Effect of austempering treatment time on strength, ductility, hardness, and abrasive wear resistance of JIS FCD 700 ductile iron

H Rinaldo, M Januar*, M M Sinaga
Department of Mechanical Engineering, Atma Jaya Catholic University of Indonesia
E-mail: moch.januar@atmajaya.ac.id

Abstract. The effects of austempering treatment time on the strength, ductility, hardness, and abrasive wear resistance of JIS FCD 700 were studied. The austempering was carried out at a temperature of 315 °C and treatment times of 19, 30, 50, and 77 mins. The results indicate that the variations of the austempering time alternate the mechanical properties of the material, depending on the retained phases in the microstructure. A higher retained austenite induces a higher abrasive wear resistance and strengths, while the presence of acicular ferrite produces a lower hardness. Moreover, the large number of losses in martensite and retained austenite phases deteriorate the overall mechanical performances, as exhibited in the austempering time longer than 50 min. It is found that the 50 min of austempering time yields the optimum performance in terms of strengths (tensile strength of 1058 MPa and yield strength of 859 MPa), ductility (4.08 %), hardness (37 HRC), and abrasive wear resistance (0.21 x 10^-6 mm³/mm at 6.32 kg load). This optimum performance was achieved mainly due to the relatively larger amounts of retained austenite in the JIS FCD 700.

1. Introduction
Nowadays, ductile irons are greatly considered as replacement materials of steels for automotive components, such as camshaft, transmission gear, truck axle, and so on [1,2]. Ductile iron generally has better strength, ductility, hardness, and abrasive wear resistance compared to steel [3]. Fortunately, these are the characteristics required for the automotive components with regard to the ability to sustain external loads, machinability, and component lifetime [1,4,5]. Moreover, the melting temperature of ductile iron is lower than steel, which makes the casting process of ductile iron relatively simpler and less expensive [6].

JIS FCD (Japanese International Standard Ferro Casting Ductile) 700 is one of widely used ductile irons for automotive component material due to its good machinability along with its superior tensile and yield strengths [7,8]. However, the mechanical properties of the JIS FCD 700 are still needed to be developed to offer higher performances of the automotive components as demanded by the industry. Austempering is one effective way to enhance the ductile iron mechanical properties [6,9]. A ductile iron subjected to the heat treatment, namely austempered ductile iron (ADI), will retain the composition of nodular graphite, martensite, retained austenite, and acicular ferrite [10]. The presence of these microstructures allows the increment of strength, ductility, hardness, and abrasive wear resistance in the corresponding ductile iron [1,6].

In the process, the microstructure and mechanical properties of ADI are strongly affected by the austempering treatment temperature and time. As reported by Sohi et al. [11], the retained austenite phase increases as the austempering time increases, leading to an improvement in tensile and yield strengths. However, while the retained
austenite increases, the martensite phase tends to decrease. Although the ductility can be improved, the hardness might be deteriorated. Moreover, the reduced amount of martensite has an impact on the loss of hardness, leading to a decrease in abrasive wear resistance [1,10]. These findings were also corroborated by the work of Suratman et al. [6], implying a decrease in hardness as austempering treatment time increases specifically on JIS FCD 700 material. Unfortunately, these trade-offs aggravate the practical use of ADI in automotive components.

Considering the trade-off between hardness/abrasive wear resistance and ductility in JIS FCD 700, investigating parameters involved in the austempering time should be performed. This research was carried out to determine the austempering time which produces the optimum strength, ductility, hardness, and abrasive wear resistance for JIS FCD 700.

2. Experimental details
JIS FCD 700, with chemical composition listed in Table 1, was cast into a block-shaped mould with a dimension of 560 mm x 350 mm x 20 mm. For abrasive wear test, hardness test, and metallography, samples with 200 mm x 10 mm x 5 mm dimension were machined from the block. For the tensile test, samples were machined from the block, in accordance with ASTM E8 standard. Before all the samples were subjected to austempering, phase homogenization was carried out to all samples by normalizing at 900 °C for 30 min. After normalizing, austempering was carried out in a continuous furnace. All samples were austenitized at 900 °C for 30 min, and then quenched into a salt bath (50% NaNO\textsubscript{3} + 50% KNO\textsubscript{3}) at 315 °C for 19 min, 30 min, 50 min, and 77 min. Prior to metallography, all samples were ground using sand papers ranging from 180 mesh to 1500 mesh, polished with aluminium oxide, and then etched with Nital 5%. The metallographic testing was subsequently performed by using Olympus BX53MRF-S optical microscope. Rockwell hardness test (HRC) was conducted using Mitutoyo AR-10 hardness tester. Each sample was indented at 10 different points along the surface. Vickers hardness test (0,1 HV), conducted to characterize the phases present in metallography, was carried out using Willson-VH1102 hardness tester. The mean hardness of one phase was obtained from indentation at 5 different points. Tensile tests were carried out on each sample at room temperature, using Hung Ta tensile testing machine. Ogoshi High-Speed Universal Wear Testing Machine was used to conduct abrasive wear test. Abrasive ring 3 mm in width and 15 mm in radius span at 1,97 m/s for a sliding distance of 1 m. The applied load was increased from 3,16 kg to 6,32 kg, to 12,64 kg. Each load was applied 3 times for each sample. All three wear marks were measured using a Nikon measurement microscope. The mean wear mark was obtained from the measurement, and wear rate of each sample was calculated using $W_s = \frac{B \times b \times r \times l}{12 \times \pi \times l^3}$, where $W_s$ is the wear rate (mm$^3$/mm), $B$ is the width of the abrasive ring (mm), $b$ is the mean wear mark (mm), $r$ is the radius of abrasive ring (mm), and $l$ is the ring sliding distance (mm).

