

# Influence of Tempering Time on the Microstructure, Hardness and Impact Toughness of Ductile Cast Iron

Herry Oktadinata<sup>1(⊠)</sup>, Sri Mulyati Latifah<sup>2</sup>, and Sastrawan Sastrawan<sup>1</sup>

<sup>1</sup> Mechanical Engineering Department, Universitas Jenderal Achmad Yani, Bandung, Indonesia herry.oktadinata@yahoo.com

<sup>2</sup> Metallurgy Engineering Department, Universitas Jenderal Achmad Yani, Bandung, Indonesia

Abstract. This study aims to obtain a suitable tempering time for ductile cast iron on microstructure, hardness, and toughness. The investigated ductile cast iron Yblock contains a carbon content of 3.45 wt%, silicon 2.6 wt%, manganese 0.635 wt.%, chromium 0.275 wt.%, nickel 1.58 wt.%, molybdenum 0.177 wt.%, and magnesium 0.1 wt% with a hardness of 41.5 HRC in the as-cast condition. In this experiment, the heat treatment was carried out with heating at 850 °C and holding time for 1 h. Then it was quenched with oil-cooling media and followed by a tempering process. Tempering at 350 °C was conducted with differences in holding times, 1 h, 2 h, and 3 h, respectively. The results show that a tempering with a holding time of 1 h had a hardness of 52.83 HRC and an impact energy of 1.90 J. Tempering with a hold time of 2 h had a hardness of 50 HRC and an impact energy of 2.28 J. And tempering for 3 h had a hardness of 46.3 HRC and impact energy of 2.80 J. From these results, it can be concluded that tempering time influences the mechanical properties of ductile cast iron. The longer the tempering time, the lower the hardness value and the higher the toughness. It can be attributed to fragmented carbides and graphite nodule evolution during heat treatment.

Keywords: Tempering · Microstructure · Cast Iron

# 1 Introduction

Ductile cast iron is a class of cast iron in which there is a spherical or nodular form of graphite carbon in the metal matrix. The microstructure of ductile cast iron is characterized by spherical graphite nodules, which make it ductile. This microstructure is composed of spherical graphite in a ferrite-pearlite matrix. The spheroidal graphite form provides superior ductility and higher impact strength than gray cast iron [1-3]. The application of ductile cast iron has continued to increase over the years due to its relatively inexpensive production. It can be produced with a wide range of microstructures and mechanical properties [4].

Ductile cast irons are a family of cast irons with an exciting combination of mechanical properties in both as-cast and heat-treated conditions [5, 6]. Variations in the mechanical properties of ductile cast iron can be obtained by applying to vary the heat treatment cycle [7, 8]. The matrix structure of ductile cast iron can be modified by heat treatment to produce the required properties [9]. The mechanical properties of ductile cast irons are also influenced by their chemical composition. Previous research reported the addition of nickel could improve the impact toughness [10].

The microstructure of ductile cast iron consists of a metal matrix and graphite nodules. Graphite develops along with the metal matrix during solidification. The graphite formation consumes carbon from the surrounding molten iron [11]. The relationship between mechanical properties and the number of nodules for cast materials can be seen in that as the number of nodules increases, the mechanical properties increase [4, 6, 9, 12]. This nodularization process is achieved as a consequence of nodularization effect of several elements such as magnesium, calcium, and others from the rare earth elements [2, 8].

Nodule count has significantly influenced the mechanical properties of ductile cast iron. It is defined as the number of graphite nodules per certain unit area. Nodule count can be increased by inoculation treatment. Increasing the number of nodules by inoculation treatment usually makes the nodules more spherical. Therefore, a high nodule count is generally associated with increased nodularity [12]. Previous works reported that nodule count increased when BiLaCeSb oxide and FeSi inoculants were mixed and added into molten iron [13]. Nodule count is also correlated with the solidification time, increasing when the section thickness decreases [4].

The aim of this work is to obtain a suitable tempering time for ductile cast iron on microstructure, hardness, and toughness. Observation using optical microscopy, Rockwell hardness test, and Charpy impact toughness measurement were performed to understand the microstructure and mechanical properties.

