

Описано результати досліджень можливостей підвищення механічних властивостей чавунів з кулястим графітом марки FCD-50 шляхом комбінованої термічної обробки. Запропоновано схему багатоступінчастої термічної обробки, характерною особливістю якої є операція охолодження у рідкому азоті. Використання такої схеми дозволяє отримати підвищені характеристики твердості HRC. Дослідженням мікроструктури чавунів, оброблених за такою схемою, доведена ефективність криогенного етапу термічної обробки та визначено її раціональні режими.

Ключові слова: FCD-50, чавун з кулястим графітом, термічна обробка, криогенна обробка, твердість, мікроструктура

Описаны результаты исследований возможностей повышения механических свойств чугунов с шаровидным графитом марки FCD-50 путем комбинированной термической обработки. Предложена схема многоступенчатой термической обработки, характерной особенностью которой является операция охлаждения в жидком азоте. Использование такой схемы позволяет получить повышенные характеристики твердости HRC. Исследованием микроструктуры чугунов, обработанных по такой схеме, доказана эффективность криогенного этапа термической обработки и определены ее рациональные режимы.

Ключевые слова: FCD-50, чугун с шаровидным графитом, термическая обработка, криогенная обработка, твердость, микроструктура

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EFFECT OF HEAT TREATMENT AND CRYOGENICS ON HARDNESS OF DUCTILE CAST IRON MICROSTRUCTURE (FCD-50)

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1. Introduction

Ductile cast iron, also known as nodular cast iron, spheroidal graphite iron (S.G. iron) and spherulitic iron, has the best mechanical properties when compared to other types of cast iron. When compared to steel, however, the mechanical properties of cast iron fall short. For both motor vehicles and production machinery, component-component machines have been developed as indicated by field data. The components of these machines that contain cast iron graphite round include gears, shafts, piston rings, and cylinder liners and heads. Similarly, when agricultural lands and plantations are transformed into residential areas and multi-story buildings, the construction requires a significant amount of reinforcing steel, which is the predominant role of steel in the construction of buildings and housing. Improved ductile cast iron can be used as a steel partner to meet the need for reinforcing steel.

2. Literature review and problem statement

This study is supported by several previous studies, including a two-step austempering process, the process is carried out by heating at a temperature of 900 °C, holding for 60 minutes. All specimens were cooled at 260 °C for 10 minutes. In the subsequent process, the specimens were reheated at different temperatures of 280 °C, 310 °C and 340 °C held respectively for 60 minutes and 120 minutes. The highest yield for mechanical properties (toughness) is achieved when heating at a temperature of 280 °C, while the highest tensile strength is obtained at a heating temperature of 340 °C with each holding for 60 minutes [1]. The nodular cast iron (NCI) was investigated according to its mechanical properties. The result of austempering two-step heat treatment on mechanical properties obtained a higher value than one step. The optimum value of mechanical properties occurred at tensile strength and hardness at 340 °C austempered temperature with a holding time