| C     | Si  | Mn  | P    | S    | Cr   | Cu   | Mg   |
|-------|-----|-----|------|------|------|------|------|
| 3,678 | 2,406 | 0,021 | 0,021 | 0,022 | 0,061 | 0,427 | 0,048 |

3. Results and Discussion
The phase hardness of JIS FCD 700 after normalizing and austempering treatments are presented in Table 2. The obtained phases agree with the phase hardness data as stated in Iron Castings Handbook [12]. According to the handbook, the hardness ranges of martensite, acicular ferrite, pearlite, ferrite, and nodular graphite are > 550 HV, 260 - 350 HV, 217 - 269 HV, 149 - 187 HV, 42 - 52 HV, respectively.

The microstructure of JIS FCD 700 after normalizing treatment is shown in figure 1(a). Ferrite, pearlite, and nodular graphite are found in this sample. Upon cooling to room temperature, carbon atoms diffuse away from austenite to nodular graphite, the carbon solubility of austenite starts to decrease at this point. This will create carbon depleted zones, which are ferrite nucleation zones. Pearlite nucleates at ferrite/austenite boundary as the eutectoid temperature is reached [3].
Table 2. Phase hardness of JIS FCD 700 after normalizing and austempering.

| Heat Treatment  | Nodular Graphite | Ferrite | Pearlite | Martensite | Acicular Ferrite |
|----------------|------------------|---------|---------|------------|-----------------|
| Normalized     | 47,5             | 170,3   | 252,0   | -          | -               |
| Austempered 19' | 47,7             | -       | -       | 792,2      | -               |
| Austempered 30' | 46,4             | -       | -       | 651,3      | 328,9           |
| Austempered 50' | 47,7             | -       | -       | 638,3      | 322,0           |
| Austempered 77' | 47,9             | -       | -       | 628,8      | 315,1           |

Figure 1(b) shows the microstructure of JIS FCD 700 after austempering for 19 min, which consists of martensite and nodular graphite. The martensite presents in the microstructure because of the 19 min of treatment time is inadequate for the residual austenite to decompose into acicular ferrite and high carbon austenite [13]. Consequently, the residual austenite will not be able to reach enough carbon contents required to stabilize at room temperature. Thus, it transforms to martensite upon cooling process to room temperature [14].

The microstructure of JIS FCD 700 after austempering for 30 min is presented in Figure 1(c). The treatment time of 30 min generates nodular graphite, martensite, and acicular ferrite. Acicular ferrite containing carbide is formed due to a fair amount (2.406 %) of silicon alloying content. The silicon increases carbon activity in ferrite and inhibits cementite formation. Thus, the ferrite will reach a carbon supersaturation, and carbides will precipitate within the ferrite [15]. Although retained austenite cannot be seen directly from the metallographic and Vickers hardness tests, its formation is highly possible in the 30 min of austempering. According to Panneerselvam et al., this is probably due to the formation of high carbon austenite as a result of residual austenite decomposition [16]. Austenite with high carbon content (approximately 1.7 %) will cause the $M_s$ to drop below room temperature. Therefore, the high carbon austenite will be retained at room temperature [17]. Some amounts of residual austenite, which are not decomposed into acicular ferrite and high carbon austenite, transform into martensite during the cooling process to room temperature [14].

Figure 1(d) shows the microstructure of JIS FCD 700 after austempering for 50 min. Similarly, the 50 min of treatment time generates microstructure consists of nodular graphite, martensite, and acicular ferrite. Moreover, retained austenite might also be produced in the 50 min treatment time, but possibly in a larger amount compared to the 30 min of treatment time [15,18]. The microstructure of JIS FCD 700 after austempering for 77 min is shown in Figure 1(e), which also consists of nodular graphite, martensite, and acicular ferrite. Although carbides cannot clearly be detected by the metallographic and Vickers hardness tests, the decomposition of the high carbon austenite into carbides is highly possible to occur at such longer treatment time [16].

