

Effect of Nickel Content on Tensile, Hardness Properties and Dry Sliding Wear Behaviour of Austempered Ductile Iron

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Abstract. The increasing interest in the development of austempered ductile cast irons has led to a great deal of study being done on their physical metallurgy and mechanical properties. The exceptional strength and hardness of these cast irons are well recognised. In this study, samples of permanent moulded toughened austempered ductile iron (PMADI) that are subjected to an austempering heat treatment are used to investigate the effects of nickel content on their tensile strength and hardness strength. PMADI samples were developed using Casting and samples with 1% nickel 2% Nickel content were prepared. The characteristics of PMADI samples were compared with those of samples that had been hardened and subjected to normal austempering heat treatment. Investigations were conducted on mechanical characteristics, such as tensile and hardness tests. ADI samples have shown higher levels of hardness and tensile strength. The findings showed that the mechanical characteristics of PMADI samples with 2.0% nickel were noticeably better. The scanning electron microscopy images revealed both ductile and brittle mode of fracture. Wear testing was conducted utilising a pin-on-disk machine in dry sliding method. Large quantities of austenite, which are produced at higher austempering temperatures, help to increase wear resistance through martensitic transformation. The coarse ausferrite structure in ADIs experiences adhesion, delamination, and mild abrasion in addition to oxidational wear, according to an analysis of worn surfaces and the kind of wear debris. An increase in mechanical strength seems to be correlated with an increase in retained austenite content.

Key words: nickel, austempered ductile iron, tensile, hardness, wear.

Introduction

Through heat treatment, ADI, a grade of iron, is transformed into austenite, a metastable face-centred-cubic (FCC) matrix solid solution that is stable at room temperature due to its saturation with 1.8–20% carbon [1]. The unique microstructure of ADI is composed of carbon-stabilized austenite and acicular ferrite.

Austempered Ductile Iron (ADI) is created when ductile iron undergoes the isothermal heat treatment procedure known as "Austempering." Compared to ductile iron, it offers superior characteristics. The microstructure that results from austempering ductile cast iron is called "Ausferrite" and is made up of stabilised austenite that has been enhanced with carbon and fine acicular ferrite. Compared to several ferrous and aluminium alloys, the novel microstructure produces outcomes with greater capabilities. For a given degree of ductility created by traditional heat treatment procedures, Ausferrite displays double the strength as compared to pearlitic, ferritic, or martensitic structures. The ausferrite microstructure affects the austempered ductile iron's mechanical characteristics. Compared to other grades of ductile iron, the austempered matrix provides a superior tensile strength to ductility ratio [2]. Due to its ausferrite microstructure, that is influenced by alloyed components and heat treatment parameters, austempered ductile iron can have a wide range of combined characteristics.

Even though there has been a lot of research done on the aforementioned topics by many researchers, more research is needed in this area to better understand alloyed and un-alloyed PMADI samples. This is because PMADI alloys are used to make critical parts for mining, power plants, and automobiles, and these alloys extend component life by increasing fracture toughness [3]. The following are some of the literatures analysed on Austempered Ductile iron subjected to austenitization Table 1.

Table 1. List of literatures on Austempered Ductile iron.

| Ref | Investigation details | Outcome |
|-----|--|--|
| [4] | Investigation of nickel content and austempering temperature effects. XRD analysis for retained austenite content estimation. | Nickel content improves strength and hardness, peaking at 0.8%. Retained austenite increases with higher austempering temperatures. |
| [5] | Experimental investigation of microstructures and mechanical properties. Characterization of hardness, compressive strength, and wear resistance. | Vanadium increases hardness and compressive strength in ductile iron. Optimal austempering time is 2 hours for V-containing iron. |
| [6] | Austempered at different temperatures: 240 °C, 280 °C, 320 °C. Analyzed microstructure, mechanical properties, and wear resistance. | The morphology of bainite changes from needle to featheriness with increasing austempered temperature. The optimal austempered temperature is 280 °C, resulting in improved impact toughness and wear resistance |
| [7] | The research aims to study the influence of austempering temperature on the microstructure and properties of austempered ductile iron with carbides, specifically focusing on how these factors affect abrasive resistance | The study demonstrated that the austempering temperature significantly affects the microstructure and properties of CADi, with higher temperatures leading to thicker and larger acicular ferrite, increased impact toughness, and decreased hardness. |
| [8] | The research aims to investigate the effect of austempering temperature on the microstructure of Carbodic Austempered Ductile Iron (CADi). | Also, CADi consists of bainite, retained austenite, and carbides, with nodular graphite dispersed throughout the matrix. |

