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The effect of first step holding time of low high austempering heat treatment to the mechanical properties of Austempered Ductile Iron (ADI)

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Abstract. A low-high austempering process can be referred to as the application of undercooling condition in the austempering process of Austempered Ductile Iron (ADI). Undercooling condition promote more grain nucleation and producing finer grains with good mechanical properties. This study aims to investigate the influence of the first stage of lower temperature austempering holding time on its mechanical properties and find the best holding time which can produce the highest toughness of ADI materials. The experiment began with the heating of the nodular cast iron sample up to 927 °C and held for 120 minutes. Then it quenches to a liquid salt bath with a temperature of 260 °C as a lower temperature of the first austempering stage with a variation of holding time at 30, 60 and 90 minutes. Then it was transferred to a second salt bath medium of 400 °C as the second austempering stage with a holding time of 120 minutes then it was given the air cooling. The sample was tested by hardness, tensile, impact, and metallographic examination. The results show that the best toughness value in this research is obtained at 30 minutes holding time on first austempering stage with mechanical properties is tensile strength 1273 MPa, yield strength 1162 MPa, elongation 4,87% and impact value at 54.15 J. Value modulus of toughness is 5.93×10^7 J/m^3.

1. Introduction

Austempered Ductile Iron (ADI) has long been used as an engineering material. If SG Iron is given an austenitization process and then followed by an austempering process, it will produce a mixed microstructure consisting of ausferrite, ferrite, and austenite with graphite nodules dispersed in it. This unique microstructure produces mechanical properties in ADI equivalent to steel forging [1]. ADI also has excellent fatigue strength, [2] high fracture toughness [3], and excellent wear resistance [4]. The interesting properties of ADI are related to its unique microstructure consisting of ferrite (α) and high carbon austenite (γHC). The ADI microstructure is different from forged steel where the bainite microstructure of steel consists of ferrite and carbide. Because of these differences, austempering products in SG iron are often referred to as ausferrite rather than bainite. The ausferrite is free from carbide which makes it has better toughness than bainite structure. The substantial amount of silicon present in SG iron suppresses carbide formation during the austempering reaction and retains large amounts of stable high carbon austenite (γHC) [5–7].

1.1. Conventional Austempering Process

ADI's conventional austempering process can be implemented first with the SG Iron austenitizing process to a temperature of 871-927 ^{o}C and held for 2 hours; then SG Iron is given a quench process to temperatures between 260 and 400 ^{o}C. The SG Iron is then isothermally held at this temperature for about 2 hours and finally cooled by air. During the austempering process, a phase transformation reaction occurs in two stages [8,9]. In the first stage, austenite (γ) decomposes into ferrite (α) and high carbon austenite (γHC):

$$\gamma \rightarrow \alpha + \gamma_{HC} \quad (1)$$

If the casting is held at austenitizing temperature for too long, then a second reaction occurs, where the high carbon austenite (γHC) further decomposes into ferrite (α) and carbide (ε)

$$(\gamma_{HC}) \rightarrow \alpha + \varepsilon \quad (2)$$

This excessive reaction is undesirable because it produces brittleness in ADI because the presence of carbides can eliminate the ductility and toughness. Therefore, for the successful production of ADI, SG Iron must be processed austempering in the bainite transformation region before the end of the bainite transformation.

The other problem that some alloying element such as Mn and Mo make the austempering reaction becomes slow, and as a result, some unreacted austenite remains, which may turn into martensite during the final cooling process. This unreacted austenite leaves a continuous film layer in martensite in the intercellular region and consequently can trigger crack propagation through this region, and this may result in poor ductility and lower fracture toughness in ADI [10].

1.2. Two Step Austempering

Due to the limitation of the austempering results above and to obtain a better combination of tensile strength and toughness, the two-step austempering process has been proposed by several researchers [11–15]. The process which is used in this research is the step-up austempering method. In this process (often called a low-high austempering), SG iron, after austenitizing, is given the quench process to a lower austempering temperature and held there for a predetermined period. The second austempering step carried out at higher temperatures either by raising the first salt bath temperature or transferring the casting to another salt bath which is maintained at a higher temperature than the first salt bath. The sample is austempered at this second temperature for the period needed and finally cooled in air. The austempering step-up process scheme is shown in figure 1.

