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Niobium Effects on Properties of Austempered Ductile Iron – ADI

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ABSTRACT : *In order to obtain adequate mechanical strength, samples of Austempered Ductile Iron (ADI) were melted with 0.2% of Niobium (Nb), which received austenite thermal treatment performed at 325°C with different cooling times in salt bath. To characterize the materials, chemical tests, structural metallographic assay, Vickers microhardness and mechanical tensile strength were performed. It was possible to verify the effect of the Nb element mainly in the appearance of niobium carbides (NbC) in the ausferritic matrix, reducing the elongation and increasing the boundary of the alloy, besides the element acting as a perlitizing agent, increasing its proportion. The Vickers micro hardness test revealed hard particles of niobium carbide (NbC) in Nb samples. The metallographic analysis revealed that there were no significant changes in the proportion of graphite when adding Nb in the samples. The carbon equivalent of samples with and without niobium addition remained 4.46% and 4.56% respectively; both alloys are in the hypereutectic area of the Fe-C equilibrium diagram.*

KEYWORDS *- Austempered ductile iron, casting, Fe-Nb alloys, heat treatment, mechanical properties.*

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I. INTRODUCTION

In most of the cast iron, the carbon appears as graphite, so both the microstructure and their mechanical behavior are influenced by the amount and shape of the graphite, by the chemical composition and the heat treatment of the alloy [1].

The addition of magnesium, cerium and other metals to the cast iron results in a completely different microstructure; the graphite forms nodules or spheres, and no longer forms flakes, giving rise to nodular cast iron. Cast and thermally treated parts based on nodular cast irons have high indices of ductility and strength between the cast irons, being comparable to some unalloyed steels. The heat treatment is crucial in the manufacture of ADI, it is necessary to strictly control the entire process in order to obtain the best results for the alloy. In the crude state of casting, the most common structure of nodular cast iron is the perlite matrix with spheroidal graphite. In certain applications where the casting part does not meet the minimum mechanical requirements, it is possible to use austenitic heat treatment, since this treatment gives a substantial improvement in the mechanical properties of nodular cast iron [2].

The austenite consists of preheating the part, then heating above its critical temperature, in the range of 840 ° C to 950 ° C, remaining at that temperature for a sufficient time to obtain a saturated carbon matrix. The part is then cooled rapidly to the temperature of the furnace, which is generally between 230 ° C and 400 ° C, remaining at this temperature for the isothermal treatment and finally the part is cooled at room temperature before the reaction starts bainitic [3].

The choice of the temperature of the heat treatment is a critical stage of the process, since the careful choice of the austenitization temperature will reflect on the resulting microstructure and consequently affect its mechanical properties [4].

Austempered nodular cast iron, known as ADI, has an excellent combination of mechanical properties, presenting in some applications higher values compared to some unbound cast steel.

In the 1980s, the ADI had its main application in engine parts, having in 2002 a great application in the automobile industry. An interesting aspect of ADI is its competitiveness with light alloys. ADI can replace components made from forged, cast or welded aluminum, with equivalent or reduced weight [5].

Although the ADI has a density 2.5 times higher than aluminum, it presents a 2.3 times elasticity mode, a three or four times greater flow limit, a higher fatigue limit and also a vibration absorption capacity [6].

In order to obtain or further improve certain properties, it is possible to add alloying elements to the ADI, so the automotive industry has benefited from the ADI, as it has a good relative weight per unit of flow resistance, producing parts with less thick walls, lighter and with the same mechanical resistance, generating projects with greater efficiency. When using niobium in ADI parts, the automotive industry has obtained great gains in mechanical properties, mainly in resistance [7].

The high affinity between niobium and carbon tends to form hard particles of niobium carbide (NbC), increasing the fraction of perlite in the matrix, as the niobium acts as a perlitizing agent, also providing gains in tensile strength and yield limit [8].

With this context, this work aimed to investigate the properties resulting from the addition of Niobium (Nb) to nodular cast iron, in order to improve the mechanical properties of the alloy Fe-Nb.

