Austenitization and formation of ausferrite structure in austempered ductile iron with dual matrix

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Abstract

The effect of austenitizing conditions on the microstructure and mechanical properties of an austempered ductile iron (ADI) with a dual matrix was investigated. Carbon diffusion plays a critical role in the phase transformation of austenitization. In initially pearlitic structures, the carbon diffusion distances involved during austenitization are smaller compared to those in ferritic structures. The study was carried out to examine the influence of temperature and times of the austenitization process on the maximum carbon content in austenite and then its effect on the processing window of ADI with the dual matrix. An alloyed ductile iron (3.6%C; 2.44%Si; 0.36%Mn; 0.9%Ni; 0.61%Cu; 0.11%Cr; 0.036%Mg; 0.015%S và 0.006%P) was fully austenitic at various temperatures 870 °C–930 °C. The minimum hardness reaches the value of 270 HB. An increase in austenitization temperature increases the carbon content dissolved in the austenite, which in turn, decreases the free energy controlling the transformation of austenite to bainite ferrite and high carbon austenite. Raising the austenitization temperature makes the process window shift toward the extending time. The carbon atoms must diffuse out of the ferrite needle with a diffusion distance equal to the ferrite layer thickness. At 360 °C austempered temperature, diffusion coefficient $D = 4.60.10^{-17}(m^2/s)$. With a ferrite needle thickness of $1/\mu m = 10^{-6}m$, the diffusion time would be $t = 5330(s)$.

1. Introduction

Austempered ductile iron with dual matrix (Dual matrix ADI) is a ductile iron that has higher ductility but lower strength than traditional ADI. This iron is produced by heat treatment to create a structure consisting of the pro-eutectoid ferrite, ausferrite, and possibly little martensite. The initial iron has the pearlite + ferrite structure which is austenite in the $\gamma$ region, then annealed in the three-phase region where both ferrite, austenite, and carbon coexist. In this region, austenite will precipitate pro-eutectoid ferrite and graphite. The proportion of the pro-eutectoid ferrite phase depends on the chemical composition and the heating temperature [1–5]. The iron is then austempered at a temperature less than the A1 line in the Fe–C diagram. In the period I reaction, from austenite, the needle ferrite (AF) and high carbon austenite are precipitated ($\gamma_{HC}$: $\gamma \rightarrow AF + \gamma_{HC}$). The microstructure consists of ferrite needles on a high carbon austenite matrix called ausferrite. If keep soaking, period II reaction occurs, bainite and carbides will be precipitated from high carbon austenite: $\gamma_{HC} \rightarrow FB + Xe$. The time between the end of a period I reaction and the start of a period II reaction, there is a pause, without any reaction. This period of time is called the processing window.

The austenitic transformation is a diffusion-controlled transformation and also a restructuring transformation. In this transformation, the diffusion of carbon determines the transition kinetics. Because it is also considered a transposition, the nucleation process is a key step in forming the structure of iron [4, 6–9].


The high content of C in iron and steel will promote the transformation rate of pearlite to austenite because increasing carbon content increases the carbide particles, the length of the ferrite/cementite boundary increases, the distance between the cementite plates decreases, the diffusion distance is reduced, resulting in an increased rate of transformation.

High-temperature austenitization will increase the carbon content of austenite. The high carbon content in austenite reduces the free energy due to control the transformation, the final structure will be coarser austenitic grains, with more bulk austenitic grains. The coarser the austenitic grain, the less carbon it contains and the less stable it becomes, easily transformed to martensite as cooling continues. This is a bad effect on the impact strength of the iron [10–12].

Thus, an increase in the carbon content will increase its diffusion rate in austenite. This is due to two reasons: (a) due to a very large displacement of the austenitic lattice and (b) due to increased thermodynamic reactivity.

The diffusion depth of carbon in austenite mainly depends on temperature. The diffusion coefficient of carbon increases with increasing temperature. The change of carbon content in austenitic depends on the carbon concentration on the phasing surface and the carbon content in the metal matrix and the depth of diffusion. The carbon current diffused along the grain boundary is proportional to the concentration gradient [13–15].

Reduce the austenitic temperature, and the carbon content in the austenite decreases. Reducing the carbon content will also decrease the starting temperature of martensite Ms transformation [16]. Reducing the temperature of austenitization causes the rate of the ausferrite reaction to increase [17], the ferrite needles become finer and more homogenous distribution, and the volume fraction of residual austenite decreases. Reducing the temperature of austenitization also reduces the austenite grain size, the grain boundaries will increase, because the ferrite will have nucleation on the austenite grain boundaries, so if the area of the austenitic grain increases, it promotes nucleation of the ferrite needles, the ferrite needles will be finer and the ausferrite transformation rate will be higher. Austempering at a temperature of 250 °C for low-carbon globular cast iron, the resulting structure consists of martensite needles mixed with needle-ferrite [17–19].