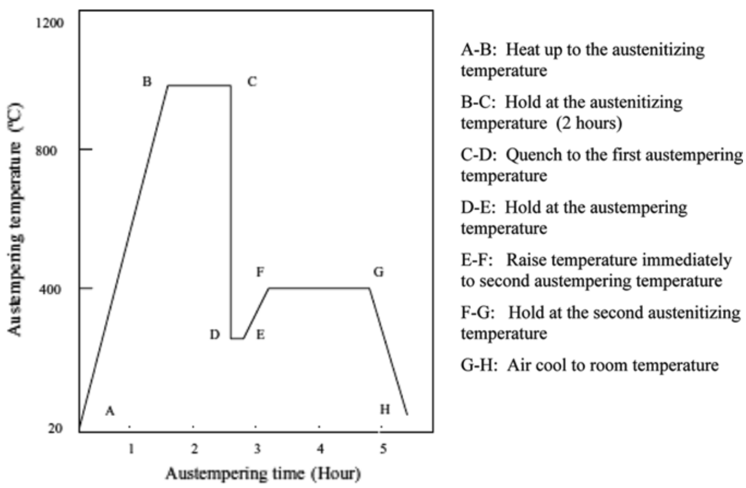


Figure 1. Step-up austempering process [10].

Many researchers for ADI material development have applied the step-up austempering process methods. It was found that the step-up austempering process has a significant improvement in the properties of ADI due to improved ferrite properties and higher austenitic carbon content in ADI avoiding the formation of iron carbide bracket structures [16,17]. The step-up austempering process produces a combination of lower and upper ausferrite. The first step of the austempering process is carried out at temperatures above the initial temperatures of martensite (MS) to 330 °C resulting in lower ausferrite, SG Iron has a high strength/hardness but low toughness, while in the second step of austempering holding time is done at 330 °C to 425 °C produce upper ausferrite with opposite behavior. [18,19]. A low-high austempering process can also be referred to as the application of undercooling condition in the austempering process of Austempered Ductile Iron (ADI) [20]. With undercooling, more grain nucleation can be promoted finer grains which are producing good mechanical properties.
This study aims to investigate the influence of holding time of the first (lower) temperature of the low–high austempering process to the mechanical properties of ADI. How long is the best time for the first austempering process which give the best combination of lower and upper ausferrite structure and also give the finer structure. It was expected to find the good toughness of ADI materials among the variable used.

2. Methodology

2.1. Material

The sample for the experiment was made from SG Iron material which was cast in the form of keel Block using ASTM A897 standard with a thickness of 13-38 mm. Keel block castings are cut in a square shape with a size of 22x15x15 mm for hardness test and microstructure examination, 13 x 13 x 58 mm for impact test and cylinders with dia. 10 x 50 mm for the tensile test. The number of samples each is arranged to the variations needed. The chemical composition of SG Iron as-cast material is verified using optical emission spectroscopy and the results as shown in the following table 1:

Tabel 1. Chemical composition of SG iron as-cast.

C	Si	Mn	P	S	Mg	Cu
3.300	3.550	0.260	0.010	0.004	0.047	0.850

2.2. Heat Treatment Process

The heat treatment process was carried out by heating the material to the austenitizing temperature at 927 °C with holding time for 120 minute, then continued with quenching to the first salt bath medium with a temperature of 260 °C with a holding time variation of 30 minute, 60 minute and 90 minute. Then each sample was rapidly transferred to a second salt bath medium which has a higher temperature of 400 °C with a holding time of 120 minute after the hold time is reached then the sample is taken to be air cooled. Figure 2 shows a graph of the heat treatment experiment on the sample which is arranged in this study:

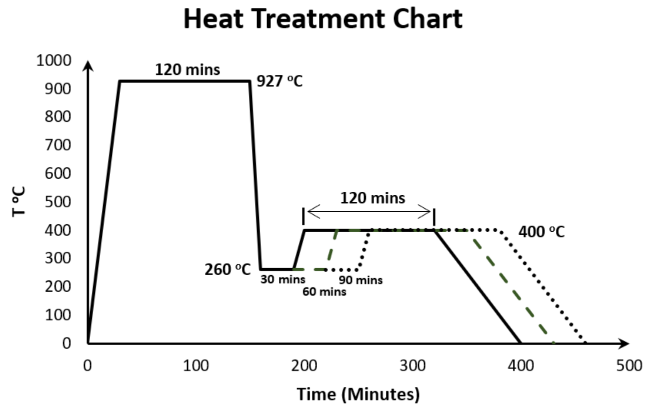


Figure 2. Heat treatment cycle.