II. MATERIALS AND METHODS

This section describes the process for obtaining the alloy, obtaining the test specimens, the thermal treatment cycle and the method for the analysis of the mechanical and microstructural properties of the materials obtained.

2.1 Casting the Fe-Nb alloy

The alloy was forged in the Foundry Laboratory of the University Center TUPY - Boa Vista Unit, in Joinville, Santa Catarina, Brazil, according to the following steps: a) Mold making in wood following the standards of ASTM A897 / A; 897M-02 (b) Loading of the furnace with pig iron, steel scrap and silicon in stone; c) Charge melting and addition of alloying elements at 1520 ° C; d) Removal of the metal at a temperature of 1520 ° C for treatment of nodularization, then carried out the inoculation treatment; e) Leakage of the material into the molds in order to obtain the samples. After demolding at room temperature, Y-type blocks were obtained as shown in Figure 1:

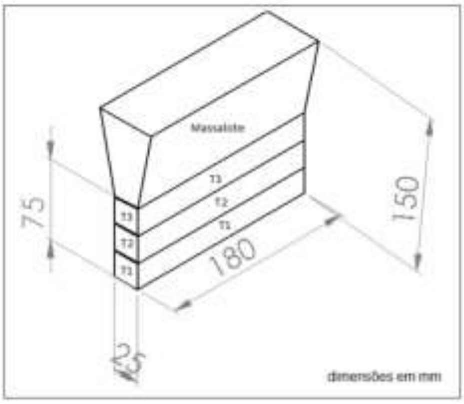
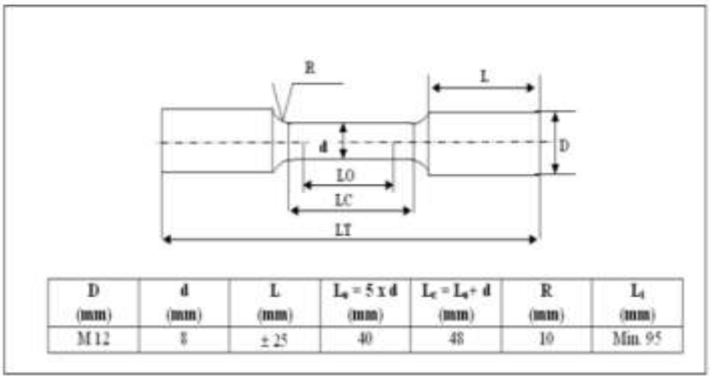


Fig. 1: Dimensions of Y-type block [11].

2.2 Obtaining the test specimen

After melting and demolding of the alloy, the Y-type blocks were machined using CNC equipment in order to obtain the normalized test bodies according to Figure 2:



D (mm)	d (mm)	L (mm)	$L_o = 5 x d$ (mm)	$L_c = L_o + d$ (mm)	R (mm)	L_t (mm)
M12	8	± 25	40	48	10	Min. 95

Fig. 2: Dimensions of the test specimen [11].

2.3 Fe-Nb thermal treatment

After the machining of the test specimens, the heat treatment step was started. The temperature control of the baths was carried out using a thermocouple. The cycles of the treatment used is illustrated in Figure 3:

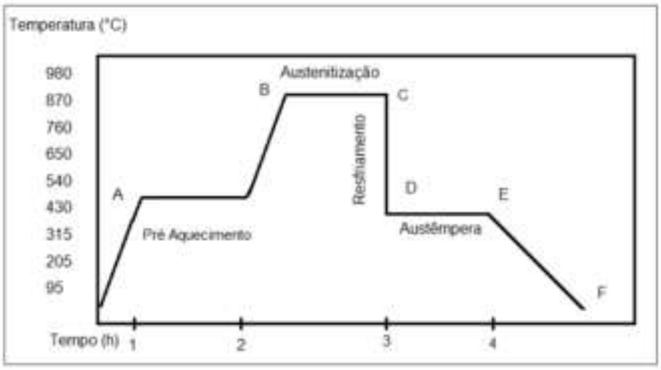


Fig. 3: Austempering thermal treatment.