of 60 min and followed by a lower austempered temperature, 310 °C, and 280 °C. The impact toughness value decreased at 340 °C austempered temperature with a 60-minute holding time [2]. The purpose of this study was to investigate the effect of cryogenic, martemper and temperament cooling and cryogenic cooling directly on hardness and cast iron microorganisms (FC-20). The results of this trial show that the indirect test of FC-20 (As-cast) shows a decrease in hardness while the FC-20 (AS-cast) test results directly cool the liquid nitrogen with increasing hardness. The mechanical properties of As-Cast, its hardness is 86.65 HRB on the indirect treatment of decreased hardness. Indirect hardness treatment increased from 123.6 HRB to 126.6 HRB. So if calculated carefully, there is an increase >40 % for hardness [3]. The research with Cryogenic and Temperature treatment was done on carbide chisel at AL-6061. The purpose of this study is to determine the wear and tear that occurs both on cutting chisel of As-Cast and cut chisel of modifications. The results of this research are: the deeper the cutting result, the worsening edge of cutting chisel is getting bigger, the result of the cutting tool (carbide chisel) from the Cryogenic treatment results, better in wear resistance. Similarly, carbide chains with cryogenic + temper treatment are also better resulted in wear resistance compared to As-Cast carbide chisels, the buildup of grains of AL-6061 is thinner than that of As-Cast carbide [4]. In this study, the steel was processed by an austempering method with a temperature of 325 °C and produced specimen with high toughness as for its microstructure is bainitic perlite, this research is assisted by observation of crystallography, TEM, and SEM [5]. A study of Austen ductile iron (ADI) was carried out, in which the microstructure was studied. In austempered temperatures, the results of this study are as-cast microstructure changes that result in improved crack propagation performance due to the reduction of eutectic carbide and the relatively high amount of austenite due to heat treatment [6]. The specimens studied were Austempered Austen iron with the austempering process at 450 °C, from which the coating process proceeded and produced tensile properties and increased fatigue properties [7]. This study resulted in austempering time on optimum mechanical properties of austempered materials at 315 and 350 °C of 240 and 180 min. Further results are austempering at 350 °C, compared with austempering at 315 °C, obtaining higher ductility and less toughness and strength and hardness. The best mechanical properties are at the bottom of block Y [8]. In this study, we discussed the influence of chemical elements on heat treatment on the Austempered Ductile Iron (ADI) specimen and also discussed the effect of the initial casting quality on the structure and properties of ADI pourers [9]. In this study, we discuss the tensile properties at room temperature, as well as the impact energy to break at room temperature, -20 °C and -40 °C, measured. The results of a multivariate analysis performed on the data emphasize the nature of the ferritic matrix [10]. While this research solves the improvement of mechanical properties of FCD-50.

3. The aim and objectives of the study

The aim of the research is to improve the mechanical properties of FCD-50.

To accomplish the set aim, the following tasks were set:

1. FCD-50 is processed by austemper cryogenic temper ductile iron method.
2. FCD-50 is processed by heat treatment (600–900 °C).

4. Materials and Methods

The aim of this study was to improve the mechanical properties of ductile cast iron (FCD-50). The test equipment was prepared in several laboratories and included 2 electric kitchen units, 2 liquid nitrogen containers and some supporting equipment. To determine whether the mechanical properties of the ductile cast iron specimens increased after the ACTDI process, the researchers measured hardness and observed the microstructures of the samples [6]. The ACTDI process resulted in increased hardness and microstructure changes in ductile cast iron (FCD-50) as listed in the hardness data table (Table 1).

Cast iron is included in the ferrous metals, which are used widely in the automotive industry. The properties of cast iron include better vibration dampers of steel, machining capability, wear resistance and hardness [7]. In addition, during the formation process cast iron has advantages in terms of depreciation. Shrinkage for cast iron is 0.5–2.0 %, whereas for steel it is 3.0–5.0 %. In addition, cast iron is much less expensive than steel. Until now cast iron can be classified into five.

This type of cast iron is produced during the casting process with rapid cooling, and its microstructure is composed primarily of cementite (Fe₃C). It is, therefore, very fragile and cannot be applied except as raw material for malleable cast iron with a small load.

This type of cast iron is produced by casting with metal melting using the cupula furnace, and its characteristics are better than those of white cast iron because the microstructure is different. The microstructure contains graphite, perlite and perrite. One characteristic of the microstructure in cast iron that can weaken it and reduce tensile strength is the flake shape of the graphite, which makes cast iron brittle. Despite these weaknesses, however, cast iron of this type still has advantages, including (1) a high damping capacity (damping vibration), (2) the capacity to enter into the cavity in a complicated form at the time of casting, (3) the relatively lower price, (4) ease of machining, (5) compressive strength (which is higher than its tensile strength), (6) ability to withstand heat up to 4,000 °C, (7) better resistance to corrosion compared to ordinary steel constructions, (8) low shrinkage (0.5–2 %), and (9) ability to withstand wear (friction), because graphite can serve as a lubricant.