The application of ADI in marine conditions has been made easier by the PMADI sample's superior mechanical capabilities and enhanced fracture toughness characteristics. The use of alloying materials, changes in critical heat treatment parameters such as austenitization, temperature and length of austempering, and the use of permanent moulds all contribute to the enhancement of ADI's fracture toughness qualities [9]. Studies that use unalloyed PMADI samples to look at how various austempering temperatures affect important mechanical properties as hardness, tensile strength, and fracture toughness over an extended length of time [10]. During austenitizing, the cast iron is essentially heated between 850 and 950 degrees Celsius. The eutectic grain development and homogeneity of components, the input structure of cast iron prior to quench hardening, graphite emissions, temperature and duration of warming, and the matrix's chemical structure all affect the austenitizing procedure. [11][12].

Ausferritic transformation research is the primary focus of ADI cast iron investigations. This is a very logical attitude given that the microstructure of the metal matrix mostly influences the development of the cast iron's high strength, excellent plasticity, and impact strength [13].

This work is focused on improving mechanical parameters, such as ultimate tensile strength and ductility, in Nickel-added Permanent Moulded Toughened Austempered Ductile Iron. This research is expected to provide key insights and approaches for developing next-generation steels.

The conditions of isothermal heat treatment, particularly the temperature of austenite's isothermal transition, can have a significant impact on the microstructure and mechanical characteristics of ADI. This research is being conducted to develop austempered alloyed ductile iron since huge rollers and automotive components are becoming more and more in need of high strength and hardness resistance qualities. In this specific case, the goal of the research is to determine the ideal austempering conditions based on microstructural control and evaluation of many attributes including tensile strength and hardness.

The impact of nickel addition on austempered ductile iron's microstructure, tensile strength, and wear resistance was investigated. Austenitization and austempering temperatures, austenitization and austempering durations, and cooling rate are some of the heat treatment process factors that are crucial to the ADI microstructure that is produced in ductile iron. Understanding the mechanical properties and wear characteristics of ausferritic cast irons under wear and friction behaviour environments was the primary goal of the investigations discussed in this article. Specifically: An analysis was conducted on the impact of the nickel concentration in ADI on wear resistance.

1. Materials and methods

The current study aims to evaluate the feasibility of improving the mechanical characteristics of ductile cast iron by heat treatment of a permanent mould casting. In the current investigation, two classes of ductile cast iron samples have been taken and processed through the Austempering procedure.

For the experimentation, an electric induction furnace with a 500 kg crucible furnace capacity was utilised. High-temperature metallic charge material, up to 1530°C. The samples that were received were machined and cut in accordance with EN1563.

The following table 2. Shows the details of sample designation along with Nickel percentage and Austenitization Temperature and time.

Table 2. Sample details with its composition and processing parameters

| S/No | Test Sample Designation | Nickel (%) | Austenitization Temperature | Austempering Temperatures | Austempering Time (Minutes) |
|------|-------------------------|------------|-----------------------------|---------------------------|-----------------------------|
| 1 | A (Unalloyed PMADI) | 0 | 780°C | 350°C | 150 |
| 2 | B | 1 | 780°C | 350°C | 150 |
| 3 | C | 2 | 780°C | 350°C | 150 |

To perform the austempering method, ductile cast iron specimens were created from the melt. There were two phases to the heat treatment process. The samples were first heated to an austenitization temperature of 780°C for 110 minutes. After that, they were immediately transferred to a salt bath that contained 50% NaNO₃ and 50% KNO₃ and was kept at a 350°C austempering temperature. The same techniques were used, based on earlier research by Wiczorek A.N [14].

The material was kept at this temperature for a variety of periods of time before being quenched to preserve the microstructure that existed at each stage of the transformation. The composition details of PMADI received samples are shown in Table 3.

Table 3. Final chemistry of the received sample (wt %)

| Sample | C | Si | Mn | S | P | Cr | Ni | Mo | Cu | Mg |
|--------|------|------|------|-------|-------|-------|------|------|-------|-------|
| A | 3.49 | 2.70 | 0.4 | 0.006 | 0.013 | 0.017 | 0 | 0.32 | 0.598 | 0.044 |
| B | 3.68 | 2.22 | 0.16 | 0.011 | 0.026 | 0.02 | 1.03 | 0.02 | 0.003 | 0.048 |
| C | 3.68 | 2.22 | 0.16 | 0.011 | 0.026 | 0.02 | 2.02 | 0.02 | 0.003 | 0.048 |