Section AB - Preheating the material at 425 ° C for 1 hour in a controlled atmosphere oven, then performed the austenitization at 900 ° C for 1 hour. Section CD - Quick cooling in salt bath oven at 320 ° C in order to start the heat process. Section DE - The treatment of austempering is performed at different times of the day: 8, 16, 32, 64 and 96 minutes for the treatment in salt baths. Section EF - The samples were cooled to room temperature.

2.4 Chemical analysis

The chemical composition of the samples was obtained from shrouded coins shortly after the fusion of the iron alloy. To perform the chemical analysis, an optical emission spectrometer (Spectrolab) was used.

2.5 Metallographic analysis

The preparation of samples was performed according to standard procedures, taken samples from the useful areas of the specimens in order to carry out the sanding and then polishing treatment. The chemical attack for the observation of the microstructure was carried out using a solution of nitric acid and alcohol - Nital 4% (Alphatec). Samples with and without niobium addition were analyzed after thermal treatment. For the analysis, an optical microscope (Olympus BX-51) was used, coupled to an image analyzer software (Image Pro-Plus).

2.6 Microhardness analysis

The Vickers microhardness test was performed using a durometer gauge (Liyi LYHV-1000). The assay was based on ASTM E-384 [11]. The indenter used has a pyramidal shape. The purpose of the measurement was to define the measurement of the diagonals printed on the sample and then perform the calculation to define the Vickers microhardness, as shown in Equation 1:

$$HV = 1.854 \times F / d^2 \quad \text{(Eq.1)}$$

Where: HV = Vickers microhardness, F = applied load (g) and d = diagonal of the square or average print of the deformed printing diagonals (µm).

2.7 Tensile test

The tensile tests were performed under climatic conditions at the Materials Science Laboratory of the University of Santa Catarina State (UDESC) Campus Joinville - SC. A universal mechanical testing machine (EMIC) was used; the test speed was maintained at10 mm / min. The Figure 4 shows the fixation of the specimens in the mechanical testing machine.

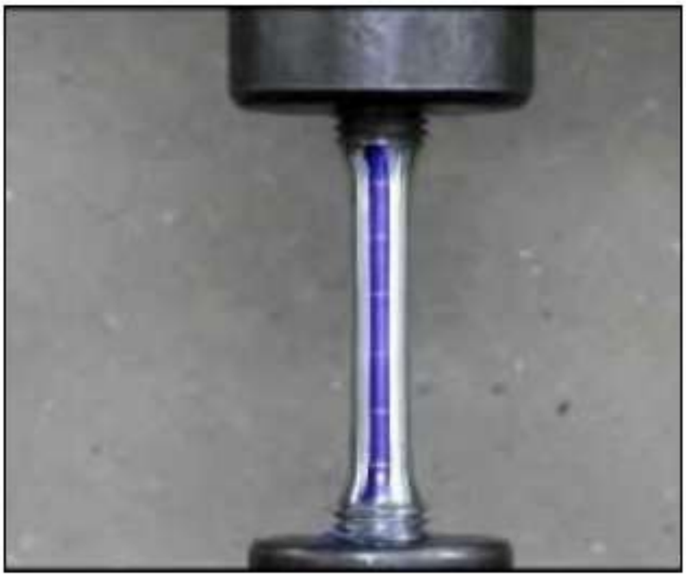


Fig. 4: Fixation of the specimens for the tensile test.

III. RESULTS AND DISCUSSION

After melting the alloy and the preparation of the specimens, the thermal treatment of austempering was performed, in sequence the chemical, metallographic and mechanical analysis of samples with and without addition of niobium was done. The thermal treatment of austenite was carried out at 320 ° C at times: 8, 18, 32, 64 and 96 minutes, in order to verify the influence of niobium addition on the mechanical properties of ADI.

3.1 Chemical results

The results obtained in the chemical analysis are set forth in Table 1:

Table.1 Chemical analysis of samples.

