the systematic point technique. The unreacted austenite would transform into martensite when cooled to room temperature or subjected to external force. Hence, X_{uy} was taken as the volume fraction of martensite, X_M , in this paper.

III. Results and Discussion

1. Characteristics of stage 1 reaction

Before stage 1 reaction was completed, the unreacted

Fig. 2 The shape and dimensions of tensile specimen (mm).

austenite would transform into martensite. **Figure 3** shows the variation in X_M with austempering time. X_M decreased rapidly during the initial period of the stage 1 reaction. The X_M shown in Fig. 3 was measured from the heat tinted metallurgical specimens. After about 540 s of austempering, the values of X_M decreased to below 50%; it further decreased to 10% after austempering for 1.08 ks. As austempering time increased in the later half of stage 1 reaction, the phase transformation rate of the four austempering temperatures became evidently slower and longer austempering time was needed to reduce the martensite content. For X_M to be decreased to 1%, the required isothermal holding time of austempering at 573, 598, 623 and 648 K needed to be increased to 1.8, 2.16, 2.64 and 3.12 ks, respectively.

In general, stage 1 reaction at the four austempering temperatures follows the reaction mode of Johnson-Mehl equation⁽¹⁵⁾, and is a type of controlled nucleation and growth of diffusion mechanism. In the initial period, the transformation rate of austenite is faster. As austempering time increases in the final period, the transformation rate is slowed down due to the increased average carbon content of austenite and the decreased driving force of transformation.

The austempering times for decreasing the martensite content from 10 to 1% of the four austempering temperatures were approximately within the range of 0.6–1.8 ks after 1.08 ks of austempering. If the embrittlement effect of martensite formed in this time interval could be improved by tempering, then the operation of austempering treatment would be more flexible, *i.e.*, the sensitivity of mechanical properties to austempering conditions could be improved to increase the reliability of production. Therefore, the variation in mechanical properties of specimen with martensite content below 10% is the focus of the present study.

2. Variation in microstructures

Figure 4(a) shows the microstructure of a specimen austempered at 598 K for 510 s after austenitizing at 1173 K for 3.6 ks, while Fig. 4(b) shows the microstructure of

Fig. 3 Variation in volume fraction of martensite with austempering time for austempering temperature of 573, 598, 623 and 648 K.

Fig. 4 Microstructures of (a) a specimen after austempering at 598 K for 510 s and (b) after austempering at 598 K for 510 s followed by tempering at 473 K for 7.2 ks.

a specimen tempered at 473 K for 7.2 ks after austempering at 598 K. In Fig. 4(a), the darker and lath-shaped area was ausferrite while the light and blocky area was unreacted austenite. This microstructure shows that the most ausferrite nucleated close to the graphite nodules or at prior austenite grain boundary during the initial period of reaction. On the other hand, the blocky unreacted austenite appeared in the intercellular boundary. It implies that the reaction rate in the intercellular boundary was slower than that in the eutectic cell. Figure 4(b) shows that the color of ausferrite became light gray while that of unreacted austenite became dark gray after tempering at 473 K for 7.2 ks. Since the unreacted austenite transformed into martensite on cooling to room temperature, the martensite could be etched more easily after tempering and therefore appeared darker. This structure is known as tempered martensite⁽¹⁶⁾.

As austempering time increased, ausferrite platelets increased while unreacted austenite decreased. As a result, the microstructure became finer and the stability of austenite surrounded by ausferrite increased due to the increasing carbon content. Thus, the volume fraction of martensite decreased. **Figure 5(a)** shows that the typical ausferrite structure without blocky austenite obtained after austempering at 598 K for 4.2 ks. The martensite content was below 1% in this structure indicating that the austempering reaction had progressed into the processing window. Figure 5(b) shows the microstructure of ausferrite alone after tempering at 473 K. The characteristics of ausferrite showed no obvious change as compared with those before tempering as shown in Fig. 5(a). This explains that the influence of tempering at 473 K for 7.2 ks on ausferrite was insignificant, but this tempering was effective in producing tempered martensite, eliminating the adverse effect of martensite including lattice distortion and transformation stress.

