Mechanical evaluation of the nodular gray cast iron with the application of thermal treatments

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Abstract- For this experimental study, commercial samples of nodular cast iron were used. Samples were subjected to different heat treatments such as quenching, tempering, and annealing. These treatments were carried out to obtain an increase in specific mechanical properties such as hardness, where the new values of this property were obtained. In this process, different laboratory tests as metallographic characterization, mechanical characterization by Brinell hardness, physical and chemical characterization by Optical microscopy, Scanning Electron Microscopy (SEM), X-ray Energy Dispersion Spectrometry (EDS) and X-ray Diffraction (XRD) were applied. The results obtained show that specific thermal treatments can be implemented together and different temperatures, achieving an increase of three times the value of the hardness for the manufacture of brake discs, able to withstand the thermal conditions caused by this type of component.

Keywords – Iron, Cars, thermal, treatments, hardness, mechanical properties.

I. INTRODUCTION

Disc brakes have full acceptance in the automotive industry because, compared to drum brakes, they absorb more kinetic energy when the braking mechanism is activated, which translates into a reduction in the time it will take for the vehicle to stop running. This phenomenon occurs because the devices or elements that make up the brake mechanism, in this case, the disc brakes, are in the open air; that is to say, the circulating air passes directly through the disc allowing higher heat dissipation [1].

The heat or thermal energy is caused when the elements of the disc brake (pads and disc) come into contact to transform the mechanical energy produced by the engine and the transfer through the different means that make up the automobile such as the box change, gimbal, differential and last transmitter to the wheels, thermal energy on the components of the brake, which rotates in conjunction with the rims [2].

The essential element in a braking system is the brake disc. This element is generally made of cast iron, but in some cases, it is made of composite materials such as reinforced carbon, ceramic matrix composites, and metal matrix composites. The brake material must have the following properties: a high coefficient of friction, impermeability, ability to withstand high temperatures (thermal stability), high wear resistance, flexibility, and adaptability to any surface.

The first appearance of nodular casting dates from the end of 1940 in England and later in the United States. Ductile iron was obtained by adding magnesium to the mixture present in the furnace. The formation of graphite nodules appeared during the solidification process, which is a significant discovery because a material with high ductility and higher hardness could be obtained directly from the casting, avoiding the use of heat treatments and, therefore, minimizing the production costs. This material belongs to the family of graphite castings that have higher ductility, property that gives excellent resistance to impacts, with excellent capacity to withstand deformations. It is an alloy with the highest elongation property obtained directly by fusion in its ferritic nodular grade. It can be bent, warped, and deformed without fracturing, making it ideal for use in the manufacture of vehicle parts exposed to thermal impacts [3].

In 2012, Canzar *et al.* [4] evaluated the fatigue-service life of nodular casting with four different microstructures, in which it was observed that the largest irregularly shaped nodules reduce fracture toughness and fatigue resistance. Also, the results showed that the pearlite phase does not strongly affect the fatigue life if its proportion does not exceed 10%. Catastrophic failures in devices made from cast iron, it is essential to take into account the influence of the geometric characteristics of graphite nodules (size, shape, and distribution of nodules) on the fatigue crack initiation. In addition to the form of graphite nodules, the mechanical properties of cast iron are determined by the metal matrix. Ferritic cast iron usually is soft and ductile, while the pearlitic matrix exhibits high strength and hardness and is prone to brittle fracture.

In 2011, Blaz Samec *et al.,* [5], carried out a work-related to nodular cast iron. They used railway disc brakes, with which they performed stress and cyclic fatigue tests where it was examined the microstructure of the material at ambient temperature, 300 °C and 400 °C. They also found a ferritic-pearlitic matrix, where it was observed that at 400 °C, the matrix of the material remains stable. Besides, tensile tests at ambient temperature present practically identical results, while at higher temperatures, there was a higher dispersion of results. Young's modulus remained stable at temperature changes, while stress testing and ultimate tensile strength decreased as temperature increased. Another study on the braking process consisted of the contact between the pad (brake pad) and the disc where it was demonstrated that, due to local intensive friction heating near a contact surface, the field of compression stresses, after the start of braking at some point the normal tensile stresses occur near the subsurface region. When these stresses exceed the tensile strength of the material, the onset of surface cracks is possible, causing failures that would be unforeseen by part of the driver of the automotive [6].