Sample	C	Si	Mn	P	S	Cr	Mo	Al	Cu	Mg
With Nb*	3,7	2,2	0,2	0,073	0,024	0,11	0,18	0,04	0,39	0,065
Without Nb	3,8	2,2	0,2	0,074	0,026	0,09	0,17	0,03	0,39	0,071

Nb *0,2

The values obtained of the chemical composition of the alloy meet the norm NBR 6916: 2017 [12]. Samples with niobium addition showed a lower carbon value compared to samples without niobium addition. This reduction of the carbon content is due to the fact that during the melting of the alloy, after the addition of niobium, the temperature of the furnace was maintained for a few minutes to incorporate the niobium into the alloy.

The chemical composition of ADI is of extreme importance in obtaining its mechanical properties. Silicon is the most important element, that is, it allows the formation of an ausferritic structure. By increasing the silicon content from 2.4 to 3.8%, tensile strength, yield strength and resilience are increased while maintaining the same elongation. [10].

According to Table 1, the silicon obtained remained within the minimum range recommended for an ADI, but very close to the minimum limit, and may not be enough, which could influence losses in the mechanical properties of the samples.

It is worth remembering that values between 0,02% and 0,074% of phosphorus, the reduction of mechanical properties occurs, due to micro-segregations of this element to the contour of the cells, forming inclusions rich in phosphorus, containing magnesium, sulfur and oxygen 10.

The values of phosphorus obtained are close to the mentioned limit, favoring the reduction of the mechanical properties, especially the elongation.

The carbon equivalent for no-addition addition of Nb remained at 4.46% and 4.56%, respectively. Both alloys are in the hypereutectic area. The equivalent carbon of the sample without niobium presented a high value, practically leaving the ideal field of the diagram of Henderson, being able to have damages in the mechanical properties.

Samples with Nb addition showed lower carbon equivalent than samples without niobium addition. This reduction of the carbon content is due to the fact that during the melting of the alloy, after the addition of niobium the temperature of the furnace was maintained for a few minutes, to better incorporate the niobium to the alloy.

The increase of the equivalent carbon raises the number of nodules, reducing the amount of perlite; since the perlite favors the abrasion of the material [10].

The nodularization is performed prior to inoculation, by the addition of alloying elements such as magnesium, cerium and calcium, with magnesium being the most used.

The nodularizers increase the surface tension and the graphite / metal interface, favoring the growth of the graphite in the nodular form. It is important to control the amount of nodularizers, so that residual magnesium contents do not exceed 0.08%, because when this value is exceeded, the formation of carbides during solidification may occur, as well as the formation of degenerate graphite. The chemical composition of ADI is of extreme importance in obtaining its mechanical properties. Silicon is the most important element, that is, it allows the formation of an ausferritic structure. Increasing the silicon content from 2.4 to 3.8% increases tensile strength, yield strength and resilience while maintaining the same elongation [10].

The silicon obtained is within the minimum range recommended for an ADI, but very close to the minimum limit, and may not be enough, which could influence losses in the mechanical properties.

The residual magnesium found was within the expected range, but close to the maximum range, which could lead to the formation of carbides and reduce the degree of nodularization for both samples.

The amount of Nb was higher than expected, the probable causes for this high value are: a) During the melt processing of the alloy, where less liquid metal could have been left in the furnace, which diluted the amount of niobium added; and b) The time for the incorporation of niobium into the alloy was excessive, making the alloying element excessively incorporated.

The values of phosphorus obtained are close to the mentioned limit, favoring the reduction of the mechanical properties, especially the elongation.

With values between 0.02% and 0.074% of phosphorus, the reduction of mechanical properties occurs due to micro-segregation of this element into the cell boundary, forming phosphorus-rich inclusions containing magnesium, sulfur and oxygen [10].