This type of cast iron has better quality than grey cast iron. This type of cast iron is produced by the heat treatment of the raw materials of white cast iron, and tensile strength can reach 35 kg/mm². The microstructure consists of perlite, ferrite and graphite. The graphite is in the form of graphite-like cotton, which has mechanical properties that allow this type of cast iron to be forged or formed [8].

This type of cast iron falls between grey iron castings and cast iron with round graphite. Graphite in compact cast iron has its mechanical properties located between gray and nodular cast iron. Its heat conductivity is better than that of grey iron but worse than that of ductile cast iron. Compacted graphite iron (C.G. iron) initially was developed as a heat-resistant material for use in ingot moulds and vehicle brake components before being used for other structures, such as gear pumps and eccentric gears.

Ductile cast iron originally was developed in 1948 by the British Research Association from a casting process with the addition of cerium and other elements. Though this cast iron is known in Japan and America as ductile

cast iron (or ductile iron), it is known in Indonesia as round graphite cast iron (nodular). The microstructure of this cast iron consists of perlite, ferrite and graphite. In this cast iron, the graphite is round (nodular), which has a very small degree of stress concentration. As a result, this cast iron is comparatively strong.

Another term for this cast iron is spheroidal graphite iron. Compared to other forms of cast iron, ductile cast iron is superior in terms of ductility, corrosion resistance and heat resistance; therefore, this type of cast iron is used for various purposes in structures such as pipes, mould rollers and furnace components as well as for civil engineering construction [9]. Because the graphite is solid spheroidal (compact), its strength and toughness are greater than that of cast iron with flake-shaped graphite (grey iron) – hence the name ductile cast iron. Unlike malleable cast iron, this spheroid-shaped graphite is obtained directly from the foundry, not from a hot-selling process. In addition, the spheroid is more round and compact than the tempered carbon in malleable cast iron. The total carbon content of cast iron is the same as that of grey iron, and the spheroid-shaped graphite particles (nodule) appear at the time of melting because of the presence of a small number of nodule-forming elements, usually magnesium or cerium. These elements are inserted into the iron fluid of the ladle just before casting.

One product of nodular cast iron is FCD-50, which is the focus of this study on improving mechanical qualities using the ACTDI method. The FCD classifications include numbers 37, 40, 45, 50, 60, 70 and 80 (ASTM). These associated numbers indicate the magnitude of the tensile strength of nodular cast iron. For instance, FCD-50 has a tensile strength of 50 kg/mm². In other words, a piece of nodular cast iron with an area of 1 mm² is capable of withstanding a maximum tensile load of 50 kg. This study of FCD-50 as an as-cast will further the research conducted [10] using the Austen ductile iron (ADI) method for FCD-60. In the present study, the method used is ACTDI (Table 1).

Table 1
Types of cast iron and graphite forms other than free graphite

No.	Types of Cast Iron	Graphite Forms
1	White cast iron	Shaped iron carbide
2	Grey cast iron	Shaped flake
3	Malleable cast iron	Shaped cotton wadding
4	Compact cast iron	Shaped thick flake
5	Ductile cast iron	Shaped nodule

Table 1 displays the known types of cast iron and the various forms of graphite. The graphite shapes of the various types of cast iron serve as indicators to distinguish among types of cast iron. In addition, the graphite form affects the mechanical properties of cast iron. For instance, the flake graphite form in grey cast iron produces a different tensile strength compared to the cotton graphite form in malleable cast iron, as does the nodular graphite shape of nodular cast iron.

Table 2 shows that the higher the number found in the ductile cast iron, the higher the tensile strength of the iron.