This paper focuses on studying the austenitization in manufacturing ADI with a dual matrix, and the effect of austenitic temperature on the structure and window area of ADI with the dual matrix.

### 2. Materials and methods

#### 2.1. Materials

The composition of gray cast iron was heated in a medium frequency electric furnace, denatured by the intermediate alloy VE08–099, using 2% by weight of liquid iron. Denatured temperature 1480 °C in 5 min; denatured graphite by 0.4% Fe-Si75. The Y-shaped mold according to ASTM A439 is cast in a sand mold (table 1).

#### 2.2. Experimental

The test sample is cut from the lower part of the Y-shaped sample. Heating the sample to a fully austenitic temperature of 870 °C; 900 °C and 930 °C for up to 120 min, then water-quenched. Determination of the C content in martensite (the C content in austenite at high temperature).

To study the effects of the austenitic temperature and carbon content on the pro-eutectoid ferrite and ausferrite transformation, heat the sample at 870 °C; 900 °C, and 930 °C, soaking for 2 h. Quickly transfer the sample to the annealing furnace at 780 °C for 1.5 h then convert to austempering in the salt bath: 50% NaNO₃ + 50% KNO₃, the temperature is 280 °C; 320 °C; 360 °C, and 400 °C in various minutes.

After quenching, all austenite will be transformed to martensite, and the amount of ferrite does not change. Determine the carbon content in the martensite, and at the same time determine the portion of the pro-eutectoid ferrite and austenite (which is the proportion of martensite after quenching in water) according to the color metallography principle. This color proportion on optical microscopes: After etching, the ferrite phase will have a light color, the martensite phase is a dark color phase. The software will detect these two phases based on color.

The carbon content in austenite was determined through three methods: Energy dispersive spectrometry EDS; calculated according to the Thermolcal phase diagram and empirical formula [20]:

| %C  | %Si | %Mn | %Ni | %Cu | %Cr | %Mg | %S | %P | %Fe |
|-----|-----|-----|-----|-----|-----|-----|----|----|-----|
| 3.6 | 2.44| 0.36| 0.90| 0.61| 0.11| 0.036| 0.015| 0.006| bal |

Table 1. The composition of cast iron.
The process window is determined by the thermal expansion curve of the sample. End of the period I reaction: the curve begins to be horizontal. Period II reaction initiation: there is a spike on the curve. The microstructure of samples was analyzed on Axiovert 25A optical microscope and SEM.

3. Results and discussion

3.1. Microstructure after casting

The shape, distribution, as well as metal matrix structural composition of the iron, is shown in figure 1. Graphite structure is distributed homogenously on the metal matrix, the degree of graphite fusion is 90%. The metallic matrix consists of 85% pearlite and 15% ferrite, with no carbides on grain boundaries.

The average distance between two graphite grains is 178 μm. The thickness of the ferrite layer around the graphite grain is about 20.5 μm. Thus, in the austenitization process, the carbon atoms have to cross a distance of 20.5 μm. This distance is short enough for atoms to diffuse so the diffusion time will be shorter.

3.2. Austenitization

When austenitization temperature changes, the saturated carbon content of austenite changes according to the $A_{cm}$ line on the Fe–C phase diagram. The carbon contents determined by using 3 methods are shown in figure 2. Carbon content at three temperatures 870 °C, 900 °C, and 930 °C at different times is shown in figure 3.

\[
%C = -0.345 + 0.33510^{-3} * T_1 + 1.6110^{-6} * T_1^2 + 0.006 *%Mn - 0.11 *%Si
\]  

(1)

Figure 1. The as-cast microstructure of spheroidal iron.
The carbon content in austenite increases with the austenitization temperature (figure 2). The dissolved carbon content in austenite is determined by three methods: empirical formula, calculation according to the Thermocal diagram, and EDS. There is no significant difference between them. Dissolved carbon contents in austenite measured by EDS are lower than that measured by formula and phase diagram. This may refer to that, the phase diagram is made under an extremely slow cooling rate and controlled under certain conditions, so it is considered an ideal condition. On the other hand, during three-phase annealing, carbon is precipitated from austenite, which in the experimental formula and the Thermocal diagram is ignored, therefore, the value of the carbon content determined according to EDS has the lowest value.