Figure 2(a) shows the tensile and yield strengths of the normalized and austempered JIS FCD 700. The normalizing generates the lowest tensile and yield strengths value due to the presence of ferrite and pearlite [6,19]. Austempering for 19 min increases the tensile and yield strengths due to the existence of martensite [6]. However, a high amount of martensite leads to extreme brittleness of the sample, resulting in lower tensile and yield strengths compared to the other austempered samples [20]. Austempering for 30 min causes the tensile and yield strengths to increase, because the formed acicular ferrite absorbs some of the strain, reducing the brittleness effect caused by martensite [1]. The increase in the tensile strength can be attributed to the formation of retained austenite that transforms into martensite phases during plastic deformation, as elaborated by Aranzabal et al. [13]. Moreover, the 50 min of austempering also increases the tensile and yield strengths, which are possibly due to the formation of higher amounts of retained austenite [18]. Furthermore, we also argued that decreasing
amounts of martensite and retained austenite are the cause for the reduction of the tensile and yield strengths in the 77 min of treatment time [11].

Figure 1. Microstructures of JIS FCD 700 after (a) normalizing, (b) austempering for 19 min, (c) austempering for 30 min, (d) austempering for 50 min, and (e) austempering for 77 min.

Figure 2(b) shows the elongation of normalized and austempered JIS FCD 700. Normalized JIS FCD 700 possesses a fair elongation value because it is dominated by ferrite and pearlite [6,19]. Austempering time of 19 min produces JIS FCD 700 with the lowest elongation value, as the brittle martensite dominates the microstructure [6]. Austempering treatment for 30 min generates the highest value of elongation, which can be associated with the formation of acicular ferrite [21]. Sample austempered for 50 min generates lower elongation compared to that of 30 min. The presumed higher amount of retained austenite produced by austempering for 50 min causes elongation value to decrease, as it would transform into martensite during plastic deformation [13]. Austempering for 77 min also causes elongation value to decrease. The decrease of elongation value is caused by the formation of carbides [18].

Figure 2. (a) Tensile strength and yield strength of JIS FCD 700 after normalizing and austempering. (b) Elongation of JIS FCD 700 after normalizing and austempering.
Figure 3(a) shows the hardness of normalized and austempered JIS FCD 700 in HRC. The normalizing treatment generates the lowest hardness value since ferrite and pearlite exist in the microstructure [12]. The highest hardness value is obtained by 19 min of austempering time, which is dominated by martensite [11]. The increasing amount of martensite formed and the formation of softer phase (acicular ferrite and retained austenite) as the austempering time increases are responsible for the deterioration of hardness [13,18]. Although some amounts of retained austenite are possibly formed during the austempering of 30 min, 50 min, and 77 min, these phases may not transform into martensite when the samples were indented. Based on the work of Sohi et al., the presence of retained austenite does not lead to an increase in the hardness value [11].

Figure 3(b) shows the abrasive wear rate of normalized and austempered JIS FCD 700. The abrasive wear resistance is determined from the wear rate value. The higher the wear rate value, the lower the abrasive wear resistance. The normalized JIS FCD 700 possesses the lowest abrasive wear resistance. Both ferrite and pearlite microstructures contribute to the lower level of hardness compared to other samples. This leads to the highest wear rate value of all samples [12,22,23]. The 19 min of austempering produces an improved abrasive wear resistance compared to the normalized JIS FCD 700. The high amount of martensite leads to an increase in hardness, which in turn leads to an increase in abrasive wear resistance [12,23]. As the austempering time increases, the abrasive wear resistance tends to decrease due to a larger loss of martensite. Consequently, the hardness and abrasive wear resistance of JIS FCD 700 decline [23]. However, the sample with 50 min austempering opposes the trend, as exhibited in its lowest abrasive wear rate value. The high amount of retained austenite is assumed to be the reason behind the low wear rate value since retained austenite will transform into martensite when it was subjected to wear [10]. Moreover, the fair hardness of this sample, due to the presence of acicular ferrite, also contributes to the superior abrasive wear resistance [1]. The abrasive wear resistance of ductile iron, particularly JIS FCD 700, appears to be affected by two variables, i.e., the hardness and the amount of retained austenite. This result is in line with the work of Sahin et al. [1].

The comparison of normalized and austempered JIS FCD 700 performances are shown in Table 3. It can be inferred that the optimum strengths (tensile and yield), ductility, hardness, and abrasive wear resistance of JIS FCD 700 are obtained through the 50 min of austempering treatment time. The large amounts of retained austenite could possibly be attributed to the reason behind the higher level of strengths (tensile and yield) [13]. Moreover, the existence of acicular ferrite, which is moderate in amount, results in a fair ductility of this sample [21]. The fair hardness of this sample is due to the presence of acicular ferrite [11]. The presence of retained austenite, which is large in amount, is assumed to be the reason behind the exceptional abrasive wear resistance possessed by this sample [10].