Table 2

Class divisions of nodular cast iron

Class	Class FCD	Tensile Strength kgf/mm ²	Strain (%)	Hardness (HB)
0	37	37	17	179 max
1	40	40	12	201 max
2	45	45	10	143–212
3	50	50	7	170–241
4	60	60	3	191–269
5	70	70	2	229–302
6	80	80	2	248–352

The mechanical properties of metals are traits that must be taken seriously by metal users. These properties depend not only on the chemical composition of the alloy but on its microstructure. Alloys with the same chemical composition can have different microstructures and, therefore, different mechanical properties. The properties are dependent on the processing (especially the heat treatment) that the alloy undergoes [11]. Heat treatment is a process of the heating and cooling applied to the metal/alloy in a solid state at a certain speed to obtain certain properties. Heat treatment is a powerful determinant of the nature of a metal/alloy product, but it cannot stand alone. This treatment should be viewed as part of a series of production processes. A similar heat-conducting process may produce different properties when the preceding and subsequent processes are different. The heat treatment process comprises several stages, starting with the heating of the material to a certain temperature. That material is held at the specified temperature for a while and then cooled at a certain speed (Fig. 1).

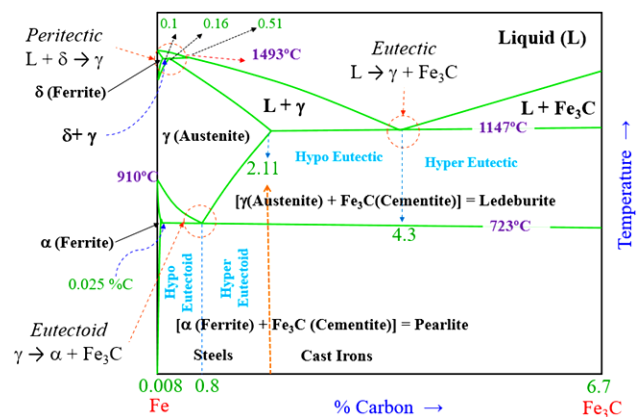


Fig. 1. Ferrous metals balance diagram (Fe–Fe₃C; ASM)

Processes on diagram Fe–Fe₃C

1. Peritectic Process: occurs at 1495 °C with the following process: liquid (0.53 % C)+δ (0.08 % C)=γ (0.18 % C). Carbon in a liquid condition with a content of 0.53 % C occurs as an alloy with iron delta (δ) in a liquid condition with a content of 0.08 % C. The reaction product is formed by gamma iron (δ) in a liquid condition with a carbon content of 0.18 % C.

2. Eutectic Process: occurs at 11,450 °C with the following process: liquid (4.3 % C)=γ (2.1 % C)+Fe₃C (6.67 %). Carbon in a liquid condition with a content of 4.3 % C is transformed into gamma iron (γ) with a 2.1 % C and carbide iron (cemented) content of 6.67 % C.

3. Eutectoid process: occurs at 7,230 °C with the following process: γ (0.83 % C) = α (0.02 % C) + Fe₃C (6.67 % C). Gamma iron (γ) with a content of 0.83 % C is transformed into alpha iron (α) with a content of 0.02 % C and into iron carbide (Fe₃C) with a content of 6.67 % C.

Information: γ (gamma iron) =
=austenite, α (alpha iron) = ferrite, δ (delta iron).

Austempering is a heat-conducting process developed directly from an isothermal transformation (I-T) diagram to obtain a structure that is entirely bainite. The austenitic temperatures for this process are the same as the austenitic temperatures in the annealing/hardening process; here, however, the cooling is done by quenching to temperatures above Ms and holding until the transformation to bainite is finished. A salt bath with a temperature of 200–425 °C is commonly used as a cooling medium; thus, the final structure is entirely bainite, and there is absolutely no martensite. The austempering results have high strength and hardness scores (RC 45–55) with high ductility and toughness (Fig. 2).