At the same temperature, increasing the holding time, the carbon content increases gradually and reaches a saturation value, then remains a constant value. Increasing the austenitization temperature, the degree of carbon saturation in austenite also increases and reaches the maximum value of 1.05%. The austenitization temperature is high, and the dissolution rate of carbon in the iron increases, it’s shown by the slope of the concentration-time curve (figure 3). Raising the austenitic temperature, the driving force of carbon in the diffusion process increases, and thus, the diffusion coefficient of carbon in Fe increases, the dissolution rate of carbon also increases, and the amount of Carbon quickly reaches its maximum value.

Austenitization at a temperature of 930 °C, after only 45–50 min, the carbon content has reached the saturation value, at 900 °C, after 100 min, the carbon content will reach saturation value. In ferrite + perlite iron, the carbon sources for austenitization are spherical graphite and carbides in pearlite. With ferrite cast iron, the carbon source to saturate austenite is only carbon from graphite. If the distance between the graphite grains is larger, the carbon diffusion distance from graphite to the metal matrix will be longer, therefore, the time to reach the carbon saturation state in austenite increases. In pearlite cast iron, the dissolution and diffusion of
carbon from the carbide plates in pearlite occur at a shorter distance, so the carbon saturation in austenite occurs more rapidly.

Austenitization in cast iron is a complex process and involves two stages, (i) austenitization of the ferrite shells around the graphite particles and (ii) austenitization of pearlite grains. In the (i) stage the diffusion of carbon from the graphite particle to the ferrite shell is major, the (ii) stage involves the decomposition of cementite to Fe and carbon, then carbon diffuses from the cementite lath to the adjacent ferrite plate. Austenitization of the shells around the graphite particles also consists of two stages. In the initial stage, when the carbon content is still low, diffusion of carbon in Fe-α is main. When the carbon content is high, the diffusion of carbon in Fe-γ will be major. Diffusion coefficient D of carbon in Fe-α and in Fe-γ is \( D = 5.7 \times 10^{-16} \, \text{m}^2 \, \text{s}^{-1} \) and \( D = 3.19 \times 10^{-12} \, \text{m}^2 \, \text{s}^{-1} \) respectively.

Assuming that, when austenitization, carbon atoms diffuse from the graphite particle to the adjacent ferrite shell, raising the carbon content to a saturation value. Carbon diffusion satisfies Fick II’s law. The solution of the Fick II equation:

\[
\frac{C(x,t)}{C_m - C_0} = 1 - \text{erf} \left( \frac{x}{2\sqrt{D}t} \right)
\]  

In which: \( C_o \)—initial concentration of carbon (%); \( C_m \)—the upper carbon concentration (%); \( x \)—diffusion distance (m), \( D \)—temperature-dependent diffusion coefficient (m²/s), \( t \)—diffusion time, (s).

Diffusion coefficient D of carbon in Fe - γ depends on temperature according to the formula:

\[
D = 1.23 \times 10^{-6} \exp \left( -\frac{1050}{T} \right) \, \text{cm}^2 / \text{s}
\]  

When the diffusion is stable, the diffusion length will reach

\[
x = 2\sqrt{D}t
\]  

Calculating the diffusion coefficient D at 900 °C by (3) expression; substituting D value in (4) formula with \( t = 7200 \, s \), diffusion length is 262 \( \mu \)m. It’s longer than the distance between two graphite grains (187 \( \mu \)m) of this work. It proves that the austenitization time of 2 h (7200s) is long enough to ensure that the carbon atoms diffuse throughout the length between the two graphite grains. This result is illustrated in figure 3, on the no.2 line.

When austenitization at 900 °C, after about 100 min, the concentration of carbon in the sample has reached a saturation state (0.91%). In table 2, the diffusion distance is calculated at different temperatures.

Consider the austenitization stage of pearlite sheaves, on the ferrite/cementite boundary, carbon content is 6.67%, and initial carbon content in the ferrite (\( C_{\alpha} \)) is 0.008%. The dimensionless concentration of carbon is defined as:

\[
C = \frac{C_{\alpha,t} - C_0}{C_{\text{max}} - C_0}
\]  

The volume of austenite is \( V_1 \); the volume of perlite is \( V_2 \); the volume of the sample is \( V \); \( V_1 + V_2 = V \)

\[
\frac{V_1}{V} + \frac{V_2}{V} = 1 \iff f + f_2 = 1
\]

The Austenite fraction in the austenitization transformation is f. When time \( t = 0 \); there is no austenite, austenite fraction \( f = 0 \). In formula (5), when \( t = 0 \), the concentration \( C_{\alpha,t} = C_{\alpha} \) the coefficient \( C = 0 \).