In 2016 Karan Dhir [7] demonstrated that the shape and geometry of the disc are directly related to the phenomenon of heat dissipation and the probability of overheating. This phenomenon was verified by simulating three different prototypes of disc brakes, obtaining; as a result, the maximum increase in temperature between the three discs from 21.85 °C to a maximum of 225.17 °C in the disc pad swept area.

On the other hand, the need arises to obtain a material with improved surface properties to withstand the conditions to which the disc is subjected during constant braking actions. Also, properties such as high hardness, and a metallographic structure with the presence of graphite nodules which give the material higher hardness are fundamental. Thus, this article presents the study carried out on the experimental set obtained from a disc brake used in the automotive industry, which is manufactured from nodular cast iron. This material exhibits adequate mechanical properties for its operation. However, these mechanisms suffer widespread failures, such as: cracking influenced by rapid temperature changes that weaken the disc material, bent discs, thickness variation, excessive wear, corrosion, among others. The temperature variations are presented when the brake is used. The brake pad and disc interact by friction to stop the vehicle, thus causing the accumulation of thermal energy coming from the mechanical energy absorption of the wheel; this excess of thermal energy affects the mechanical properties of the material. Therefore, alternatives such as heat treatments are sought to improve the mechanical properties of nodular cast iron [8].

In this study, different thermal treatments are developed on nodular grey cast iron to obtain a better experimental condition to improve the hardness on the surface and reduce the wear loss material under automotive use.

II. EXPERIMENTAL DETAILS

Nodular grey cast iron samples

Quadrate samples of nodular grey cast iron with 50 mm×50 mm×20 mm with a superficial hardness around 55 Rockwell-C were used in this study. The nominal chemical composition for the samples in (wt%) is C 3.2-3.60, Si 2.0-2.8, Mn 0.1-0.2, Mg 0.03-0.05, P 0.004-0.04, S 0.005-0.02, Cu <0.40 and balanced Iron obtained of [9][10]. In distinctive gray iron, it is generally found carbon as graphite nodules, adopting irregular shapes known as flakes. This graphite gives the grey color to the surfaces of the pieces elaborated with this material. Physical and mechanical properties, differ within wide intervals according to factors such as chemical composition, cooling speed after casting, size and thickness of parts, heat treatment, as well as microstructural parameters such as the nature of the matrix and the shape and size of the graphite flakes. The heat treatments applied are quenching, tempering, and annealing.

Thermal treatments

The use of the heat treatment of quenching a matrix of the martensitic structure is obtained, which is one of the constituents with higher hardness, optimal for the desired improvement in the nodular cast iron. Nodular iron or ductile iron can be hardened by heating above the critical transformation temperature, around 900 °C [11]. The cooling speed plays an essential role in the formation of the microstructure. Due to the fact water quenching exceeds the critical speed, distortions or cracks are formed since, in the process of transformation of the martensite, there is a notable expansion of the volume, generating internal forces and together with thermal contractions are the cause of the cracks. According to Yu Lajtin [12], the best process for quenching is obtained employing oil quenching. The hardness reached by the grey castings after applying the quenching reaches values between 48 to 55 Rockwell-C, changing the value with the quantity and size of graphite sheets; also, the wear resistance is much higher than those presented in high carbon steels.

The heat treatment of tempering is commonly used after applying a quenching since, after the quenching process, the steel parts are very hard and fragile, which often hinders the machining. Therefore, the tempering is applied to reduce hardness and resistance, while increasing toughness and minimizing the internal stresses of the material after quenching. But this does not mean that the tempering eliminates the properties acquired by the material when the quenching was applied, but it modifies them. The temperature range depends on the desired properties for the material. These properties can be hardness, toughness, wear resistance, among others. Table 1 shows the temperature ranges for applying the tempering according to the desired properties.

Table 1. Tempering temperature range.