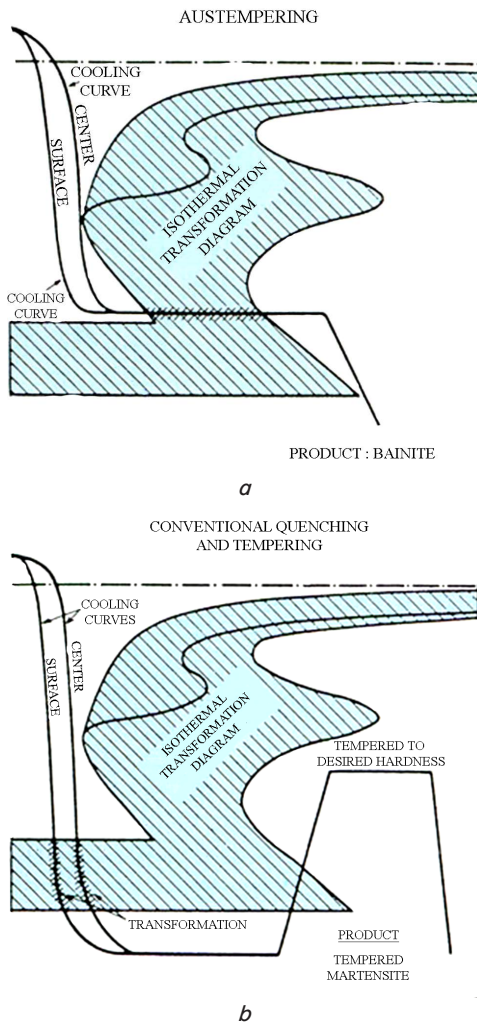


Fig. 2. The main signature: *a* – Transformation diagram with austempering cooling scheme; *b* – Quench and temper cooling scheme

The austempering process is illustrated schematically in contrast to the usual quench-and-temper process. Aus-

tempering no longer requires tempering after quenching. The final structure of the austempering process is bainite, whereas martensite is obtained from the quench-and-temper [12] (Fig. 3).

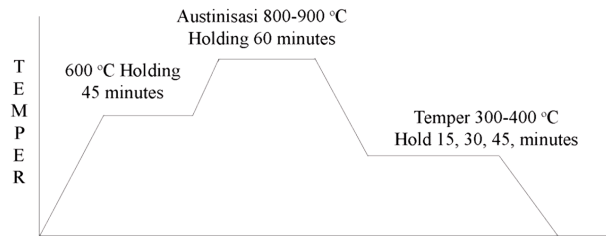


Fig. 3. Heating and cooling diagram VS Time on the Austemper Ductile Iron (ADI) Process

In the ADI process, the specimens undergoing heat treatment are preheated from room temperature to 600 °C (preheating) and held at this temperature to ensure the heat homogeneity of all samples. The specimens next undergo the austenisation process at a temperature between 800 and 900 °C and then are held at that temperature and subsequently cooled. Cooling occurs in a second electric kitchen at 300–400 °C. The austempering process is held holding after that cooled at room temperature [13].

Cryogenics is one of the freezing technologies that can improve the mechanical properties of the metal. This method of freezing (cooling) uses liquid nitrogen with temperatures below 0 °C. The process for obtaining liquid nitrogen involves compressing gas into a liquid form (e.g. nitrogen [N₂] and carbon dioxide [CO₂]). Liquid nitrogen has long been used to freeze organic materials for the storage and extraction of research materials in applied biology. Liquid carbon dioxide, in comparison, is used to fill fire extinguisher tubes. Liquid nitrogen has a boiling point of –195.8 °C, whereas liquid carbon dioxide boils at –57 °C. At higher temperatures than these, nitrogen and carbon dioxide are volatile gases, so generally liquid nitrogen and liquid carbon dioxide are at temperatures lower than their boiling points. With such cold temperatures, both liquid nitrogen and liquid carbon dioxide have the ability to freeze organic matter and are relatively more effective than ammonia or Freon coolants (Fig. 4, 5).



Fig. 4. Cryogenic installation



Fig. 5. Cryogenic container

Prior to conducting heat treatment and testing, the researchers gathered the raw material – ductile cast iron (FCD-50), made from castings with diameters of 22 mm and 320 mm. Some of the as-cast prepared served as tensile test specimens to ensure that the raw material was completely FCD-50. After the researchers determined that the as-cast was FCD-50, they cut the raw material to a length of 320 mm and then treated it with the ACTDI method, as shown in Fig. 1. The composition of the as-cast was determined using the ASTM E 415-08 and ASTM E 350-195 chemical tests with the following results: carbon (3.6%), silicon (2.6%), phosphorus (0.00413%), iron (92.64%), magnesium (0.30%) and manganese (0.27%). In Fig. 6, a, b, the line is the as-cast heating from 35–600 °C. The heating up to 600 °C was preheating, which ensured that the test objects did not crack when heated to 900 °C (austenisation process).