2. X – Ray Diffraction (XRD) Analysis

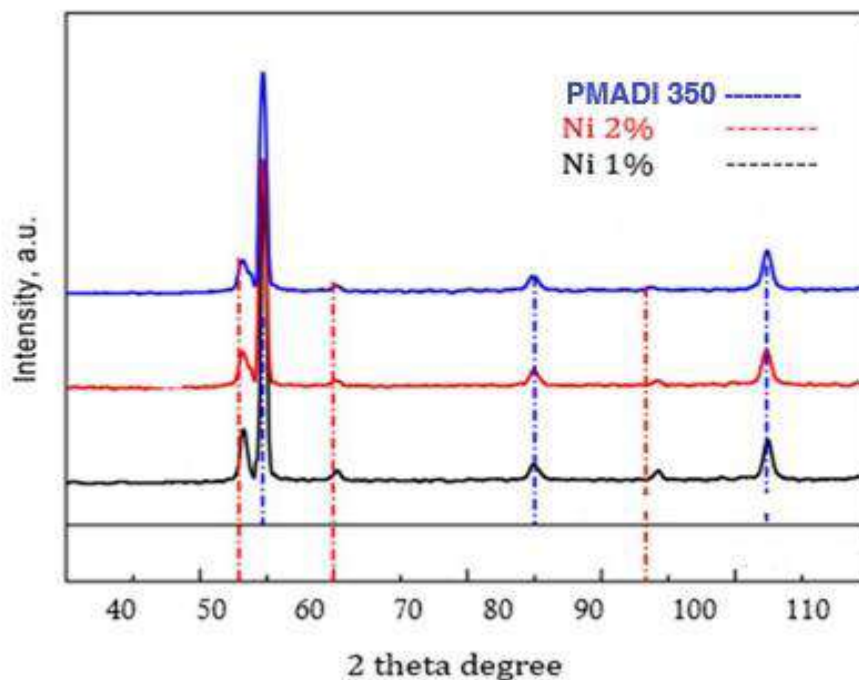


Fig. 1. - X – Ray Diffraction (XRD) Analysis of unalloyed PMADI, 1% Ni PMADI and 2%PMADI samples

The crystalline material's phase is identified using a common technique known as X – ray diffraction (XRD) analysis [9].

A Cu target diffractometer was used to perform XRD investigation on an un-alloyed PMADI sample at 40 kV and 25 mA (Bruker make). Scanning was performed over an angle range of 10° – 90° at a scanning speed of 1° min⁻¹. XRD

study of an unalloyed PMADI sample austempered for 150 minutes at 350°C. Peak locations and (shown in Fig 1) intensities of the 1 - (110) 44° - 45°, 2 - (200) 66° - 68°, and 4 - (211) 86° - 87° planes of BCC ferrite, and the 3 - (220) 78° - 79° and 5 - (311) 89° - 90° planes of FCC Austenite, were investigated. The volume fraction of retained austenite was calculated using the integrated intensities of distinct ferrite and austenite planes. The volume fraction of retained austenite was calculated based on the integrated intensity of the peaks of FCC-phase, as well as the peaks of BCC phase.

The existence of retained austenite was verified by X-ray analysis. Retained austenite is calculated using following equation (1) [15].

$$V\gamma = \frac{1.4I\gamma}{I\alpha + 1.4I\gamma} \quad (1)$$

where $V\gamma$ - retained austenite;

$I\gamma$ and $I\alpha$ are the integrated intensities of austenite and ferrite, respectively.

3. Microstructure of Nodular and cast Ductile Iron

Figure 2 shows the microscope image details of the as-cast Nodular Iron and Nickel alloyed ADI and verifies the presence of graphite in the form of nodules. The figure 2 microstructure demonstrates that the isothermal shift at 350°C is to blame for this. The amount of austenite that has been kept is at 150 minutes. When chilled to room temperature, austenitic portions that are not sufficiently stabilised with carbon transform into martensite. As the treatment duration rises, more austenite is isothermally converted to acicular ferrite, which enriches the residual austenite due to carbon rejection. Cooling the austenite to room temperature has no effect on it but the effect of carbon rejection on stabilising the residual austenite becomes much greater. Samples that were subjected to an austempering temperature of 350°C for 150 minutes also exhibited improved residual austenite.