TEMPERATURE RANGE (ºC)	ACQUIRED PROPERTIES
200 - 450	Moderate hardness and toughness
< 200	High hardness and wear resistance
> 450	High toughness

The above properties are obtained due to the different structures acquired by the matrix in pieces according to the temperature range applied. When a temperature lower than 200 °C and higher than 40 ºC, a black martensite type structure will be obtained, called this way, due to its black tonality. To the modification of its properties, internal stresses are eliminated. When the temperature range is between 200 °C and 450 °C, the martensite obtained in the quenching is transformed in ferrite, and any austenite present is converted into bainite. Also, a black zone is formed, and its mechanical properties are modified, where a strength higher than 200,000 Psi, as well as hardness between 40 and 60 Rockwell-C, and an increase in the ductility and low toughness are obtained. Finally, when tempering is applied in a range between 450 °C and 650 °C, an increase in cementite, and at the same time, there is an increase in the amount of ferrite, which will predominate in the material matrix [13].

The annealing heat treatment is, in many cases, the last one to be applied due to the properties it provides. When applied to grey or nodular castings, its hardness can be further reduced and, therefore, its machinability improved, also, to reduce stresses and increasing ductility and impact resistance. When annealing is applied to the castings, the carbon content in the matrix disappears, and only ferrite and graphite remain. The objective of annealing is to obtain better machinability, achieving a more significant softening of the material, but keeping high resistance values [12].

The base metal selected for this experimental study was obtained from a brake disc used in the automotive industry, which is manufactured from nodular cast iron, with a microstructure that has graphite nodules, which is characteristic of this material [14]. In this process of metallographic preparation, 2% Nital which is composed of 2 ml of nitric acid and 98 ml of ethyl alcohol, was used to attack the surface for one minute. Fig. 1a and Fig. 1b shows the comparison of the microstructure at 100X of the selected sample and the nodular cast iron.

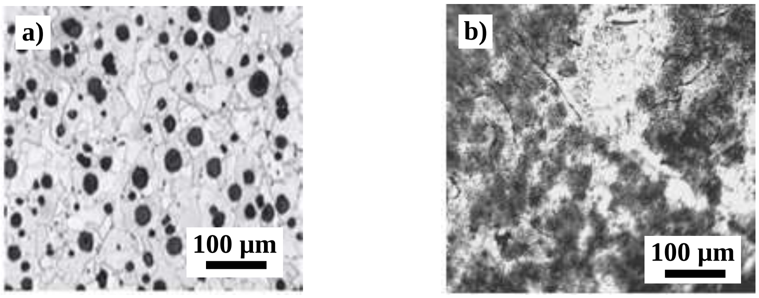

Figure 1. Microstructure comparison. a) Pearlite Nodular Foundry and b) Base material.

Experimental design

The heat treatments applied to the base material are quenching, tempering, and annealing. The application of these three treatments seeks to increase hardness, tensile, and compressive strength, eliminating internal stresses produced during the heating of the piece in the furnace.

Fig. 2 shows the methodology applied for the development of the research.

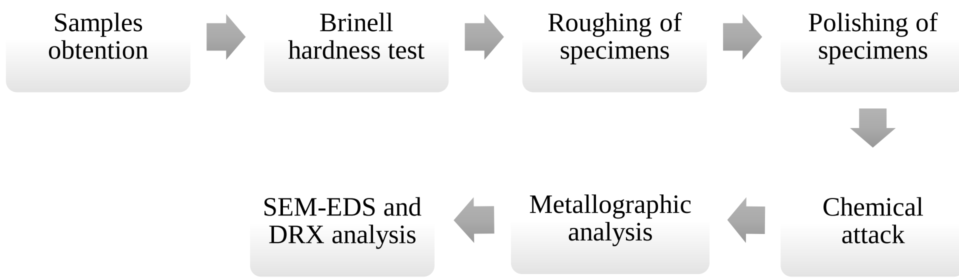

Figure 2. Processes for the development of this research.

Initially, nine samples were obtained from the brake disc selected for this study. Table 2 shows the time, temperature, heat treatment, and cooling method applied in each sample. It is worth noting that the heat treatments were carried out in a tubular muffle furnace. The cooling process was carried out by various means to observe how the metallographic structure and hardness differ. In this way, the treatments were replicated almost three times by each sample, to obtain adequate statistical values, and then an average was obtained.

Table 2. Experimental design of heat treatments.