# 2 Experimental Methods

### 2.1 Material

In this work, the melts were produced in an electric arc furnace with charge materials of steel scrap, carburizer, and alloying elements (Fe–Mn, Fe–Cr, Fe–Si Fe-Mo, and Ni square). Fe-Si-Mg was used as a nodulizer, and Fe-Si- Ca-Al was used as an inoculant. They were placed at the bottom of the ladle before tapping for spheroidization. Fe-Si-Mg converts the graphite form in cast iron from flake into nodules.

At a temperature of 1550 °C, the melts were tapped into a ladle. The melt was nodularized using Fe–Si–Mg (5.31 wt.% Mg) and inoculated in the ladle using Fe–Si (67 wt.% Si). After holding for several minutes, the molten iron was cast in a chill mold for a chemical composition test. The chemical composition was determined via spectrometer analysis, where a PMI Master Smart Oxford portable spectrometer was used. The chemical composition result is presented in Table 1. Then the melt was poured into green sand molds to obtain Y-blocks cast iron (see Fig. 1).

С	Si	Mn	Р	S	Cr	Ni	Мо	Mg	Al	Cu	V	Ti
3.45	2.6	0.635	0.03	0.002	0.275	1.58	0.177	0.1	0.026	0.024	0.011	0.013

Table 1. Chemical compositions (wt.%) of experimental cast iron



Fig. 1. a) molten metal was poured into green sand molds, b) Y-blocks cast iron

### 2.2 Heat Treatment Procedure

Samples for microstructure observation, Charpy impact, and hardness tests were taken from the Y-blocks. After cutting and machining, the samples were heat treated according to the heat treatment cycles standard. In this experiment, three as-cast ductile cast iron samples were heated to 850 °C and held for 1 h. Then those three samples rapidly cooled in the oil followed tempered to 350 °C with different tempering times: 1 h, 2 h, and 3 h, respectively. And finally, those samples were cooled in the air to ambient temperature. The condition and heat treatment cycle for each sample is presented in Table 2. In this work, the as-cast sample without heat treatment is used for reference to compare with the samples with heat treatment.

#### 2.3 Microstructure, Hardness Test and Impact Toughness

Microstructure analysis was performed on the samples which were cut from the Yblocks. Metallographic samples were prepared by grinding with abrasive paper and then polished with diamond paste. Nital 2% was selected to reveal the microstructure features. The microstructure was examined using an Olympus DP22 optical microscope on each sample: NHT, T-1H, T-2H, and T-3H.

The mechanical tests consisted of hardness and impact toughness which were performed according to the test standard. Specimens for impact and hardness tests were taken from the Y-blocks. Furthermore, they were heat treated after machining according to the heat treatment cycles described in Table 2. Hardness tests were performed using a Rockwell hardness tester. The hardness was measured over three indentations, and the average value was calculated. The impact toughness was measured using a Charpy impact machine. Impact test specimens of  $10 \times 10 \times 55$  mm were machined according to ASTM E23 standards. Three measurements were performed on each sample, and the average value was calculated.

Sample code	Condition	Heat treatment cycle
NHT	As-cast	-
T-1H	Hardened-tempered with tempering time 1 h	Heated to 850 °C and soaked for 1 h, then rapid cooled in the oil followed tempered to 350 °C for 1 h, and finally air cooled to ambient temperature
Т-2Н	Hardened-tempered with tempering time 2 h	Heated to 850 °C and soaked for 1 h, then rapid cooled in the oil followed tempered to 350 °C for 2 h, and finally air cooled to ambient temperature
Т-3Н	Hardened-tempered with tempering time 3 h	Heated to 850 °C and soaked for 1 h, then rapid cooled in the oil followed tempered to 350 °C for 3 h, and finally air cooled to ambient temperature

Table 2.	The heat	treatment	cycle of	the samples
----------	----------	-----------	----------	-------------



Fig. 2. Microstructure image of the as-cast sample under two surface conditions: a) etched, b) non-etched

# 3 Results and Discussion

### 3.1 Microstructure Characterization

The microstructure was observed, and the results are shown in Figs. 2, 3, 4 and 5. Figure 2 shows the microstructure image of the as-cast sample with the etched (2a) and non-etched (2b) surface. It can be seen that the microstructural features of the as-cast sample are composed of nodular graphite, ferrite, pearlite, and carbides. A similar result was reported by other researchers [10, 14].