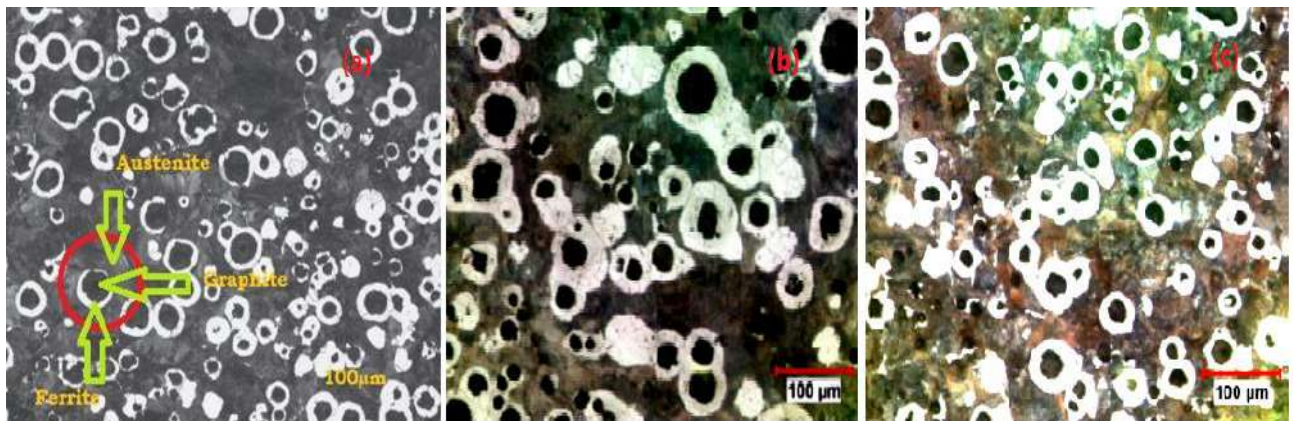


Fig. 2. - (a-c) As-cast structure of 0 %, 1%, and 2% Nickel bearing permanent molded ductile iron



Fig. 3. - (a-c) Micrographs of PMADI 350– Austenizing temperature – 900°C for 120 minutes, Austempering temperature – 350°C for 150 minutes time duration

Fig 3 a - c clearly shows the micrographs of as cast PMADI samples alloyed with 0%, 1%, and 2% Nickel in condition – etched using 2% Nital solution. The graphite nodules are surrounded by ferrite in the form of Bull’s eye structure along with pearlite and iron carbide (Fe₃C) and the same was confirmed from the photomicrograph of Nickel

alloyed PMADI samples. From the observation, It is clear that, the pearlite content in the matrix increased and the ferrite content decreased as the nickel additions were increased. Nickel is referred to as an austenite-stabilizing element because it shifts the pearlite knee to greater time intervals in solid state transformation. Traces of iron carbide were seen in all the samples. Also clearly displays the microstructural observations such as nodule size of 4-7µm for 0% PMADI sample, 6-8 µm for 1% Ni-PMADI and 7-9 µm for 2% Ni-PMADI samples (Nodule Count of 128, 150 and 180 number/mm² for PMADI , 1% Ni-PMADI and 2% Ni-PMADI samples).Fig 3 (c) displays the photomicrograph of PMADI - 350 sample austempered at 350°C for 150 minutes With a maximum degree of retained austenite of 26%, duration validates the presence of a feathery dispersion of Bainite. Pearlite and cementite were converted to ferrite and graphite by low temperature graphitisation annealing; otherwise, their presence would significantly enhance the iron's brittleness, which has a negative effect on toughness.

5. Hardness Test

A Brinell hardness tester was used to evaluate both an unalloyed PMADI sample and PMADI that had been alloyed with nickel. Three mean values were obtained. A 5 mm diameter ball is utilised as the indenter, 250 kgf is applied as the load. The BHN values of tested samples are shown in figure 4.