2.3. Salt Bath

A salt bath is used for the austempering process. Liquid salt temperature is determined according to the required temperature. Two units of the salt bath are used. The first salt bath for the first austempering process uses salt with a composition of 50% $NaNO_2$ + 50% $NaNO_3$. This salt bath is maintained at 260 °C. The second salt bath is used for austempering the second uses salt with a composition of 95% $NaNO_3$ + 5% NaCl and the temperature set is 400 °C.

2.4. Mechanical Test and Metallographic Examination

All sample variations are tested by tensile testing using a universal tensile testing machine which refers to ASTM E-8M standard and hardness test with Rockwell C with ASTM E-18 standard. The impact test was also carried out with Charpy impact machine and refer to ASTM E-23 standards with unnotches Charpy sample specifications. The metallographic examination is also carried out to analyze the microstructure resulting from each process. Metallographic samples are etched using 3% Nital and observations are done using optical microscopy. To ensure repeatability of the process, each variation was carried out on three samples given similar heat treatment. Thus the average test results from the three samples were obtained.

3. Result and Discussion

3.1. Microstructures

The SG Iron as-cast material has a microstructure with the majority of Pearlite (90%) with a small amount of ferrite and spheroidal graphite. Figure 3 shows the microstructure of SG Iron as-cast material.

Metallographic examination of low-high austempering results are shown in figure 4 (a), (b) and (c). The matrix of microstructure consists of lower ausferrite, upper ausferrite, ferrite needles, and light-colored austenite with dispersed globular graphite. The first austempering process is producing lower ausferrite. The ausferrite is consist of ferrite and austenite. Ferrite needles are getting more and more and also look finer or look closer and sharp with longer of holding time on the first austempering. This shows that the longer austempering holding time at 260 °C result in the amount of lower ausferrite much more. The two-step austempering process generally produces finer ferrite [20] which increase the strength and hardness of the material, where the low-high process produces greater supercooling for ferrite nucleation which is increasing and can produce finer ferrite.

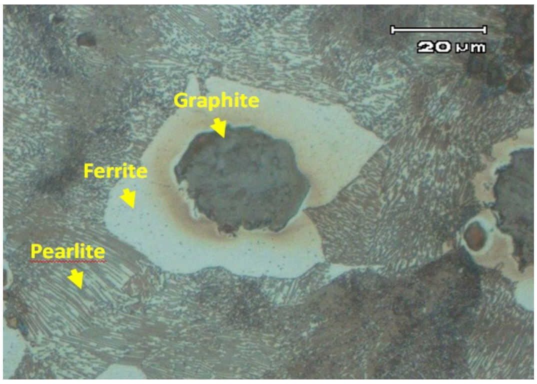
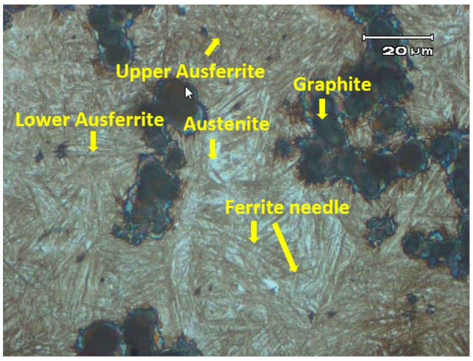
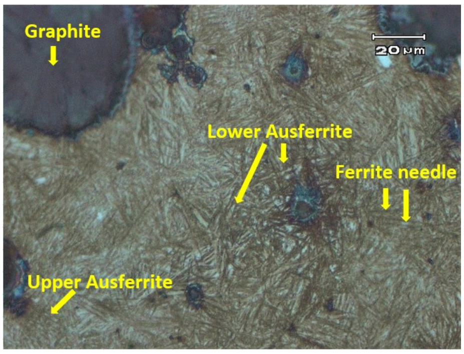


