

# Effect of Niobium Addition on The Morphological Behaviour of Au tempered Ductile Iron

S.K Alias<sup>1\*</sup>, M.N. Halmy<sup>2</sup>, M. A. Mat Shah<sup>3</sup> B. Abdullah<sup>4</sup>, M.H. Ismail<sup>5</sup>, M.F Idham<sup>6</sup>

<sup>1,2,3</sup>Faculty of Mechanical Engineering, Universiti Teknologi Mara, Pasir Gudang  
<sup>4,5,6</sup> Faculty of Mechanical Engineering, Universiti Teknologi Mara Shah Alam, M.K. Talari  
Faculty of Applied Science, Universiti Teknologi Mara, Shah Alam  
\*Corresponding author E-mail: khadijah\_alias@johor.uitm.edu.my

## Abstract

This paper investigate the effect of niobium addition at 0.5 wt% to the morphological behavior of austempered ductile iron. Austempered ductile iron (ADI) and niobium alloyed austempered ductile iron was prepared by first austenitizing the samples at 900°C before austempering process was conducted in salt bath at 1 hour holding time. The samples were then cooled to room temperature. The microstructural observation and analysis in term of nodule count, nodularity percentage and graphite percentages were conducted using Olympus B X 41M microscope with IMAPs 4.0 edition, Scanning electron microscopy (SEM) analyzer using SMARTSEM software and energy-dispersive X-Ray (EDX) spectroscopy. It was found that addition of niobium resulting in formation of niobium carbide (NbC) and much finer retained austenite and acicular ferrite structure. However, the nodularity, nodule count and graphite area were reduced due to the presence of niobium carbide.

**Keywords:** Austempered ductile iron, niobium, morphological behaviour.

## 1. Introduction

Austempered Ductile Iron (ADI) which was obtained through heat treatment of ductile iron possesses very good mechanical comprising of enhance tensile strength, fatigue toughness, ductility and hardness properties and wear resistances that make it the right substitute for wrought steel [1-3]. The enhancement of ductile iron and austempered ductile iron in term of mechanical and wear properties could be achieved through addition of alloying elements such as silicon [4-5], aluminium [6], titanium [7] and also controlling of heat treatment parameters [8-10]. The addition of silicon had an effect of increasing the amount of small spheroids of graphite and refining the microstructure which resulted in improvement of mechanical properties such as yielding stress and impact toughness [4]. Addition of aluminium in ductile iron up to 0.5 wt% profoundly increased the ultimate tensile strength but adding more than 0.5 wt% up to 2.0 wt % unfortunately reduced the ultimate tensile strength of ductile iron and austempered ductile iron [6]. The effect of titanium ranging from 0 to 1.47wt % in hypereutectic high chromium cast iron microstructure was investigated [7]. The primary  $M_7C_3$  carbides refined gradually with the increment of titanium concentration, thus affecting the properties of high chromium cast iron. This could concluded that the addition of alloying element help strengthen and improve the properties of cast iron in both as cast and heat treated conditions. Past researcher had proven that the addition of niobium in between 0.48-0.74 wt % had significantly improved the impact toughness and wear resistances of high chromium cast iron [11] while adding niobium at the rate of 0.1 to 0.4 wt% in grey cast iron resulted in improvement of both hardness and tensile properties [12]. Addition of niobium at 0.5 wt% in austempered ductile iron was also found to improve the tensile strength and elongation properties due to

production of fine ferrite platelets and lower bainite structures [13]. Although the effect of niobium addition on the properties of cast iron had been extensively studied in the past, there are least study on the microstructural development of this material. Thus, this study focused on the effect on niobium on the morphological behaviour of austempered ductile iron.