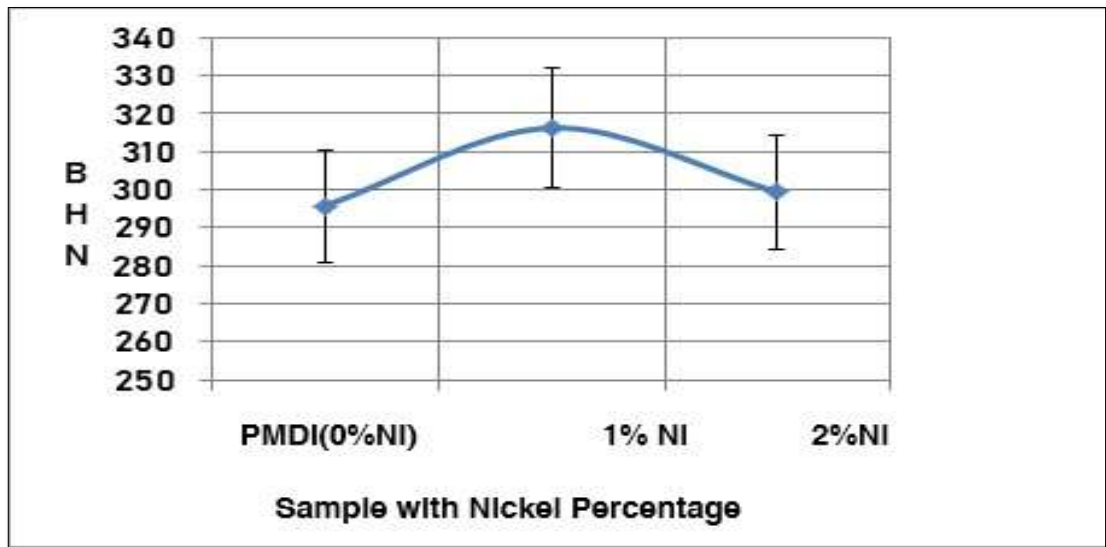


Fig. 4. - Results of Hardness Test

The results of the hardness test showed that the PMADI's 1% Ni PMADI sample had the highest hardness, while the unalloyed PMADI sample had the lowest. This might very well be explained by the ideal composition of ausferrite. As the austempering temperatures rose, the extent of retained austenite increased while the amount of acicular ferrite decreased gradually. Hardness was decreased by both ferrite coarsening and retained austenite [16]. As the temperature of austempering increases, there is a corresponding rise in the quantity of austenite and microstructure coarsening. As the proportion of the softer austenite phase increases, the hardness of the austempered ductile irons falls. Furthermore, the coarsening of the grain also results in a decrease in hardness [17].

6. Tensile Test

The tensile parameters of PMADI sample include ultimate tensile strength (UTS), yield strength and percentage (percent) elongation, which were tested using a computerised FIE manufacture universal testing equipment (UTM). The average readings of three samples in PAMDI-350, 1%Ni and 2%Ni specimens were taken and recorded. The evaluated details are shown in table 4.

Table 4. Tensile strength results of Unalloyed P PMADI, 1%Ni PMADI and 2%PMADI

| | | UTS MPa | % Elongation | YS MPa |
|-------|----------|---------|--------------|--------|
| PMADI | Sample 1 | 986.21 | 4.81 | 805.12 |

| | | | | |
|--------|----------|---------|------|---------|
| | Sample 2 | 983.54 | 4.76 | 805.08 |
| | Sample 3 | 969.47 | 4.71 | 806.41 |
| A 1%Ni | Sample 1 | 995.65 | 3.88 | 812.24 |
| | Sample 2 | 989.38 | 3.71 | 814.36 |
| | Sample 3 | 981.19 | 3.82 | 818.27 |
| B 2%Ni | Sample 1 | 1089.32 | 5.11 | 1084.16 |
| | Sample 2 | 1088.91 | 4.92 | 1084.12 |
| | Sample 3 | 1087.63 | 5.16 | 1084.18 |

The Ultimate tensile strength, Yield strength and % elongation results are clearly displayed in table 4. The highest tensile strength of 1089.32 MPa was discovered to be for 2% Nickel alloyed PMADI sample and the minimum of 969.47 MPa for 0% Nickel alloyed PMADI sample. The highest % elongation of 5.16% was found in 2%Ni PMADI samples and lowest % elongation of 3.82% discovered in 1% Ni PMADI Samples.

Finer acicular ferrite is found at 350°C. Tensile strength is higher at 350°C as a result. The microstructure confirms that acicular ferrite increases at austempering temperature, and the tensile strength also declines. Tensile strength drops with increasing ferrite cell size. Similar trend were witnessed in the work of Dakre et al., [18].

The high tensile elongation of these is attributed largely to the substantial volume percentage of residual austenite than to martensite production during plastic deformation [19].

According to research findings, ductility and toughness increase with rising austempering temperatures, whereas ultimate tensile strength (UTS) and hardness decrease. The ideal temperature is found to be 350°C. When the 150-minute austempering period is taken into account, the amount of retained austenite stabilises in each sample, with the PMADI 350 sample having the highest amount. The importance of nickel as an alloying element helps to stabilise the austenite phase and reinforce the Bainitic matrix. An austenite and ferrite-based ausferrite matrix is created during the austempering process from the pearlite matrix. The mechanical characteristics were impacted by the temperature that was austere [20]. Elevating the retained austenite and decreasing the acicular ferrite might be the cause. The area proportion of retained austenite increased and acicular ferrite decreased when a greater austempering temperature was applied, according to earlier studies [21].

7. Scanning electron microscope

Figure 5 show the tensile fracture surface of the broken specimen which is witnessed under scanning electron microscope (SEM; Model: JSM-7001F). As cast samples show brittle fracture. Intergranular fracture is viewed under the SEM. The fracture surface has a “rock candy” appearance that reveals the shapes of the individual grains. Thus, the fracture takes place between the grains [21].