3.2 Metallographic result

Microstructure analysis was performed on all specimens, with and without niobium addition, all in the austempered state. The Figure 5 and Figure 6 exposed above shows images of the metallographic assay obtained without using the Nital 4% etching for samples without Nb addition using 100X and 500X of magnification, respectively:

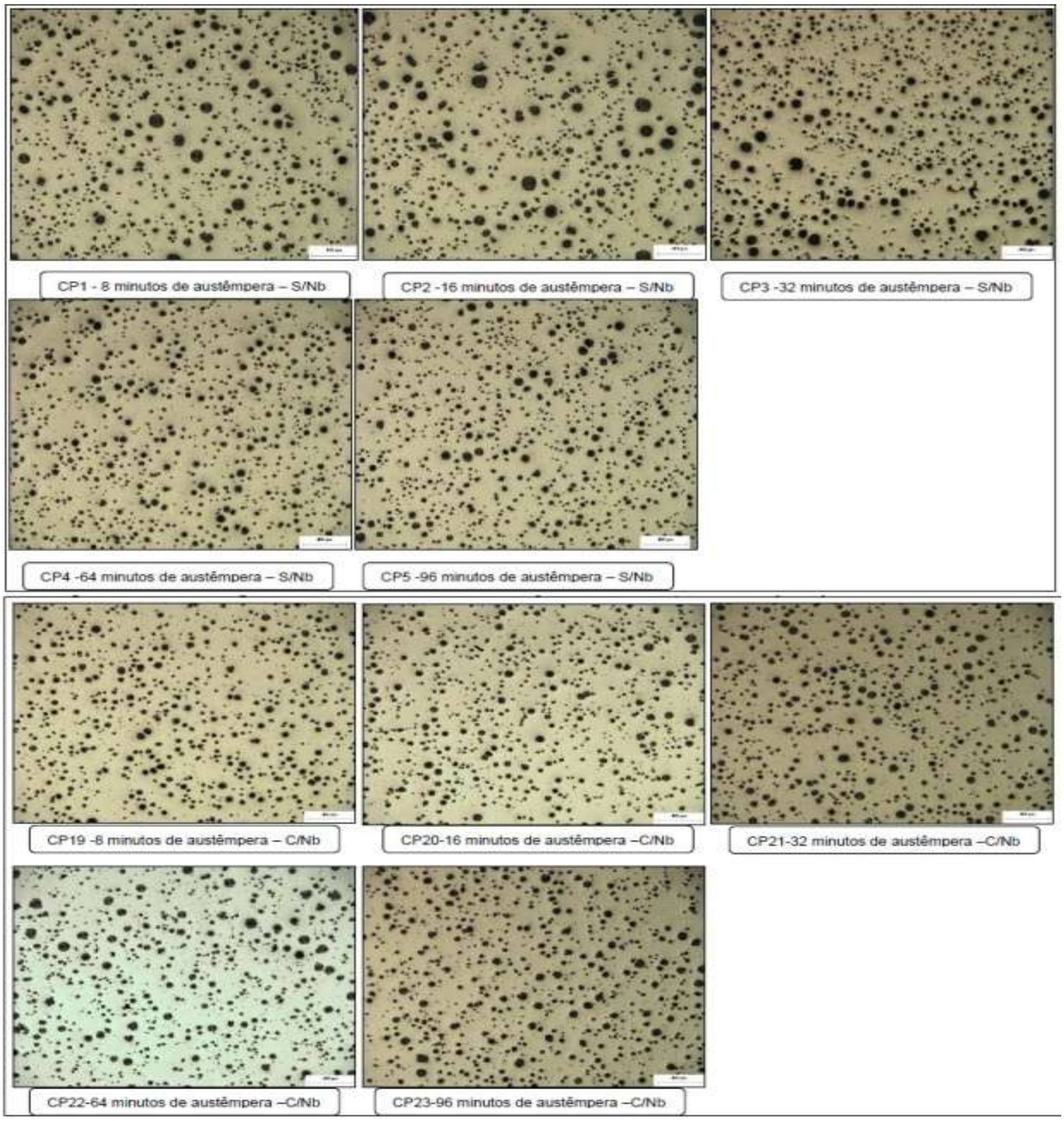


Fig. 5: Metallographic analysis of samples without addition of Nb and without chemical attack, 100x of magnification.