The variations in microstructure of specimens after austempering at 573, 623 and 648 K followed by tempering at 473 K were similar to those after austempering

Fig. 5 Microstructures of (a) a specimen after austempering at 598 K for 4.2 ks and (b) after austempering at 598 K for 4.2 ks followed by tempering at 473 K for 7.2 ks.

Fig. 6 Microstructures obtained by tempering at 473 K followed by air-cooling after austempering at (a) 573 K, 600 s, (b) 598 K, 510 s, (c) 623 K, 420 s and (d) 648 K, 360 s.

at 598 K as shown in Figs. 4 and 5, respectively. The only difference between these structures was that the size of ausferrite platelets became grosser with increasing austempering temperature.

Figure 6 shows the microstructure of specimens tem-

Fig. 7 Microstructures after austempering at (a) 573 K, 10.8 ks, (b) 598 K, 10.8 ks, (c) 623 K, 10.8 ks and (d) 648 K, 3.6 ks.

pered at 473 K after austempering at various temperatures during the initial period of stage 1 reaction. The areas of unreacted austenite increased with increasing austempering temperature resulting in a higher martensite content in upper ADI. The size of martensite platelets of upper ADI was also coarser than that of lower ADI. The influence of austempering temperature on the morphology of martensite was not obvious.

Figure 7 shows that the microstructure of specimen austempered within the processing window was ausferrite alone. The size of ausferrite platelets increased with increasing austempering temperature. The structure of ausferrite austempered above 598 K was feather-shaped while that austempered at 573 K was acicular-shaped.

3. Mechanical properties

Figure 8 shows the variation in mechanical properties of specimen austempered at different temperatures and for different duration. The line connecting data points that correspond to the martensite contents of 1, 5, 10 and 50% and the processing window derived from the resistivity curve are also shown in Fig. 8. The tensile strength, elongation and impact energy increased rapidly with austempering time from the initial period of reaction. These properties were at the lowest at the onset of reac-

Fig. 8 Variation in (a) tensile strength, (b) elongation and (c) impact energy with austempering time at various austempering temperatures.

tion because the amount of martensite was at the highest. As austempering time increased, the mechanical properties were increased as a result of the decrease in martensite volume fraction and increase in ausferrite content. The changes in these three mechanical properties with austempering time were different. Tensile strength reached a stable value at the martensite content below 5%, while elongation and impact toughness increased continuously and reached the maximum at the martensite content below 1% and then decreased again. The decrease in elongation and toughness was the result of decreasing high carbon austenite and carbide formation in stage 2 reaction. The maximum tensile strength appeared at the final period of stage 1 reaction while the maximum elongation and toughness appeared within the processing window. This shows that the influence of martensite content on elongation and toughness was more significant than that on tensile strength.

Figure 9 summarizes the variations in tensile strength with elongation and toughness. The ADI standard is also shown in Fig. 9. The mechanical properties of most austempered parts satisfied the standard requirement. As shown in Fig. 9, the mechanical properties of specimen austempered at 573 and 598 K satisfied the grade of 1400/1100/1 and 1200/850/4, respectively, while those after austempering at 623 and 648 K satisfied the grade of 1050/700/7. The martensite contents of specimen austempered at 573 to 648 K and barely reached the standard requirement fell in the range of 4–12%.

Figure 10 shows the relationship between martensite content and mechanical properties of ADI before and after tempering at 473 K. The open symbols represent the properties of ADI with austempering only, and the closed symbols represent the properties of specimens tempered at 473 K for 7.2 ks after austempering. The mechanical properties of specimen austempered within the processing window are shown at zero martensite content. These data represent the average of the three specimens. The influence of martensite content on the mechanical properties was compared on the basis of these values.

Fig. 9 Variation in tensile strength with (a) elongation and (b) impact energy. Numerals indicate austempering time in ks, and volume fraction of martensite is given in parentheses.