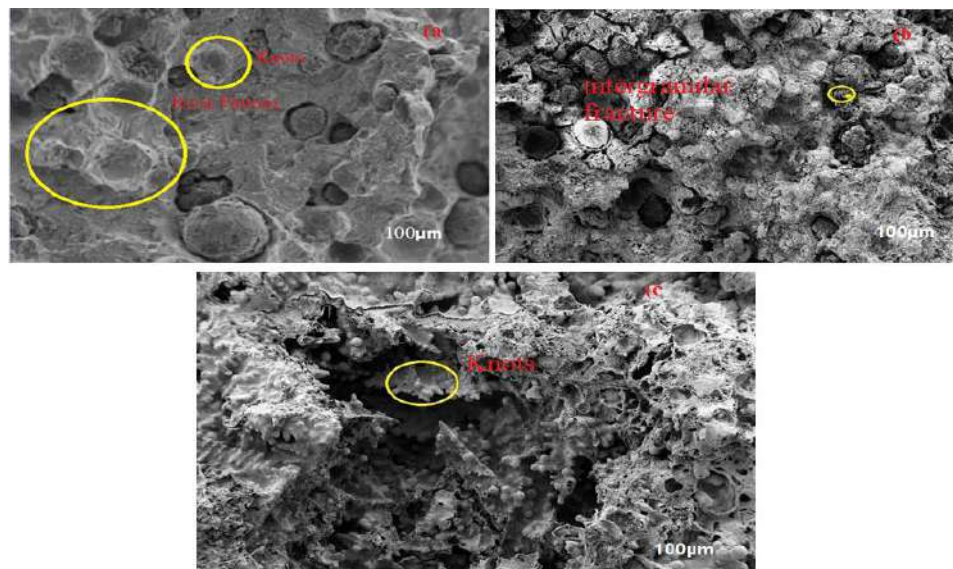


Fig.5. - (a) SEM photomicrograph of fractured Un alloyed PMADI samples; (b) PMADI samples with 1% Nickel content; (c) PMADI samples with 2% Nickel content, austempered at 350°C for 150 minutes

Mix type i.e. ductile as well as brittle fracture mode is observed in austempered samples. A significant number of dimples, a sign of ductile fracture, were seen in the nickel-alloyed samples. The samples showed unmistakable signs of dimple fracture between the nodular ligaments. Also, several of the nodules showed signs of graphite particle breakage. Additionally, nickel alloyed samples showed ductile void coalescence followed by de cohesion. Figure 5c illustrates that

the 2.0% Ni PMADI samples showed ductile fracture because of the many knots present and the resulting significant elongation prior to fracture.

As per Ravishankar et al., [22] The samples that underwent lengthier austempering durations in the first phase, such as 60 minutes, had fine austenite that was maintained as thin films between ferrite sheaths, together with a relatively high carbon content. Consequently, the austenite that remained exhibited exceptional stability. The constancy of the retained austenite is a key factor contributing to the enhanced fracture toughness observed in samples subjected to either low temperature conventional austempering or prolonged durations of first step austempering in the two-step austempering process.

The specimens displayed brittle fracture characteristics due to cleavage ductile cracks throughout the surfaces. This is characterised by the formation of relatively large cavity widths in relation to the sizes of the nodules. The stress-induced martensitic transition seems to have contributed to the development of brittle fractures, resulting in a decrease in fracture toughness [23].

The effective utilisation of permanent moulds has also contributed to the enhancement of nodule morphological characteristics, including counts and size [24]. The efficient dispersion of Bainite inside the ultimate microstructure is also accountable for the enhancement of the maximum load-bearing capacity.

The hardness and tensile properties of Al/Si₃N₄ nanocomposites were examined by Md Tanwir Alam et al. The study observed that the physical characteristics of nanocomposites, such as density and porosity, exhibited a linear improvement similarly in this work addition of nickel in ADI increased mechanical properties [25]. It is commonly known that producing dynamically loaded parts requires a combination of high ductility and high strength values. The effects of nickel alloying vary depending on the form of cast iron, and several researchers have studied this material to see how little nickel additions influenced different forms of cast iron alloys. These outcomes highlight the importance of the solidification cooling rate in ductile iron, which could affect the final material's hardness and tensile strength. The outcomes evidently show that all attributes increase with nickel content up to 2%. This is visible in the number of nodules and the amount of retained austenite in the matrix. Nickel, as a solute element dissolved in the matrix, interacts with dislocations and elements separated near dislocations to create Cottrell atmosphere, which may be the cause of this.