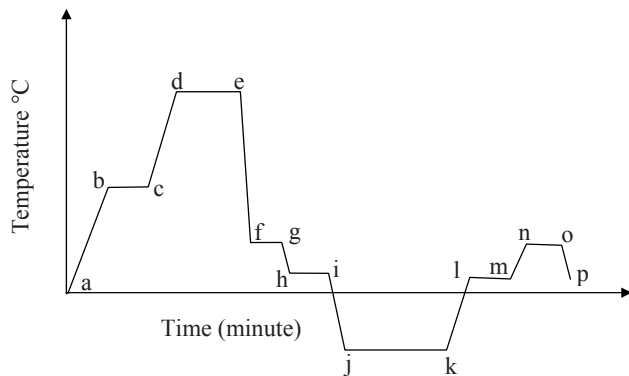


Fig. 6. Heating and cooling treatment diagram vs. time in ACTDI process

The as-cast was held at 600 °C for 45 minutes (b, c). Subsequently, the test objects were heated to 900 °C (c, d) and held at this temperature for 60 minutes (d, e). The researchers used two kitchen heaters in this heat treatment process. In the next stage, the test objects were removed from furnace 1 and immediately inserted in kitchen 2 at a temperature of 300 °C and held there for 30 minutes (e, g). When the test pieces were removed from kitchen 2, they were still at a temperature of 300 °C. They were then put into brine at a temperature of 70 °C and left to cool to room temperature (h, i). The specimen next was inserted into a tube of liquid nitrogen at a temperature of –195 °C with varying immersion times (12 hours, 24 hours, 72 hours) for each of the 4 specimens (i, j, k). Subsequently, all specimens were removed from the nitrogen tube and heated in an open bed to room temperature at 35 °C (l, m). The last step was tempering. After the heat and cooling treatment processes were completed, the specimens had formed for hardness, microstructure, SEM and other testing. To determine hardness, the researchers used the Rockwell Hardness Tester (HR-210 MR Mitutoyo). All specimens from the as-cast to the modified test object were formed according to the test to be performed. All specimens were tested.

5. Results of the study of hardness and microstructure of cast iron

Based on data of research results, there has been an increase in hardness on FCD-50 specimen with austemper, cryogenic and temper ductile iron. In Table 1, the

results of the hardness test are shown. The researchers tested four samples for each treatment for a total of 12 test samples (12 stems). The four rods of raw material (as-cast) were without treatment. Tables 3–5 show that there was an increase of hardness of 18.39 HRC. Be 21, 29 HRC, 21.67 HRC and 24.25 HRC. The highest increase in hardness value was for the ACTDI treatment at 12 hours of immersion, which yielded an increase of 5.86 HRC. When converted to HRB, the hardness increased significantly to 64.46 HRB.

Table 3

Mean hardness values for specimen 1

Treatment \ Specimen	I				
	1	2	3	4	R1
As-cast	19.40	19.30	15.80	19.90	18.60
Cooling 12 Hours	24.40	24.80	24.60	26.20	25.00
Cooling 24 Hours	19.00	20.30	21.20	20.70	20.30
Cooling 72 Hours	22.20	22.50	22.90	23.20	22.70

Table 4

Mean hardness values for specimen 2

Treatment \ Specimen	II				
	1	2	3	4	R2
As-cast	17.70	20.80	19.70	17.00	18.80
Cooling 12 Hours	20.10	22.20	22.40	23.00	21.93
Cooling 24 Hours	21.70	22.00	21.70	21.80	21.80
Cooling 72 Hours	20.00	20.00	20.30	20.40	20.18

Table 5

Mean hardness values for specimen 3

Treatment \ Specimen	III				
	1	2	3	4	R3
As-cast	16.50	18.80	18.50	17.30	17.78
Cooling 12 Hours	24.10	26.00	26.80	26.40	25.83
Cooling 24 Hours	21.90	21.80	21.70	21.70	21.78
Cooling 72 Hours	22.30	21.60	22.00	22.60	22.13

R1: The average hardness of the specimen; R2: the average hardness of the specimen; R3: the average hardness of the specimen; R4: total average hardness of specimen; as-cast: standard specimen, ACTDI (Fig. 7–11).