When the time reaches the critical value, the austenitization transformation occurs completely, the austenite volume fraction \( f = 100\% = 1 \). In formula (5), carbon content reaches \( C_{\text{max}} \) coefficient \( F = 1 \).

Thus, it is possible to refer to the austenite volume fraction (f) as the ratio of change of carbon content in austenite (C).

On the other hand, the relationship between austenite volume fraction f, temperature, and transformation energy Q can be illustrated [20]:

| T (°C) | Diffusion coefficient \( D \), \( \text{m}^2/\text{s} \) | Diffusion time, \( s \) | Diffusion distance, \( m \) |
|--------|---------------------------------|------------------|------------------|
| 870    | 7.17E-13                        | 7200             | 0.000221         |
| 900    | 7.26E-13                        | 7200             | 0.000262         |
| 930    | 7.35E-13                        | 7200             | 0.000307         |

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\[
\frac{df}{dt} = B\exp\left(-\frac{Q}{RT}\right)
\]

Where \(B\) is a constant; \(Q\) is the activation energy of the transformation; \(R\) is the gas constant; \(T\) is the temperature K.

Approximately:

\[
\frac{\Delta C}{\Delta t} \approx \frac{df}{dt} = B\exp\left(-\frac{Q}{RT}\right)
\]

Changing equation (7) by taking the logarithm of the two sides:

\[
\ln \left(\frac{\Delta C}{\Delta t}\right) = -\frac{Q}{R} \frac{1}{T} + \ln B
\]

The volume fraction and temperature data could be presented in a \(y = ax + b\) format in which \(y = \ln \left(\frac{\Delta C}{\Delta t}\right)\) and \(x = \left(\frac{1}{T}\right)\). The slope of the curve will be the \(-\frac{Q}{R}\) value. Assuming that in the first 30 min in figure 3, when the carbon content is low, the diffusion of the process is the diffusion of carbon in Fe-\(\alpha\). At each temperature, calculate the value of \(\frac{\Delta C}{\Delta t}\) with respect to the values \(\Delta t\). The relationship between \(\frac{\Delta C}{\Delta t}\) and \(\frac{1}{T}\) is illustrated in figure 4.

Finally, the simulation equation will be:

\[
y = -19097x + 11.91
\]

Therefore:

\[
-\frac{Q}{R} = -19097
\]

\[
Q = 19097 \times 8.3 = 158500(J)
\]

The Q activation energy of the austenitization transformation of this work is not much different than the result [20] which is 158000 (J). Using the formula for calculating the coefficient of carbon in Fe-\(\gamma\):

\[
D = 1.23 \times 10^{-6} \cdot e^{-\frac{15050}{T}} \left(\frac{m^2}{s}\right)
\]

At \(T = 900^\circ C = 1133K\), calculate: \(D = 3.29 \times 10^{-12}(m^2/s)\)

This data is quite consistent with many published works [9, 16, 21–23].

### 3.3. Hardness of ADI with dual matrix

The hardness of ADI with dual matrix depends on the austenitization temperature as shown in figure 5. High austenitization temperatures will increase the hardness of the cast iron. The hardness of cast iron is strongly related to the solubility of C in the cast iron matrix. Increasing the austenitic temperature, the carbon content in the austenite increases, making the austenite more stable. On the other hand, when the austenitization temperature is high, there will be less pro-eutectoid ferrite precipitated when annealing in the three-phase area. As a result, the hardness of ADI with dual matrix increases.

In figure 6, When the austenitization temperature is constant, extending the austempering time, the hardness of the ADI after quenching evenly passes through a minimum point. The austempering time affects the proportion of phases contained in the microstructure. Initially, the austempering time increases, and the proportion of the martensite gradually decreases. When transformation approaches the area near the process window, martensite composition is approximately zero. Passing through the process window, stage II reaction
occurs, the carbide phase is precipitated, and the hardness will increase. Increasing the austenitization temperature, the extreme point will shift towards a longer time and ADI has a higher value of hardness. This can be explained that the higher the austenitization temperature, the higher the carbon content in austenite, and the driving force of the process \( Q \) decreases more with \([16, 22, 24, 25]\), so the stage I reaction ends more slowly. The experiment showed that at a temperature of 870 °C, it was nearly 90 min led to an extreme hardness of 230HB; at 930 °C, it took nearly 150 min to reach the maximum value of 257HB.