SAMPLE	HEAT TREATMENT			COOLING
	QUENCHING 900 °C ×2h	TEMPERING 190 °C ×1h	ANNEALING 915 °C ×2h	
Base material	-	-	-	-
1	+	-	-	Oil
2A	+	+	+	Oil
2B	+	+	+	Water
2C	+	+	+	Air
3A	-	+	+	Oil
3B	-	+	+	Water
3C	-	+	+	Air
4A	+	-	+	Oil

Note: + are the treatments performed on each sample, and - are that were not taken into account.

2.4. Mechanical and chemical tests.

The samples obtained before the heating and cooling in the furnace were passed through different roughing processes to obtain an excellent surface; for this purpose, all samples were slightly grounded with abrasive paper. After the thermal treatments, the samples were encapsulated in cross-section in resin to facilitate the conventional metallographic preparation and hardness tests.

A conventional Felisa furnace was used to heat the samples. This furnace allowed reaching temperatures of up to 1200 °C, and enough to carry out the tests since the maximum temperature desired in the quenching process is 915 °C. In the case of the hardness tests, was used a hardness tester Mitutoyo HR-300 equipment.

Subsequently, to obtain the specimens with the sizes required to be encapsulated, a Pico 155 precision cutter was used. The encapsulation was carried out using a TP-7001 Mounting Press. In this process, the sample of nodular cast iron, together with the resin mold was introduced through the upper part of the machine. Then, it was hermetically sealed and passed through a heating stage at a constant temperature of 200 ºC and finally cooled down to room

temperature. Before performing the metallographic and hardness tests, polish the parts until reaching a mirror finish using sandpaper from 240 to 1,200, grit was necessary for the experimental analysis. The polishing process was carried out using a Nano 2000T grinder-polisher for 5 minutes with a disc speed of 200rpm. In the last stage, it was necessary to carry out a chemical attack with Picral to obtain a specimen suitable for passing through the microscope and observing its structure.
Scanning electron microscopy (SEM) and energy-dispersive x-ray spectrometry (EDS) techniques were carried used at 20 keV in COXEN INTEK equipment to evaluate the microstructure and the chemical composition over the surface. And finally, X-ray diffraction (XRD) technique was applied to identify the phases present in the samples without thermal treatment using Bruker D8 Advance equipment; patter was obtained with 40 kV, 40 mA, 2θ range (10°-70°), CuKα radiation, with a glancing angle of 1°.

III. EXPERIMENTAL DETAILS.

Fig. 3 shows the identification of each sample and its respective metallography with a magnification of 100X, which was compared and analyzed. Most microstructures show the presence of graphite nodules, where their size and distribution varies according to the cooling method, similar to [15][16].

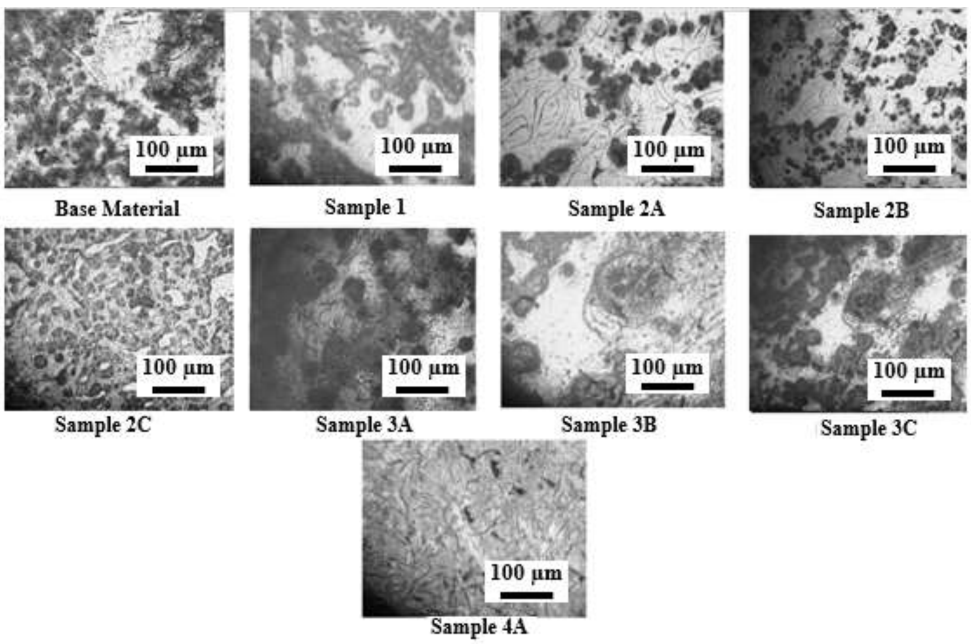

Figure 3. Metallography of the different specimens.