## 2. Research Work

Ductile iron and niobium alloyed ductile iron were prepared through conventional  $CO_2$  sand casting process. Austempering process start with austenitizing the samples at 900°C before conducting austempering process in salt bath at 1 hour holding time. The samples were then cooled to room temperature, as shown in Figure 1. Table 1 shows the chemical composition of ductile iron and niobium alloyed ductile iron extracted from Spectro Maxx Spark emission machine. The microstructural observation and analysis in term of nodule count, nodularity percentage and graphite percentages were conducted using Olympus B X 41M microscope with IMAPs 4.0 edition, Scanning electron microscopy (SEM) analyzer using SMARTSEM software and energy-dispersive X-Ray (EDX) spectroscopy.

**Table 1:** Chemical composition of ductile iron and niobium alloyed ductile iron

Material	C	Si	Mn	P	S	Cu	Ni	Cr	Nb	Fe
DI	3.04-3.85	1.97-2.02	0.383-0.480	0.024-0.084	0.071-0.085	0.65-0.71	0.414-0.456	0.013-0.052	0	balance
0.5% Nb-DI	3.41-3.73	2.12-2.27	0.348-0.380	0.038-0.061	0.049-0.075	0.57-0.64	0.481-0.500	0.034-0.055	0.5	Balance

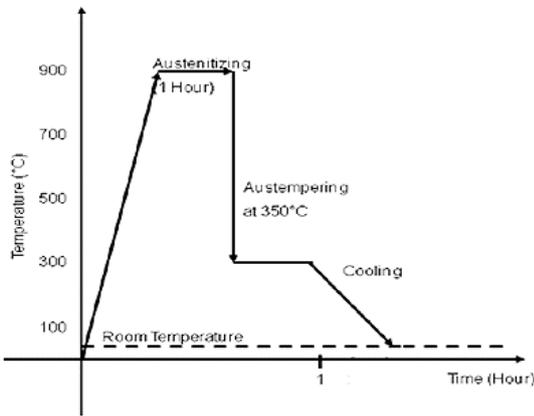


Figure 1: The schematic diagram of the austempering heat treatment.

### 3. Research Findings

#### 3.1. Microstructure analysis

Figure 2 (a) and 2 (b) shows the microstructure and SEM observation of as cast niobium alloyed ductile iron (Nb-DI) which depicted presence of graphite surrounded by pearlitic structure and niobium carbide (NbC). The compositions and graph elements of sparked spectrum obtained through EDX analysis proven that the sparked polygonal form particle is niobium carbide (NbC) with the highest atomic percentages of Nb (69.75%) and followed by C (16.06%), as shown in Figure 2 (c) and Figure 2 (d). Similarly, it found that the addition of niobium to different types of cast iron also formed niobium carbide (NbC) in their microstructure [15]. The presence of niobium carbide help improve the hardness and tensile strength of alloyed ductile iron by reducing the thickness of lamellar pearlitic and also by strengthening the matrix of the alloys due to a hard-phase dispersion mechanism.

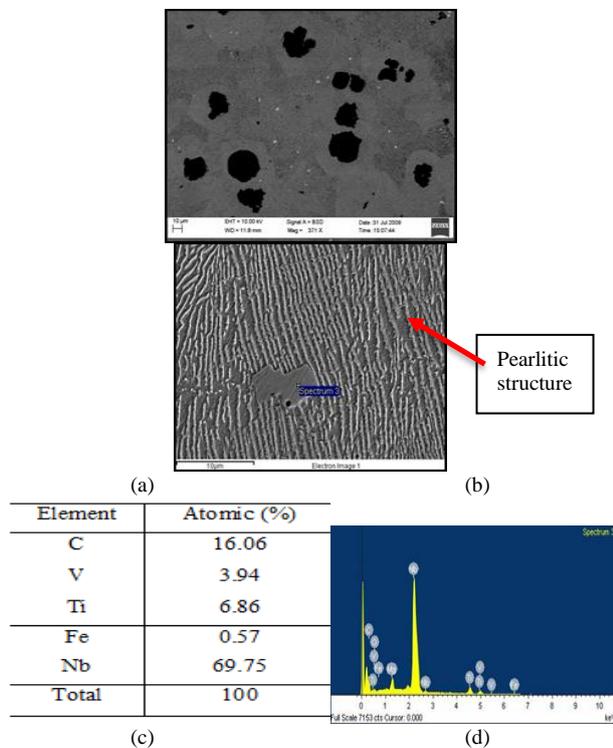


Figure 2: SEM result of 0.5 wt % Nb-DI at (a) lower magnification (b) higher magnification, and (c) composition and (d) peak of element of at sparked spectrum.