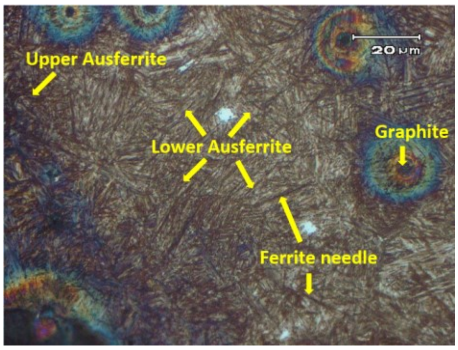
Figure 3. The microstructure of SG iron as-cast, nital 3% etchant.



(a)



(b)

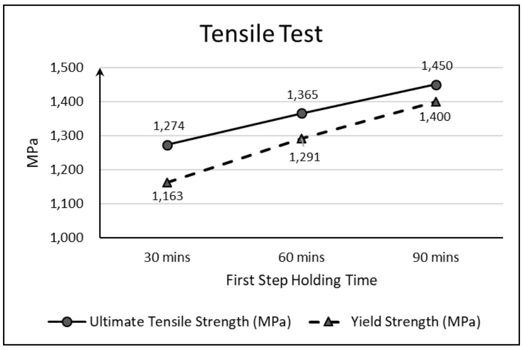


(c)

Figure 4. Microstructure heat-treated sample (a) first austempering holding 30 minute (b) first austempering holding 60 minute (c) first austempering holding 90 minute, nital 3% Etchant.

3.2. Mechanical Properties

The tensile test results are shown in figure 5 Shows that the longer holding time at first austempering the tensile strength and yield strength increases. At holding time 30 minute obtained tensile strength 1,274 MPa and yield strength 1,163 MPa, elongation of 4,87%. Whereas for the third sample for holding austempering time for 90 minute obtained tensile strength 1,450 Mpa increased by 13.8% compared to the first sample, the yield strength of 1,291 Mpa increased by 11%. Elongation for holding time 90 minute decreases to 1.22%. The increase in tensile strength and yield strength is related to the formation of lower ausferrite which has higher tensile strength and the finer form of ferrite needles. The finer form of microstructure influence the dislocation movement becomes more difficult as formulated by Hall-Petch where the finer the grain will result in higher tensile strength and yield strength.



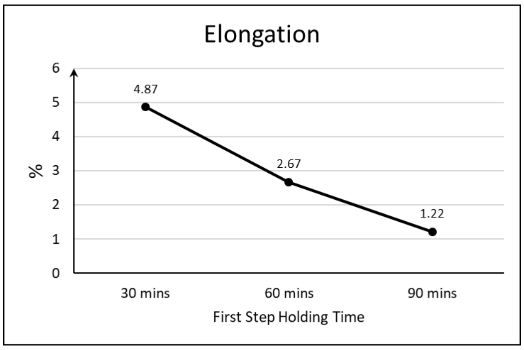


Figure 5. Tensile test result of all specimens.

From the tensile test results, the modulus of toughness for the tensile test can be calculated. The calculation of the area under the tensile test curve can be approximated by using the formula:

$$U_T = \frac{So+Su}{2} . \varepsilon f \quad \text{, where So: Yield Strength, Su: UTS and } \varepsilon f\text{: elongation}$$

The result showed that the longer holding time is given at the first austempering, then the value of the modulus of toughness is decreased. The highest value of modulus of toughness is shown by the first sample that is 5.93×10^7 J/m^3. This modulus of toughness value is correlated with the impact test result. Figure 6 shows the results of impact testing. In the graph, it shows that the highest impact energy is produced by the first sample with a value of 54 J and decreases with increasing holding time on the first austempering.