The etched microstructure of the hardened-tempered sample in Fig. 3 shows fragmented carbides and dispersed graphite particles. The presence of carbides can increase the hardness and wear resistance, but it can deteriorate the toughness. Figures 3a–c presented microstructure with tempering times 1 h, 2 h, and 3 h, respectively, which show the matrix and graphite after tempering. Micrographs with higher magnification are shown in Fig. 3d–f.



**Fig. 3.** (a–c) Microstructure with tempering times 1 h, 2 h, and 3 h, respectively, mag. 200x. (d–f) Micrographs with higher magnification, 1 h, 2 h, and 3 h, mag. 500x.



Fig. 4. The microstructure of non-etched samples after tempering for: (a) 1 h, (b) 2 h, (c) 3 h. Mag.  $100 \times$ 

Furthermore, graphite nodules were observed via a non-etched surface, as shown in Fig. 4a–c. The evolution of graphite nodules can be seen in three different tempering time conditions. Longer tempering time indicated the size of graphite nodules increased a bit.

### 3.2 Hardness and Impact Toughness Properties

The hardness test results are presented in Table 3. Compared to the as-cast condition, the hardened-tempered showed increasing hardness due to increasing the fraction area of the hard phase. The hardened-tempered treatment has improved the hardness by 12 to 22% compared to the as-cast condition. However, the hardness of the hardened-tempered

#### 104 H. Oktadinata et al.

Sample code	Condition	Hardness (HRC)			
		1st	2nd	3rd	Average
NHT	As-cast	40	43	41.5	41.5
T-1H	Hardened-tempered with tempering time 1 h	51	50	51.5	50.8
Т-2Н	Hardened-tempered with tempering time 2 h	51	48.5	50.5	50.0
Т-3Н	Hardened-tempered with tempering time 3 h	44	46.5	48.5	46.3

 Table 3.
 The hardness test results

Sample code	Specimen No.	Impact (J)	Average (J)	Impact (J/cm2)	Average (J/cm2)	
T-1H	1	2.41	1.90	2.76	2.17	
	2	1.64		1.88		
	3	1.64		1.88		
T-2H	4	2.02	2.28	2.31	2.61	
	5	2.41		2.76		
	6	2.41		2.76		
Т-3Н	7	3.57	2.80	4.1	3.21	
	8	2.41		2.76		
	9	2.41		2.76		

 Table 4.
 The impact test results

sample decreased when the tempering time increased. It can be attributed to fragmented carbides and graphite nodule evolution during heat treatment.

The impact test was performed according to the Charpy impact test, and the results are shown in Table 4. A tempering time of 3 h has improved the toughness by 47% than a tempering time of 1 h. Impact energy increased when the tempering time increased.

The hardness and impact energy relationship was described based on the hardness and impact test results, as shown in Fig. 5. A longer tempering time decreases the hardness and improves the toughness. This result corresponded to increasing ductility when the tempering time increased. Tempering relieves the internal stresses in the material and consequently expands its ductility.



Fig. 5. Relation between hardness (HRC) and impact energy (J)

# 4 Conclusion

This study investigated the hardened-tempered condition of ductile cast iron with three different tempering times to improve its toughness. Tempering at 350 °C was conducted with differences in holding times, 1 h, 2 h, and 3 h, respectively. A tempering with a holding time of 1 h had a hardness of 52.83 HRC and an impact energy of 1.90 J. Tempering with a hold time of 2 h had a hardness of 50 HRC and an impact energy of 2.28 J. And tempering for 3 h had a hardness of 46.3 HRC and impact energy of 2.80 J. It can be concluded that the longer the tempering time, the lower the hardness value and the higher the toughness. It can be attributed to fragmented carbides and graphite nodule evolution during heat treatment.