Figure 3 shows the microstructure of austempered ductile iron sample which displaying presences of graphite, retained austenite

and acicular ferrite. As the selection of austempering temperature was held to 350°C, the resultant microstructure was expected. The formation of retained austenite and acicular ferrite known as “ausferrite” give austempered ductile iron excellent combinations of higher strength and hardness [6]. Similar microstructures were also observed in the studies by past literatures [2-3] During austempering process, austenite will first produced at ferrite grain boundaries and transformed into ferrite by mean of nucleation and growth processes. The nucleation will formed ferritic matrix comprising of carbon-enriched austenite in thin lamellar form before the austenite continues to nucleate at the grain boundaries of ferrite. As austenite becomes enriched and saturated with carbon, the carbon will hinder the growth of ferrite because the diffusion of carbon occurs before the formation of ferrite. The final resultant microstructure is combination dark region of needle-like structures which is acicular ferrite and white regions which is carbon-enriched austenite, known as “Ausferrite” structure [14].

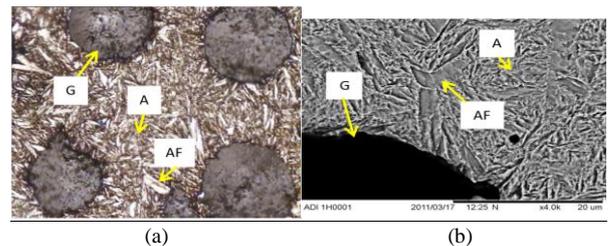
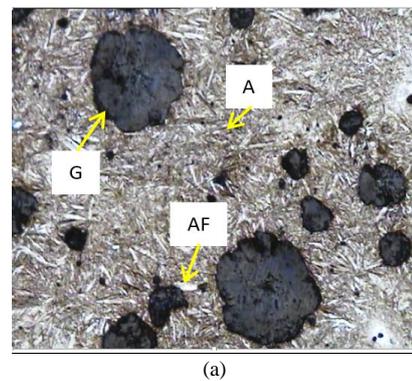


Figure 3 : (a) Microstructure and (b) SEM observation of ADI sample showing G-graphite, A- retained austenite, AF - acicular ferrite

The microstructure of Nb-ADI which consisted of graphite nodules surrounded by matrix of retained austenite and acicular ferrite with presence of niobium carbide was shown in Figure 4 (a) and 4 (b). In the microstructure, the presence of acicular ferrite and retained austenite is finer as compared to ADI samples due to the formation of niobium carbide. Addition of niobium in high chromium cast iron also present similar findings as the microstructure also consist of hexagonal primary carbide, petal-like distributed eutectic carbide and dispersive secondary carbide. The white NbC particles in the form of polygon morphologies, were found in both inside the primary carbides and on the edge of the primary carbides, which reduced the size of acicular ferrite in the microstructure of the samples [11].

Previously, Niobium has been added with the purpose of refining the austenite grain size during the solidification process as the niobium carbide had the effect of retarding the austenite grain growth. Niobium carbides which was formed at high temperature act as the nuclei for the proeutectic austenite precipitation and also the eutectic colonies. In addition, addition of niobium could also increases the volume fraction of eutectic austenite because of the reduction of the amount of carbon precipitating as graphite during the eutectic solidification process[15].



(a)

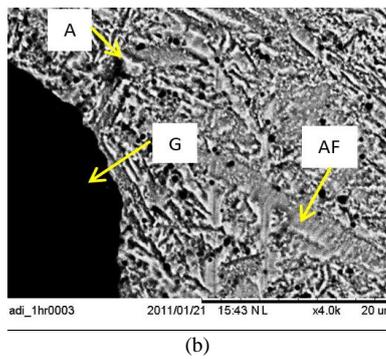


Figure 4: (a) Microstructure and (b) SEM observation of Nb-ADI sample showing G-graphite, A- retained austenite AF- acicular ferrite platelets.