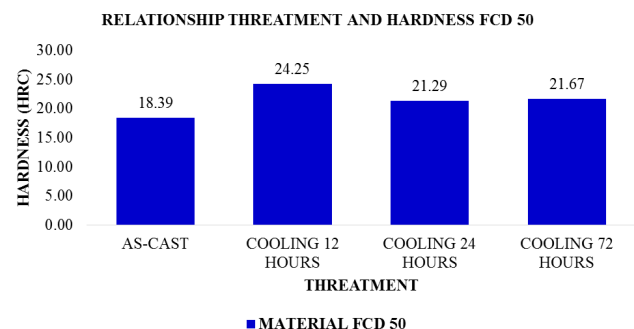


Fig. 7. Graph of average hardness in various conditions

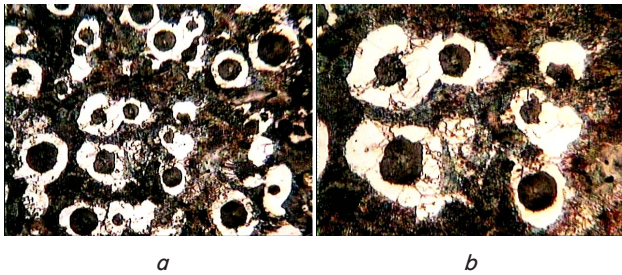


Fig. 8. Microstructure of As Cast FCD-50: *a* – $\times 200$; *b* – $\times 400$

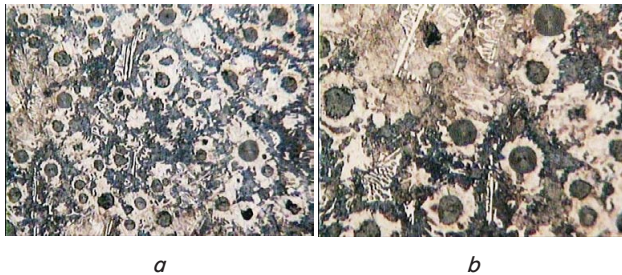


Fig. 9. Microstructure of Modification Immersion 12 hours FCD-50: *a* – $\times 200$; *b* – $\times 400$

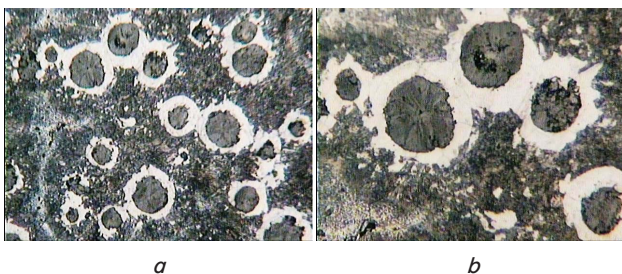


Fig. 10. Microstructure of Modification Immersion 24 hours FCD-50: *a* – $\times 200$; *b* – $\times 400$

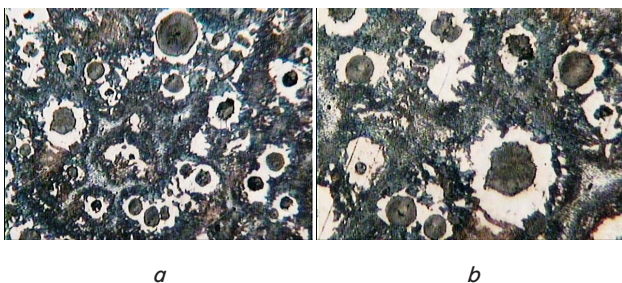


Fig. 11. Microstructure of Modification Immersion 72 hours FCD-50: *a* – $\times 200$; *b* – $\times 400$