8. Wear analysis

Wear tests were performed out using a pin-on-disc wear testing machine with cylindrical samples of 10 mm diameter and 20 mm height against a 60 mm hardened M2 steel ring. This study examined wear rate as a function of sliding velocity while maintaining a constant applied load of 50 N. Track radius of 60 mm was taken into account for sliding velocities of 1.57 m/s, 2.57 m/s, and 3.77 m/s.

The specimen's surface temperature and characteristics may be impacted by the change in the generation of heat caused by the speed increase. As the sliding speed rises, higher surface temperatures may accelerate oxidation and alter the wear process to a softer wear. It was discovered that specimens tested at greater loads had a higher wear factor. Although martensite is known to be brittle and prone to breaking, the change of austenite to martensite strengthens the surface, enabling it to sustain the high contact load [26].

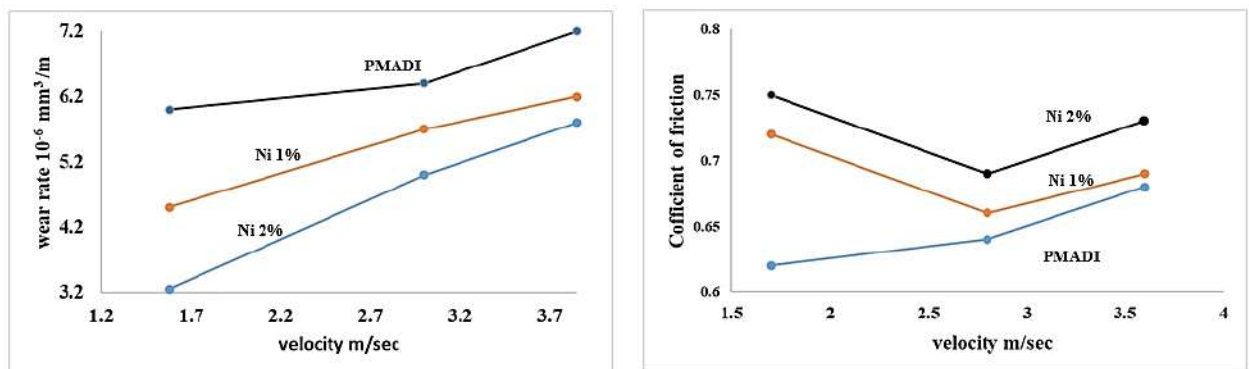


Fig. 6. - (a) wear rate as a function of sliding speed; (b) coefficient of friction as a function of sliding speed

The main factors that influence of wear rate are temperature, hardness, velocity, and environmental factors. Because temperature increases together with velocity and load, temperature has a significant impact on wear rate.

COF rises as load and sliding velocity increase (fig 6.). The contact surfaces heat up while sliding as a result of friction, and wear particles and hard asperities plough the flat areas of the sliding surfaces. Because there is not enough time for heat dispersion, the frictional heating occurs continuously when the counter surface is moving relative to the surface. The sliding action rises with the material's flow ability, which also lowers the coefficient of friction by reducing frictional heating.

The COF marginally rises in each composition as a result of mass loss decreasing and Ni% increasing, while the velocity and load increase as mass loss increases. The physical phenomena of wear at low austempering temperatures were adhesive and abrasive wear, whereas at high austempering temperatures it was adhesive wear through the creation of an intermediate or mixed layer, as the friction coefficient rose with rising austempering temperature.

In addition to lowering the wear rate and lowering the coefficient of friction, the presence of a tribo mechanically mixed layer changes the wear way from two body to three body wear. The amount of metal removed during the wear test decreased significantly as a result. Without adding Ni, it is suggested that there is more worn debris, which accelerates the rate of wear. In another way, the presence of hard, pulled-out Ni particles on the steel disc creates an obstruction and changes the adhesive wear mode into an abrasive one. A lower number of cracks and scratches is associated with optimal wear rates.