### 3.2. Graphite characteristics of ADI and Nb-ADI samples

The properties of graphite in term of nodularity percentage, graphite area and nodule count is depicted in Table 2. It was shown that addition of niobium to the austempered ductile iron had reduced the graphite nodularity up to 6 %, the graphite area to 15% and the nodule count to 10%. The addition of niobium which promoted niobium carbide was found to be the main reason to this findings as it had the effect of reducing the cell size and blunt the graphite flake sizes [12]. Other than the microstructure, the properties of austempered ductile iron also depending on the characteristics of the graphite. A good quality base ductile iron must consist of nodularity of more than 85 %, a minimum nodule count of more than 120/mm<sup>2</sup>, and also consistent chemistry. High amount of nodule count is vital in reducing the segregation of the alloying elements and avoid the carbide formation as both could lead to delaying the start of the ausferrite transformation and decreasing the transformation rate [10]. However, based on the results of graphite characteristic, it could be said that Nb-ADI samples still exhibited the properties of good quality ductile iron as all the properties are within the required range.

Table 2: Graphite characteristics of ADI and Nb-ADI samples

Type of samples	Form type	Nodularity (%)	Graphite area (%)	Nodule count
ADI	IV	98	13	216
Nb-ADI	IV	92	11	194

### 3.3. Failure surface analysis

Figure 5 (a) shows the SEM fractograph of austempered ductile iron samples after tensile test. The SEM fractograph shows ductile fracture mode surrounding the graphite nodules in intergranular failure mode. However, addition of niobium had transformed the mode of failure from ductile to ductile-brittle intergranular as less cleavage was observed in the SEM fractograph shown in Figure 5 (b). It could also be observed that niobium addition had reduce the amount and nodularity of graphite as the graphite appear to be less visible in the SEM image. similarly, addition of aluminum and titanium also exhibited the same effect on the graphite characteristics of alloyed austempered ductile iron [6] and high chromium cast iron [7].

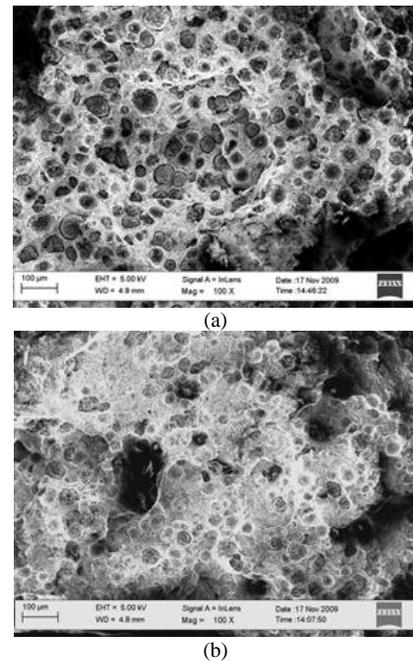


Figure 5. SEM Fractograph of the sample after tensile test for (a) ADI and (b) Nb-ADI

## 4. Conclusions

In this study, the influence of niobium addition on the microstructure of austempered ductile iron was investigated and the following conclusion could be obtained:

The microstructure of austempered ductile iron consist of nodular graphite surrounded by retained austenite and acicular ferrite while the addition of niobium to austempered ductile iron promoted the formation of Niobium carbide (NbC) to the microstructure. Addition of niobium resulting in much finer retained austenite and acicular ferrite structure but reduced the nodularity, nodule count and graphite area due to the presence of niobium carbide.

## Acknowledgement

The authors would like to express their gratitude to the Ministry of Science and Technology, Malaysia (MOSTI) and Ministry of Higher Education Malaysia (MOHE), and Universiti Teknologi Mara for the financial support given to this work through research grant of 600-IRMI/DANA 5/3 REI(12/2017).