Fig. 10 Variation in (a) (b) tensile strength, (c) (d) elongation and (e) (f) impact energy with martensite content.

Figures 10(a) and (b) show that the influence of martensite content on tensile strength was different for various austempering temperatures. Although the strengthening effect could be seen at 1% of martensite content for austempering at 573 and 598 K, but the change in tensile strength with martensite content was insignificant. For austempering at 623 K, the strengthening effect on tensile strength was obvious at 4% of martensite content, while the tensile strength decreased with increasing martensite content after austempering at 648 K. Figures 10(a) and (b) also show that the tensile strength of ADI after tempering at 473 K was higher than that before tempering. Figures 10(c) and (d) show that the presence of martensite in the matrix decreased elongation. As martensite content increased, elongation decreased but could be improved after tempering at 473 K. The influence of martensite content on impact energy is shown in Figs. 10(e) and (f), which displayed a similar trend as that on elongation. In short, the influence of martensite content on mechanical properties was more significant for upper ADI and for elongation and toughness than for lower ADI and for tensile strength. The mechanical properties were increased after tempering at 473 K. As martensite content increased, the increase in the mechanical

properties was also enhanced, which was more obvious for elongation and toughness. The increase in strength, elongation and toughness after tempering were in the range of 1–7%, 5–90% and 10–80% respectively when compared with those of ADI before tempering.

Comparing the mechanical properties of ADI after tempering with those of ADI austempered within the processing window shown at zero martensite content. It is found that the tensile strength of all specimens after tempering at 473 K exceeded the values in the processing window. The elongation at martensite content below 5% and 3% for austempering at 573 and 623 K respectively were larger than those in the processing window, while the maximum elongation for austempering at 598 and 648 K were equal to those in the processing window. At the martensite content range of 2–5%, the impact energy of the four ADI after tempering were close to those in the processing window. The data mentioned above show that the amount of tempered martensite in ADI with obvious strengthening effect after austempering at 573 K was in the range of 1–5%, while those at 598, 623 and 648 K fell within 1–3%. Figure 10 also shows that the strengthening effects of tempered martensite were independent of the austempering temperature. This

situation was different from that for high strength steel⁽¹³⁾. It may be due to the presence of 20–40% high carbon austenite in the matrix in both upper and lower ausferrite.

The mechanism of transformation-induced plasticity (TRIP) of ADI has been investigated⁽¹⁷⁾⁽¹⁸⁾. The enhancement of ductility and toughness are attributed mainly to the suppression of the initiation and propagation of cracks by the formation of martensite during deformation and are dependent on the test temperature and the carbon content of austenite. In this paper, the high carbon austenite was quite stable under the tensile testing at room temperature. **Figure 11** shows the variation in volume fraction and carbon content of high carbon austenite with austempering time. The beginning of the martensite transformation, M_s could be estimated by using $M_s = 400 - 260(C_\gamma) + 273$ ⁽¹⁹⁾ while the onset of martensitic transformation caused by the deformation, M_d , would be at 70 K above the M_s ⁽²⁰⁾. The average carbon content in high carbon austenite with below 5% of tempered martensite and having obvious strengthening effect in ductility and toughness was above 1.8%. Because both the temperatures of M_s (205 K) and M_d (275 K) were below the test temperature (298 K), hence, the high carbon austenite would not transform into martensite during static tensile testing at room temperature. Further evidence can be found from the insignificant difference of high carbon austenite content of specimen austempered at 573 K before and after tensile testing as shown in Fig. 11(a). This result also coincides with the observation⁽¹⁸⁾

Fig. 11 Variation in (a) volume fraction and (b) carbon content of high carbon austenite with austempering time.

that the austenite was stable due to the higher carbon content and to the finer distribution of this phase in the microstructure. The improvement in ductility and toughness after tempering at 473 K could be attributed mainly to the elimination of lattice distortion and stress concentration associated with the formation of martensite during quenching.