### 3.4. The effect of austenitic temperature on ausferrite transformation

Decrease in the austenitization temperature, the carbon content in the austenite decreases (figure 3), increasing the temperature Ms of the martensitic transformation. Reducing the austenitic temperature increases the transformation rate of the stage I reaction by relationship (5), the ferrite needles become finer and their distribution is more uniform, and the volume fraction of residual austenite will decrease. Thus, by reducing the austenitization temperature, the system quickly reaches an equilibrium state, the stage I reaction ends sooner, and the starting point of the process window will be shifted towards a shorter time (figure 7). On the other hand, the austenitization temperature decreased, the austenite grain size also decreases, and the grain boundaries increase. Because ferrite nucleated on the austenitic grain boundaries, increasing the grain boundary means promoting the formation of ferrite needles and making the ferrite needles become finer, the ausferrite transformation rate is also faster. Thus, low austenitization temperature will make the process window shift toward a shorter time (figure 7).

On the other hand, the austenitic temperature decreased, the austenitic grain size also decreased, and the grain boundaries increased. Because ferrite is nucleated on the austenitic grain boundaries, increasing the grain boundary means promoting the formation of ferrite needles and making the ferrite needles become finer, the transformation rate of the ausferrite reaction is also faster. Thus, the low austenitic temperature will make the transition window in a shorter time (figure 7).

When austenitization at a temperature of 930 °C, the diameter of the austenite grain is about 28 μm (figure 1). As austempering, the nucleation of ferrite needles occurs on the austenite grain boundaries. The
The distance between ferrite needles is very small, only about 1 μm. In this case, the carbon atoms must diffuse out of the ferrite needle with a diffusion distance equal to the ferrite lath thickness. Assume that the diffusion coefficient $D$ is shown in the formula (9). At 360 °C austempering, diffusion coefficient $D = 4.60 \times 10^{-17} \text{m}^2/\text{s}$.
The diffusion distance after the time \( t \) will be: 
\[ xD t^2 = \frac{x^2}{4D} = 5330 \, \text{s} \]

For example, with a ferrite needle thickness of \( 1 \, \mu\text{m} = 10^{-6} \, \text{m} \), the diffusion time would be:
\[ t = \frac{x^2}{4D} = 5330 \, \text{s} \]

Thus, a ferrite needle is nucleated and grows completely, it takes 5500s, approximately 90 min. In fact, the process of formation and growth of ferrite needles is complicated and competitive, so the actual diffusion time is slightly larger than this value, as shown in figures 8 and 9.

4. Conclusion

1. Raising the austenitization temperature, the driving force of carbon in the diffusion process increases, and thus, the diffusion coefficient of carbon in Fe increases, the dissolution rate of carbon also increases, and the carbon content quickly reaches its maximum value. At an austenitization temperature of 900 °C, it takes only 90 min for the content of carbon in the saturated austenite to reach 0.95%.

2. The carbon diffusion distances during solution treatment may be relatively large. Completely austenitization requires either very long solution treatment cycles or a very high carbon diffusion rate. The calculated diffusion length at 900 °C is 262 μm. It’s longer than the distance between two graphite particles (187 μm) of this work. It proves that the austenitization time of 2 h (7200s) is long enough to ensure that the carbon atoms diffuse throughout the length between the two graphite particles.

3. The curves of the hardness of ADI with dual matrix versus the tempering time have the minimum value. Increasing the austenitic temperature, the extreme point will shift towards a longer time and the absolute value of hardness is higher. At austenitization temperature of 930 °C and after 150-min austempering time, the minimum hardness reaches the value of 270 HB.

4. An increase in austenitization temperature increases the carbon content dissolved in the austenite, which in turn, decreases the free energy controlling the transformation of austenite to bainite ferrite and high carbon austenite. Raising the austenitization temperature makes the process window shift toward the extending time.

5. The calculations of the times required for the diffusion of carbon out of supersaturated ferrite laths to form the austerity structure indicated that there is a necessity for carbide precipitation from ferrite. The thickness of ferrite needles is very small, only about 1 μm. The carbon atoms must diffuse out of the ferrite needle with a diffusion distance equal to the ferrite layer thickness. At 360 °C austempered temperature, diffusion coefficient \( D = 4.60 \times 10^{-17} \, (\text{m}^2/\text{s}) \). With a ferrite needle thickness of \( 1 \, \mu\text{m} = 10^{-6} \, \text{m} \), the diffusion time would be: \( t = \frac{x^2}{4D} = 5330 \, \text{s} = 1.48 \, \text{h} \).

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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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