Pearlitic gray cast iron, its structure is complete with nodules with lamellar graphite inclusions, as shown in Table 3. The graphite is seen in the form of small veins. Perlite contains 0.8% Carbon; therefore, this carbon value is found in gray pearlitic cast iron in the bound state; that is, in the form of cement. The remaining quantity has a free state; that is, in the form of graphite. In this way, due to the geometry in the samples of ordinary gray cast iron, the graphite takes the form of small veins, this graphite is called laminar [17]. Fig. 3 shows the ferritic smelter structure where the carbon is in the free state, forming sheets of graphite streaks [18].
Due to the structure of the samples analyzed, it is a type C graphite, when a foundry has a hypereutectic composition in percentage by weight (Wt. %) (C+Si/3)+(P/3), it is higher than 4.25%, the Solidification begins with the formation of original graphite, in the interval between the temperature of the liquid and the eutectic temperature. This graph develops freely inside the liquid, without obstacles to its growth, and provides straight sheets whose thickness exceeds that of the eutectic graph when a formation arrives. This type C graphite has resistance to thermal shock, but weak tensile strength [18].
It is interesting to note that in practice, it is complicated to find funds in which all the carbon identified in the form of graphite, as just indicated. However, this kind of casting is referred to, because marking them makes it easier to study all the other kinds. The foundries in this group are called ferritic gray foundries, and in them, all the carbon is in the form of graphite. Generally, cast iron with nodular graphite is called malleable cast iron. Thus, lamellar cast iron is called ordinary gray cast iron, and cast iron with spheroidal graphite is high-strength cast iron.

The measurements were made on some parts in Rockwell B due to its low hardness, and others in Rockwell C due to its higher hardness [19][20]. Fig. 4 shows the behavior of the hardness obtained for each experimental sample.

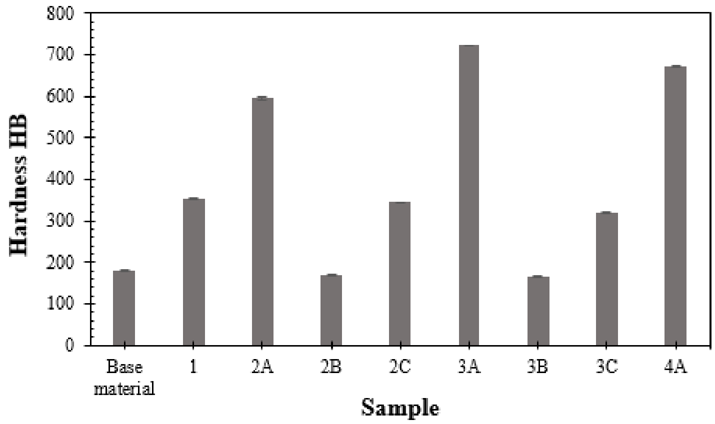

Figure 4. Graphical behavior of hardness values.

In most specimens, the hardness increased for the base sample. It was observed that with the application of quenching alone, an increase of almost twice its hardness was achieved. This notable increase in hardness can affect the material in a certain way since it brings with it an increase in the fragility of the material. This is influenced by the internal stresses generated inside the material after quenching; therefore, it is necessary to apply other heat treatments to relieve these stresses.

From Fig. 4, show that the best samples in terms of hardness are 2C and 3C because their hardness increase was substantial to the base sample. Also, after applying the tempering and annealing, an improvement in machinability is achieved for sample 1. In other words, to obtain a piece with acceptable properties, the cooling medium in the annealing must be using air, and therefore, a slow cooling. Sample 4A has a high hardness due to its rapid cooling, using oil as a means to reach ambient temperature.

Besides, it can be deduced that the best samples in terms of hardness are 2C and 3C because their hardness increase was substantial concerning the base sample. Furthermore, after the tempering and annealing are applied, an improvement in the machinability is achieved for sample 1. In other words, to obtain a part with acceptable properties, the cooling medium in it must be annealed using the air; therefore, a slow cooling [21][22]. Sample 4A has a high hardness due to its rapid cooling, using oil as a means to reach room temperature. Phenomenon appreciated is also the samples 2, 2A, and 3A, where the highest hardness is, therefore, pieces very fragile and almost impossible to machine.