**Figure 3.** (a) Hardness of JIS FCD 700 after normalizing and austempering. (b) Abrasive wear rate of JIS FCD 700 after normalizing and austempering.
addition, the fair hardness of this sample is also assumed to be contributing to the superior abrasive wear resistance this sample exhibits [1].

Table 3. Comparison of JIS FCD 700 performances.

| Heat Treatment      | σ(U) | σ(Y) | ε | Hardness | Abrasive Wear Resistance |
|---------------------|------|------|---|----------|--------------------------|
| Normalized          | 5    | 5    | 2 | 5        | 5                        |
| Austempered 19'     | 4    | 4    | 5 | 1        | 2                        |
| Austempered 30'     | 2    | 2    | 1 | 2        | 3                        |
| Austempered 50'     | 1    | 1    | 3 | 3        | 1                        |
| Austempered 77'     | 3    | 3    | 4 | 4        | 4                        |

*1 = best, 5 = worst.

4. Conclusion
In summary, strengths (tensile and yield), ductility (elongation), and hardness of JIS FCD 700 increase along with the treatment time up to 50 min, 30 min, and 19 min, respectively, but then decrease afterward. On the contrary, the abrasive wear resistance of JIS FCD 700 tends to be deteriorated as the austempering time increases. Nevertheless, 50 min of the austempering time yields an enhancement in the abrasive wear resistance. We argued that this is due to the relatively higher retained austenite contents and a high enough hardness. Hence, the optimum strength, ductility, hardness, and abrasive wear resistance of JIS FCD 700 can be obtained through the 50 min of austempering treatment time.

References
[1] Sahin Y, Erdogan M and Kilicli V 2007 Mater. Sci. Eng. A 444 31-38
[2] ASM Speciality Handbook, ASM Handbook: Cast Irons (Ohio: ASM International)
[3] Gagne M and Labrecque C 1988 Can. Metall. Q. 37 343-378
[4] Keough J R and Hayrynen K L 2000 Journal of Materials and Manufacturing 109 344-354
[5] Fatahalla N, Bahi S and Hussein O 1996 J. Mater. Sci. 31 5765-72
[6] Suratman S and Bandanadjaja B 2002 Jurnal Teknik Mesin 17 2
[7] Yanda H, Ghani, J A, Rodzi M N A M, Othman K and Haron C H C 2010 International Journal of Mechanical and Materials Engineering 5 182-190
[8] 1981 JIS Handbook 1981: Ferrous Materials and Metallurgy (Tokyo: Japanese Standard Association)
[9] Fuller A G 1985 Mater. Des. 6 127-130
[10] Shepperson S and Allen C 1988 Wear 121 271-287
[11] Sohi M H, Ahmadabadi, M N and Vahdat A B 2004 J Mater. Process. Trch. 153 203-208
[12] Walton C F and Opar T J 1981 Iron Castings Handbook: Covering Data on Grey, Malleable, Ductile, White, Alloy, and Compacted Graphite Irons (Michigan: Iron Castings Society, Inc.)
[13] Aranzabal J, Gutierrez I, Rodriguez-Ibabe, J M and Urcola J J 1997 Metall. Mater. Trans 28A 1143-56
[14] Eríc O, Rajnović D, Zec S, Sidjanin L and Jovanović M T 2006 Mater. Charact. 57 211-217
[15] Bhadreshia H K D K 1992 Bainite in Steels: Transformations, Microstructures, and Properties, (London: The Institute of Materials)
[16] Panneerselvam S, Martis C J, Putatunda S K and Boileau, J M 2015 Mater. Sci. Eng. R Rep. 626 237-246
[17] Aranzabal J, Gutierrez I and Urcola J J 1994 J. Mater. Res. Technol. 10 728-737
[18] Blackmore P A and Harding R A 1984 *Journal of Heat Treating* **3** 310-325
[19] Iacoviello F, Di Bartolomeo O, Di Cocco V and Piacente V 2008 *Mater. Sci. Eng. A.* **478** 181-186
[20] Kaczorowski M and Krzynska A 2008 *Arch. Foundry Eng.* **8** 87-92
[21] Erić O, Jovanović M, Šidanin L, Rajnović D and Zec S 2006 *Mater. Des.* **27** 617-622
[22] 2001 *ASM Metals Handbook* Vol. 18: *Friction, Lubrication, and Wear* (Ohio: ASM International)
[23] Khruschov M M 1974 *Wear* **28** 69-88