6. Discussion of the effects of heat treatment on the mechanical properties of cast iron

The research is about improving the mechanical properties of Ductile Cast Iron (FCD-50) with Austemper Method, Cryogenic Temper Ductile Iron (ACTDI). In Fig. 7, the histogram shows the final results of the ACTDI treatment of FCD-50 specimens. This treatment resulted in an increase in hardness. The increase in hardness occurred after FCD-50 was austenitized and held for 1 hour. The austenite microstructure transformed into bainite after being austempered

and cooled in brine at 70 °C. In the cooling process from 900 °C, the specimens were held at a 300 °C temperature in the furnace 2 for 30 minutes. The change began to occur when the FCC crystals turned into BCT crystals with a bainite microstructure. When the specimens were removed from the furnace, they were still at 300 °C. They were then immersed in salt water at 70 °C. After reaching room temperature, the specimens were inserted into a tube containing liquid nitrogen at –195 °C. At the time of the immersion process, there was rapid cooling. The lower the cooling temperature, the more austenite transforms into martensite. The ACTDI process resulted in microstructures of martensite, graphite nodules, ferrite and perlite. In Fig. 8, *a*, *b*, FCD-50 microstructures are shown before modification. The microstructure comprises perlite, ferrite and graphite nodules, which distinguish FCD from other iron castings such as white cast iron, grey cast iron, compact cast iron and malleable cast iron based on the shape of the graphite. The average graphite microstructure has a round FCD shape that is due to the influence of elements contained therein, such as silicon and magnesium. This rounded graphite makes this FCD superior to cast iron.

Fig. 7 shows an increase in hardness resulting from the ACTDI process. This increase in hardness is caused by cooling with liquid nitrogen to –195 °C for 12 hours, 24 hours and 72 hours. In the process of cooling from 35 °C to –195 °C, the remainder of the austenite transforms into martensite. The addition of martensite as seen in the cryogenic cooling microstructure is shown in Fig. 8–11. Fig. 9 also shows an increase in hardness for all modified FCD-50. The highest hardness value of the modified FCD-50 in the ACTDI process occurred after 12 hours of immersion as smaller graphite nodules appear more and more densely and martensite becomes more neatly structured compared with a microstructure of the ACTDI process with a soaking time of 24 hours and an immersion of 72 hours. This study indicated that the optimal immersion time in liquid nitrogen was 12 hours. Because of a combination of microstructures for graphite nodules, the presence of martensite, perlite and ferrite in the ACTDI process is also optimized after 12 hours of soaking time, which resulted in the greatest increase in hardness compared to other processes.

The advantages of this research are:

- Improvement of the mechanical properties of ductile cast iron.
- Assistance of development in the automotive and manufacturing industries.

The disadvantages of this research are:

- Occurrence of cracks in specimens when in austempering long period of time.
- Occurrence of residual stress on the specimen when in austempering long period of time.

The results of this study can be useful to improve the quality of products of automotive components. The results of this study are usually applied to the automotive industry. This research is a continuation of previous research that is Improvement of mechanical properties of FC-25.

7. Conclusions

1. FCD-50 has the increased hardness when undergoing the ACTDI process. Through the ACTDI process, ductile cast iron immersed for 72 hours obtained a hardness of

21.67 HRC. This represents an increase when compared to standard specimens (as-cast), for which the standard specimen had a hardness of 18.39 HRC. Similarly, when compared to a modified specimen immersed for 24 hours, the specimen immersed for 72 hours obtained a greater hardness than the 24-hour immersed specimens.

2. Ductile cast iron (FCD-50) modifications soaked for 24 hours obtained a hardness of 21.29 HRC. When com-

pared to standard (as-cast) specimens, the samples soaked for 24 had an increase in hardness of 2.8 HRC. Ductile cast iron (FCD-50) modified through the ACTDI process with 12 hours of immersion obtained a hardness value 24.25 HRC. When compared to standard (as-cast) specimens and modified specimens soaked for 24 and 72 hours, the modified samples soaked for 12 hours had the highest hardness value.

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