Figure 12 shows the comparison of the maximum values of mechanical properties of ADI with and without tempering. The maximum values of mechanical properties of ADI without tempering were derived from Fig. 8. In Fig. 12, the numerals by data points indicated austempering time in ks. With tempering, the elongation was smaller than that without tempering but the tensile strength and toughness evidently exceeded those without tempering. It shows that tempering at 473 K could strengthen the mechanical properties resulting in larger tensile strength and toughness than those of ADI treated

Fig. 12 Variation in (a) tensile strength, (b) elongation and (c) impact energy with austempering temperature for the maximum values of mechanical properties within the martensite content range of 1–10%. Numerals indicate austempering time in ks.

Fig. 13 Variation in tensile strength with (a) elongation and (b) impact energy.

with austempering only. Although the elongation was still less than the maximum of that of ADI without tempering, the required isothermal holding time for obtaining the maximum values of mechanical properties could be shortened from an average of 10.8 ks without tempering to only 1.8 ks with tempering. It means that tempering at 473 K could be used to extend the effective range of austempering time into shorter end of treatment and to obtain the same mechanical properties of ADI treated with austempering alone.

The variation in tensile strength with elongation and toughness with and without tempering is shown in Fig. 13. In Fig. 13, ADI_{max} means the maximum value of mechanical properties of ADI before tempering, and ADI standard represents the required mechanical properties of ASTM standard. The experimental data of 0.37%Mn ductile iron treated with stepped austempering⁽²¹⁾ are also shown in Fig. 13. As can be seen, the tensile strength, elongation and impact energy of ADI with tempering were improved to exceed the ADI standard and to reach the maximum of those treated with austempering only. When the mechanical properties of specimens tempered at 473 K after austempering at 648 K were compared with the data of 0.37%Mn ductile iron treated with 648 and 593 K stepped austempering. The tensile strength and toughness of ADI with tempering reached the same level as those of specimen treated with stepped austempering. The required austempering time for stepped austempering was in the range of 9–264 ks while that for tempering after austempering used in this paper was only 2.1 ks. This again implies that tempering at 473 K after austempering could strengthen the mechanical properties of ADI and could obtain higher tensile strength and toughness compared with those of ADI treated with austempering only. Tempering at 473 K could also extend the processing window of ADI and could obtain the maximum values of mechanical properties with a shorter austempering time. Compared with the conventional austempering treatment, this new process has the advantages of less sensitivity to austempering conditions and greater ease in successful production of

ADI with maximum values of mechanical properties. For increase in mechanical properties, the effectiveness of tempering after austempering was the same as that of stepped austempering, but the required austempering time for obtaining the maximum values of mechanical properties could be shortened significantly.

IV. Conclusions

A ductile iron was tempered at 473 K for 7.2 ks after austempering to obtain a microstructure mixture of ausferrite and tempered martensite. The change in microstructure and the related changes in mechanical properties have been investigated. The conclusions obtained are as follows.

(1) Before stage 1 reaction of austempering was completed, martensite formed in the matrix of ADI on quenching to room temperature. The size of martensite platelets increased with increasing austempering temperature. The reducing rate of martensite content decreased as the austempering temperature increased.

(2) The existence of martensite in ADI had a significant influence on its mechanical properties. As martensite content increased, ductility and toughness decreased. This decrease was more obvious in upper ADI.

(3) After tempering at 473 K, the unfavorable effect of martensite could be eliminated and the strength and toughness could be improved to exceed the maximum of those of ADI treated with austempering only. This new process could also shorten the austempering time for obtaining the maximum values of mechanical properties, extending the effective range of austempering time. So the sensitivity of mechanical properties to austempering conditions was improved to increase the reliability of production.

(4) The increase in mechanical properties after tempering at 473 K increased with increasing martensite content. These increases were independent of the morphology of ausferrite and austempering temperature. The amount of tempered martensite with evident

strengthening effects for austempering at 573 K was in the range of 1–5%, while that at 598, 623 and 648 K fell within 1–3%.

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