# References

- R. F. Pereira, V. E. R. da Silva Filho, L. B. Moura, N. A. Kumar, A. R. de Alexandria, and V. H. C. de Albuquerque, "Automatic quantification of spheroidal graphite nodules using computer vision techniques," J. Supercomput., vol. 76, no. 2, pp. 1212–1225, 2020.
- A. de Albuquerque Vicente, J. R. S. Moreno, T. F. de Abreu Santos, D. C. R. Espinosa, and J. A. S. Tenório, "Nucleation and growth of graphite particles in ductile cast iron," J. Alloys Compd., vol. 775, pp. 1230–1234, 2019.
- H. Oktadinata, S. B. Pratomo, S. A. Dwiwanto, Y. Nugraha, and U. Mardono, "Microstructural Investigation of Acicular Nodular Cast Iron Used for Work Rolls," in Materials Science Forum, 2022, vol. 1067, pp. 47–52.
- J. M. Borrajo, R. A. Martínez, R. E. Boeri, and J. A. Sikora, "Shape and count of free graphite particles in thin wall ductile iron castings," ISIJ Int., vol. 42, no. 3, pp. 257–263, 2002.
- H. Khodaverdizadeh and B. Niroumand, "Effects of applied pressure on microstructure and mechanical properties of squeeze cast ductile iron," Mater. Des., vol. 32, no. 10, pp. 4747– 4755, 2011.
- 6. M. A. Kenawy, A. M. Abdel-Fattah, N. Okasha, and M. El-Gazery, "Mechanical and structural properties of ductile cast iron," Egypt. J. Sol, vol. 24, no. 2, pp. 151–159, 2001.
- R. PJJ, A. AF, F. VE, A. FR, A. V. HN, and S. JA, "Low temperature impact tests in austempered ductile iron and other spheroidal graphite cast iron structures," ISIJ Int., vol. 41, no. 4, pp. 372– 380, 2001.

- 8. J. Bai et al., "Effect of cyclic quenching treatment on microstructural evolution and properties of ductile cast iron," J. Iron Steel Res. Int., pp. 1–11, 2022.
- 9. O. J. Akinribide et al., "A Review on Heat Treatment of Cast Iron: Phase Evolution and Mechanical Characterization," Materials (Basel)., vol. 15, no. 20, p. 7109, 2022.
- Y. Sun, S. Hu, Z. Xiao, S. You, J. Zhao, and Y. Lv, "Effects of nickel on low-temperature impact toughness and corrosion resistance of high-ductility ductile iron," Mater. Des., vol. 41, pp. 37–42, 2012.
- M. A. Azeem, M. K. Bjerre, R. C. Atwood, N. Tiedje, and P. D. Lee, "Synchrotron quantification of graphite nodule evolution during the solidification of cast iron," Acta Mater., vol. 155, pp. 393–401, 2018.
- D. de Oliveira Fernandes, C. T. M. Anflor, J. N. V. Goulart, and B. Baranoğlu, "Nodular Cast Iron GGG40, 60, 70 Mechanical Characterization from Bars and Blocks Obtained from Brazilian Foundry," Metals (Basel)., vol. 12, no. 7, p. 1115, 2022.
- H. Tsuji, H. Chono, N. Yamamoto, T. Kai, and Y. Igarashi, "Analysis of Graphite Nuclear in Spheroidal Graphite Cast Iron and Mechanism of Nodule Count Increase by Bismuth Oxide," Mater. Trans., vol. 62, no. 9, pp. 1393–1400, 2021.
- P. Weiß, M. Riebisch, and A. Bührig-Polaczek, "Mechanical properties and impact toughness of nickel and aluminum alloyed high silicon ductile iron," in Materials Science Forum, 2018, vol. 925, pp. 304–310.

**Open Access** This chapter is licensed under the terms of the Creative Commons Attribution-NonCommercial 4.0 International License (http://creativecommons.org/licenses/by-nc/4.0/), which permits any noncommercial use, sharing, adaptation, distribution and reproduction in any medium or format, as long as you give appropriate credit to the original author(s) and the source, provide a link to the Creative Commons license and indicate if changes were made.

The images or other third party material in this chapter are included in the chapter's Creative Commons license, unless indicated otherwise in a credit line to the material. If material is not included in the chapter's Creative Commons license and your intended use is not permitted by statutory regulation or exceeds the permitted use, you will need to obtain permission directly from the copyright holder.