These hardness variations are due to the different microstructures obtained, where it can be observed that where there are large nodules, such as in sample 3A, the hardness is the highest, and as their size decreases, so does the hardness. Also, sample 2B exhibits the lowest hardness due to the smaller size and dispersion of the graphite nodules. Therefore, to reach a higher hardness, without affecting machinability, it is necessary to obtain a microstructure with the presence of graphite nodules of average size, but with a large number of them, as can be seen in the metallography of samples 2C and 3C.

The results obtained in Fig. 4 are compared to observe the variation of the microstructure. It can be seen in sample 2 and sample 3 that at high cooling speeds during annealing, the number of graphite nodules decreased, but at the same time, their size increased. In the sample that was quenched and subsequently annealed, a microstructure with the presence of martensite and retained austenite platelets is observed. Also, oil-cooled parts have a high hardness due to the larger size of the graphite nodules contained in a ferrite structure, this can be seen in the base sample and in the heat treatments that used oil as a cooling medium (Fig. 5), such as the sample that was quenched only at 900 °C (Sample 1), was subjected to the three heat treatments (Sample 2A), that was tempered and annealed (Sample 3A).

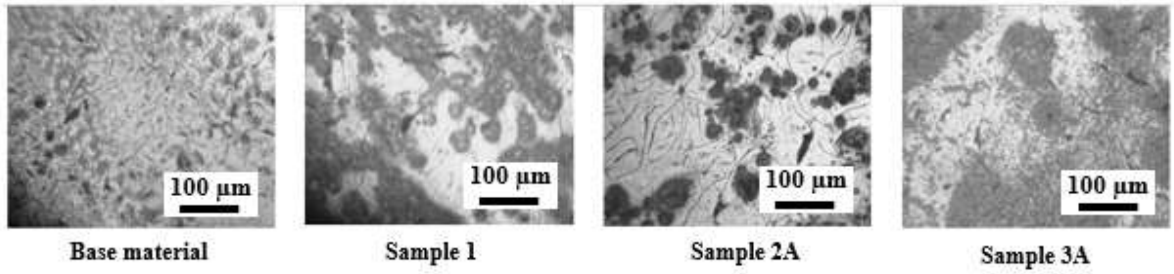

Figure 5. Metallographic samples with larger graphite size.

These results can be compared with the microstructure obtained in a research work carried out by Vélez-Restrepo [23], shown in Fig. 6, where it is observed that by applying an annealing treatment, a ferritic microstructure was obtained. In the samples that were finally annealed, a large number of graphite nodules can be observed, with a better spheroidal shape in comparison with the quenching and base material.

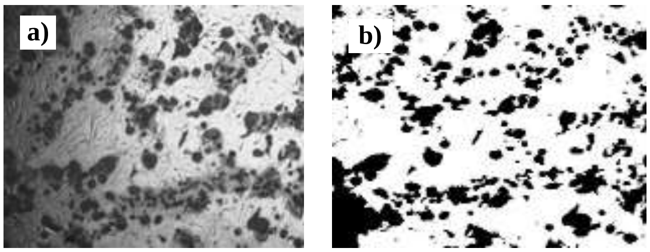

Figure 6. a) Annealed sample with a high presence of graphite nodules, and b) Modified nodular cast iron microstructure in ImageJ.

ImageJ software was used to determine the number of graphite nodules count and the amount of spheroidal graphite present in the microstructure, which has an increase of 100X. In this way, Table 3 shows the values obtained from the analysis of these images, where it can be seen that the higher the cooling speed, the lower the presence of graphite nodules in the samples that were annealed.

Table 3. Number of graphite nodules in the microstructure.

Sample	Base material	1	2A	2B	2C	3A	3B	3C
Amount of nodules	207	300	247	308	321	227	230	319

After carrying out the heat treatments, Fig. 7 and Fig. 8 shows the SEM-EDS and XRD analysis for sample 3A, which contained the highest hardness of the experimental set.