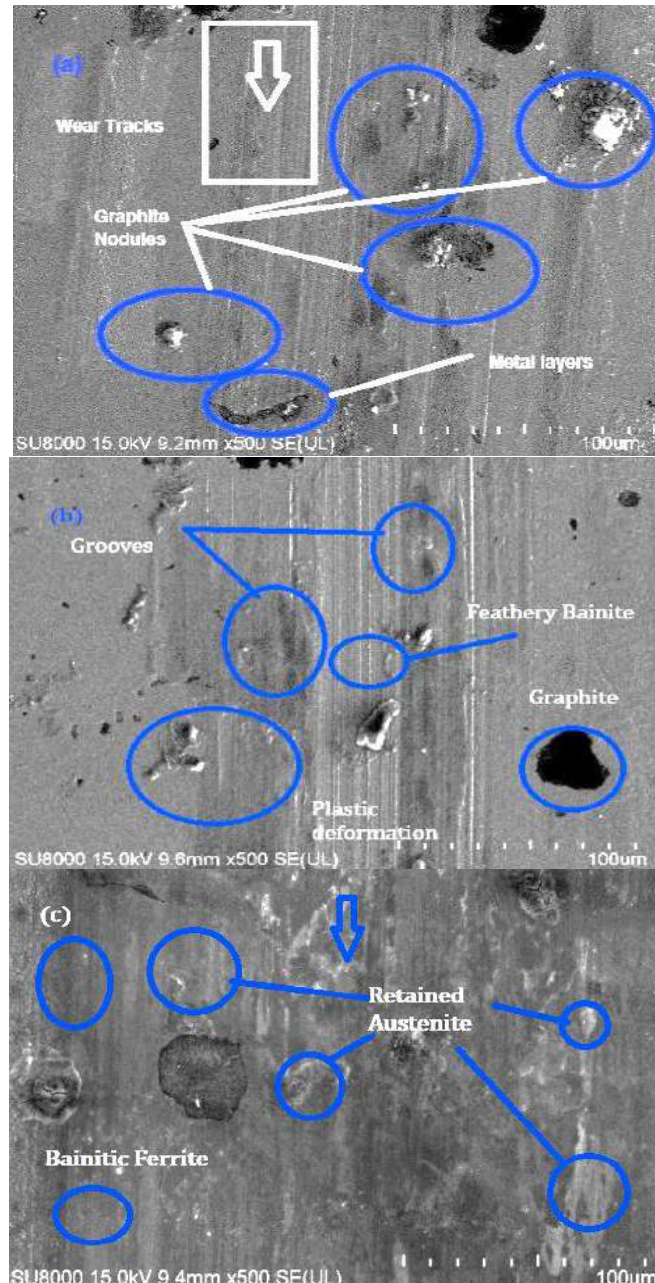


Fig. 7. - Worn morphology of the ADI tested samples. (a) PMADI (b) Ni 1% added ADI and (c) Ni 2% added ADI sample

This micrograph revealed a smooth surface with some wear traces due to ductility. There was a smearing of the material across the nodules, indicating a more ductile structure, although the black one on the worn surface was graphite-shaped and noduled. Because graphite nodules function as porosity flaws, this micrograph also showed that certain graphites had been distorted from the pin surface and altered shape.

From Figure 7. It has been demonstrated that the depth of the scratches seemed to increase as the abrasive particles approached the leading edge of the graphite nodule. Additionally, edge chipping was observed in this case along with plastic deformation. Owing to a greater number of grooves and microchips created by plastic deformation at the contact areas between the sample and abrasive particles, Fig. b shows a rougher surface. Because the shape of the particle and the increase in ductility affected the penetration depth, it varied. In this instance, wear caused the majority of the groove volume to be lost without staying at the groove sides.

Conclusions

The following are the primary conclusions drawn from this investigation:

By adding nickel by weight 1% and 2%, respectively, to austempered ductile cast iron, the material's mechanical properties such as its hardness and tensile strength were enhanced. The iron was then austempering at a variety of temperatures, including 350 °C. This was a result of the specific combination of alloying elements utilised and a well-formed microstructure mostly composed of acicular ferrite and austenite.

The PMADI sample, alloyed with 2 percent Nickel, has superior yield strength qualities. This may be attributed to the effective utilisation of permanent moulds, a greater volume percentage of retained austenite, and the dispersion of Bainite.

The precise combination of heat treatment parameters, which includes ideal nodule count and size, effective austenite management, and proper Bainite dispersion, leads to an enhancement in both tensile and hardness properties for both the PMADI – 350 samples and the nickel alloyed PMADI samples. Considering these factors, PMADI samples might be utilised as a substitute material for applications that need enhanced yield strength qualities. The 2 % Nickel carrying PMADI sample exhibited a 12.36% improvement in its highest ultimate tensile strength compared to the unalloyed PMADI sample when both were subjected to equal austempering conditions.

The addition of 1 percent Nickel to a PMADI sample resulted in a 6.92% increase in its maximum hardness strength when compared to an unalloyed PMADI sample that underwent comparable austempering conditions. Severe adhesive wear, plastic deformation, and surface delamination were the most common wear processes identified on worn surfaces at low loads in both materials. The enhanced wear resistance of ADI under normal loads was connected with the deformation-induced transition of austenite to martensite and the resulting increase in hardness. Nickel presence of 2 wt% in ADI strengthened the material's hardness and wear resistance at low loads.

Compared to other alloys, ADI alloys, when appropriately heat-treated, have the ability to replace more expensive alloys in the construction of industrial products like as rolling mills. This substitution can result in cost savings.

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