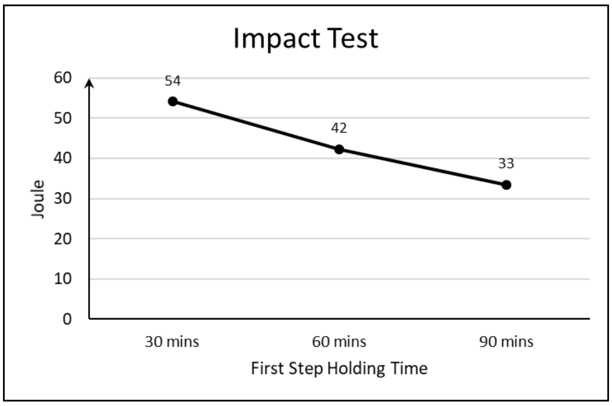


Figure 6. Impact test result of all specimens.

Hardness testing was carried out on the sample, and the results can be seen in figure 7 it shows that the hardness of the holding time at 30 minute is 38 HRC and increases to 40 HRC at the holding time of 90 minute.

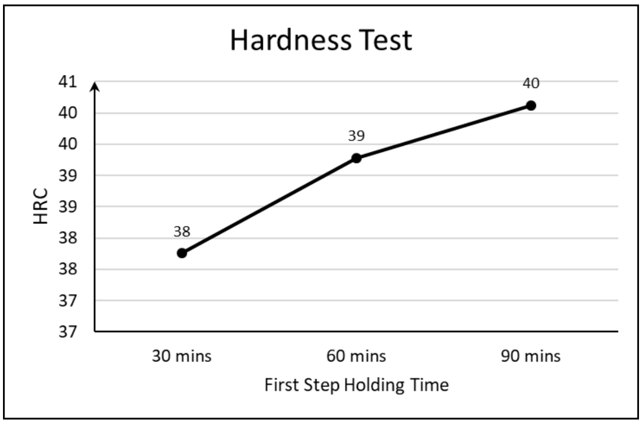


Figure 7. Hardness rockwell C test of all specimen.

The increase in hardness value followed by a decrease in the value of elongation is related to the holding time condition that is too long. If the sample is held at austenitizing temperature for too long, then it is possible to a second reaction takes place; where is the high carbon austenite (γHC) can further decompose into ferrite (α) and carbide (ε) [10]. Thus, if the desired material is obtained that has good ductility, the ADI material must be austempered in the process window. The process window is defined as the time interval between the completion of the first reaction and onset of the second reaction (figure 8). Thus the formation of carbides can be avoided.

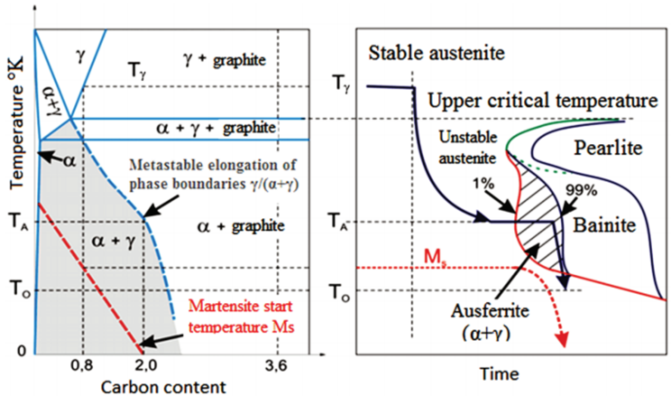


Figure 8. Process window of ADI [21].

4. Conclusion

The experimental results done in this study can be concluded that the longer holding time on first austempering affects ADI's material with increasing of tensile strength, yield strength, and hardness. But on the other hand elongation and impact values decrease. Low-high austempering can produce finer ferrite needle structures that contribute to the increase in ADI material's tensile strength and yield strength. This is also related to the formation of a combination of lower and upper ausferrite structures. The longer the holding time is given at, the lower temperature then, the lower ausferrite is formed more, this can increase the material brittleness. It is also analyses that the formation of carbide (ε) can be possible occurs due to long of holding time which can exceed of the process window in the ADI material (Figure 8).

As the combination of tensile and elongation then the toughness can be calculated to find the best value among the experimental variable. The highest value of modulus of toughness is shown by the holding time of 30 minutes that is 5.93×10^{7} J/m^{3}. To obtain tougher material, the total holding time at austempering process must not be too long, and it is better not exceed the process window in the ADI material. Thus the formation of a brittle carbide structure can be avoided.

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