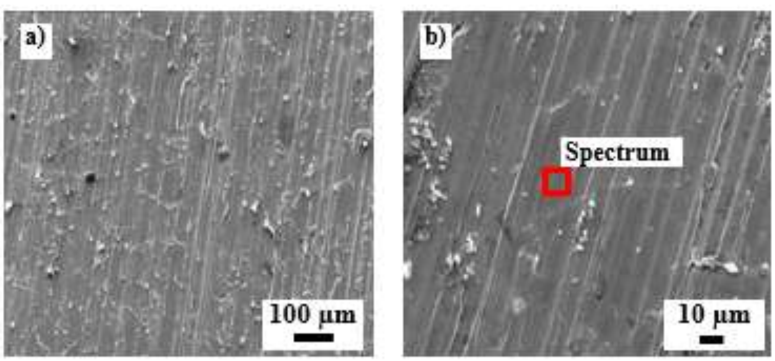

Figure 7. Micrographics of sample 3A a) 200X, and b) 1000X

Taking into account Fig. 7b, a punctual EDS was carried out to identify characteristic alloying elements [24], and the spectrum showed in Fig. 8 was obtained.

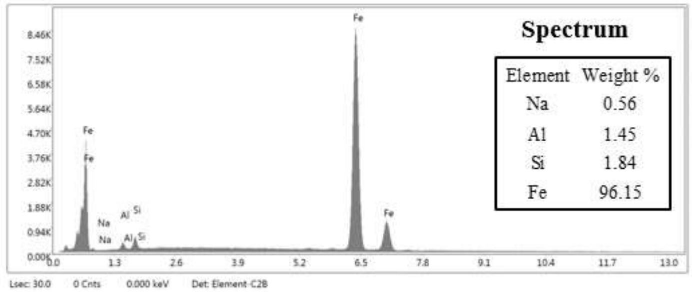

Figure 8. EDS of sample 3A.

Subsequently, to identify the phases present in the material, the XRD analysis was performed on the surface of this material, from which Fig. 9 was obtained.

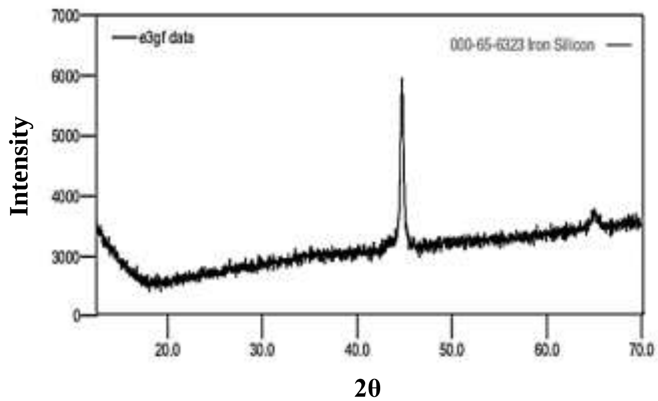

Figure 9. XRD of sample 3A.

The crystalline phases were identified with the help of the PDF-2 database of the *International Centre for Diffraction Data (ICDD)*, obtaining Table 4.

Table 4. Crystalline phases of sample 3A.

PHASE	CRYSTALLOGRAPHIC CHARACTERIZATION		NAME (PDF)
$Fe_{0.905}$ $Si_{0.095}$	Cubic Crystal System A = B =C = 2.86	α =β = φ = 90	Iron Alloy (000-65-6323)

The previous figure showed the presence of FeSi due to the alloying elements in a more significant proportion analyzed by the EDS. This phase is the beginning of the formation of the graphite nodules through the heat treatments carried out, in which the grain boundaries (nodules) can be observed according to the results obtained in Table 4.

V. Conclusions

When nodular cast iron is first subjected to a heat treatment of quenching, it achieves high and optimum hardness values for the manufacture of the discs that make up the disc brake mechanism.

According to the images obtained in the ImageJ software, it can be concluded that at high cooling speeds, the number of nodules increases, but their size decreases.

In terms of the hardness values provided by the hardness tester, it can be said that cooling in oil is adequate because it gives the material a high hardness together with a metallographic structure, where there is a large number of graphite nodules giving the material a high hardness and machinability.

A high value of hardness obtained by pos-thermal treatment improves the behavior of fiction and wear loss material, aim to the useful life of the brake disc for this use principally.

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