## References

- [1] N. Bhople, S. Pahl, M. Hame, and S. Dhande "Austempering parameters and machinability of austempering ductile iron: A comprehensive review on effective parameters" *International Journal of Innovative Research in Science, Engineering and Technology* 5(2) (2016) 1197-1211.
- [2] S. Balos, I. Radisavljevic, D.Rajnovic, M. Draicanin, S. Tabakovic, O. Eric-Cekic and L. Sidjanin "Geometry, mechanical and ballistic properties of ADI material perforated plates" *Materials and Design, Elsevier Series*. 83 (2015) 66-74.
- [3] S. Méndez, U. de la Torre, P. Larrañaga (2015) "Processing Thickness Window for As-cast Ausferritic Castings" *AFS Proceedings 2015* © American Foundry Society, Schaumburg, IL USA. Paper 15-010, Page 1 of 7.
- [4] A. Alhusein, M. Risbet, A. Bastien, J.P. Chobaut, D. Balloy and J. Favregeon "Influence of silicon and addition elements on the mechanical behavior of ferritic ductile cast iron" *Materials Science & Engineering A* 605 (2014) 222–228.
- [5] A.G.F. Alabi, J.O. Aweda, and F.I. Aluko "Influence of Ferrosilicon Manganese on the Sulphur Content and Microstructure in the Production of Austempered Ductile Iron (ADI)" *Journal Of Production Engineering* (2017) Vol.20 (2).

- [6] M.M. Anil Kumar, Dr. R Suresh and B Vinay “Development of Effect Austempered Ductile Iron and Property Evaluation with Addition of Aluminium” *International Journal of Advances in Scientific Research and Engineering* 3(2017) 8.
- [7] Z. Xiaohui, X. Jiandong, F. Hanguang, G. Yiming “Effect of titanium on the as-cast microstructure of hypereutectic high chromium cast iron” *Materials Characterization* 59(9)(2008) 1221-1226.
- [8] S. Savićević, H. Avdušinović, A. Gigović-Gekić, Z. Jurković, M. Vukčević, M. Janjić “Influence of The Austempering Temperature on The Tensile Strength of The Austempered Ductile Iron (ADI) Samples” *Metallurgija* 56 (2017) 1-2, 149-152.
- [9] F. Zanardi, F. Bonollo and G. Angella “A Contribution to New Material Standards for Ductile Irons and Austempered Ductile Irons” *International Journal of Metal Casting* 11(2017)1.
- [10] M. Janjić, H. Avdušinović, Z. Jurković, F. Bikić, S. Savićević “Influence of Austempering Heat Treatment on Mechanical and Corrosion Properties of Ductile Iron Samples” *Metallurgija* 55 (2016) 3, 325-328.
- [11] Z. Zhiguo, Y. Chengkai, Z. Peng and L. Wei “Microstructure and Wear Resistance of High Chromium Cast Iron Containing Niobium” *Research & Development* 11(2014)3.
- [12] M.R. Muhaffel and G. Yıldırım (2016) “Effect of Niobium Addition in Grey Cast Iron on Mechanical Properties. *UCTEA Chamber of Metallurgical & Materials Engineers, Proceeding book*.
- [13] B. Abdullah, S.K. Alias, A. Jaffar, A.A. Rashid, M. Haskill and A. Ramli “Tensile Strength Properties of Niobium Alloyed Austempered Ductile Iron on different austempering time” *Advanced Materials Research* (2012) 457-458.
- [14] P. Sellamuthu, D. G. Harris Samuel, D. Dinakaran, V. P. Premkumar Zushu Li and S. Seetharaman. “Austempered Ductile Iron (ADI): Influence of Austempering Temperature on Microstructure, Mechanical and Wear Properties and Energy Consumption” *Metals* 8(2018)53.
- [15] A. Bedolla-Jacuinde (October 19th 2016). Niobium in Cast Irons, Progress in Metallic Alloys Vadim Glebovsky, IntechOpen, DOI: 10.5772/64498. Available from: <https://www.intechopen.com/books/progress-in-metallic-alloys/niobium-in-cast-irons>