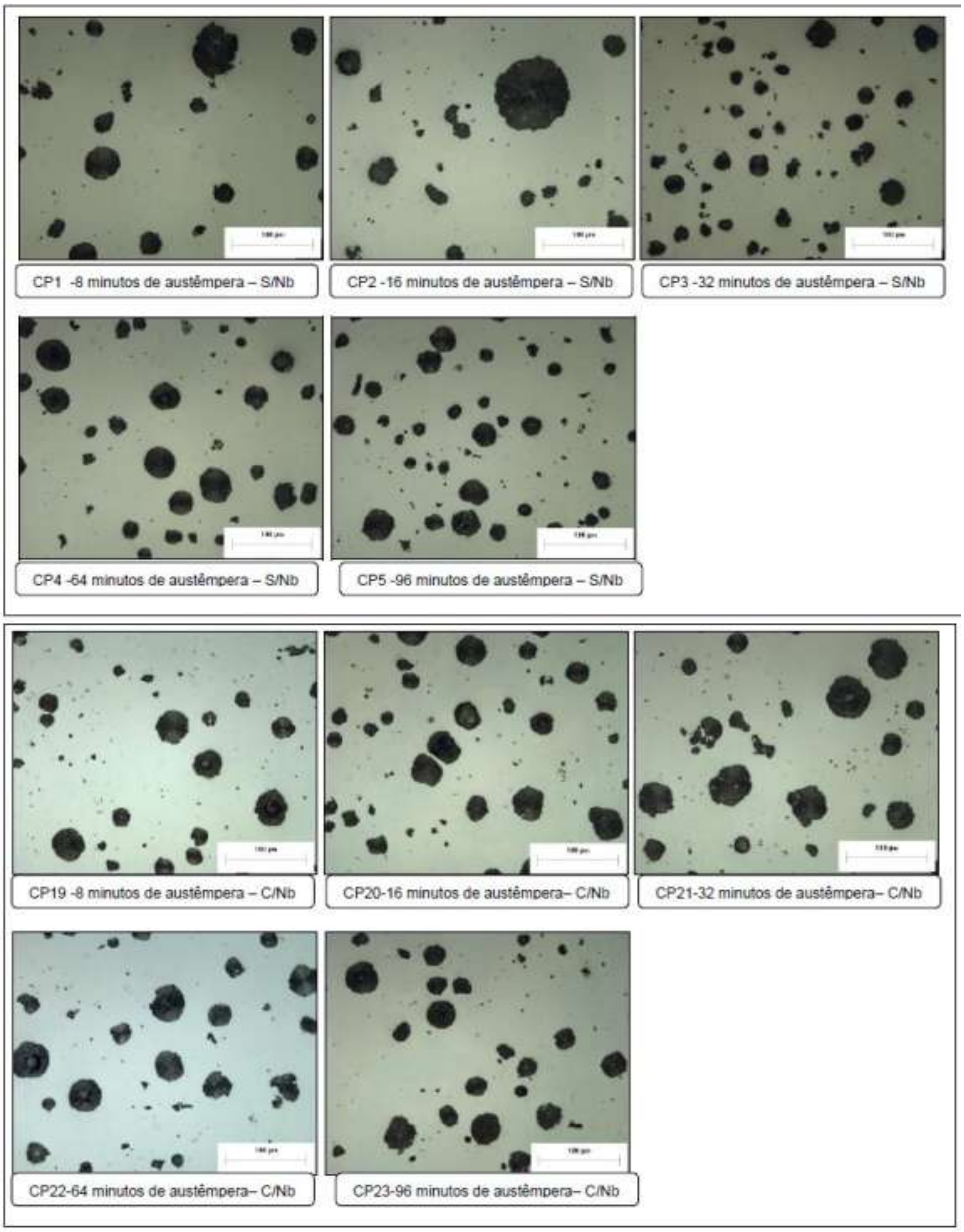


Fig. 6: Metallographic analysis of samples without addition of Nb and without chemical attack, 100x of magnification.

The Figure 7 exposed below shows images of the samples without and with addition of Nb with 500x magnification:

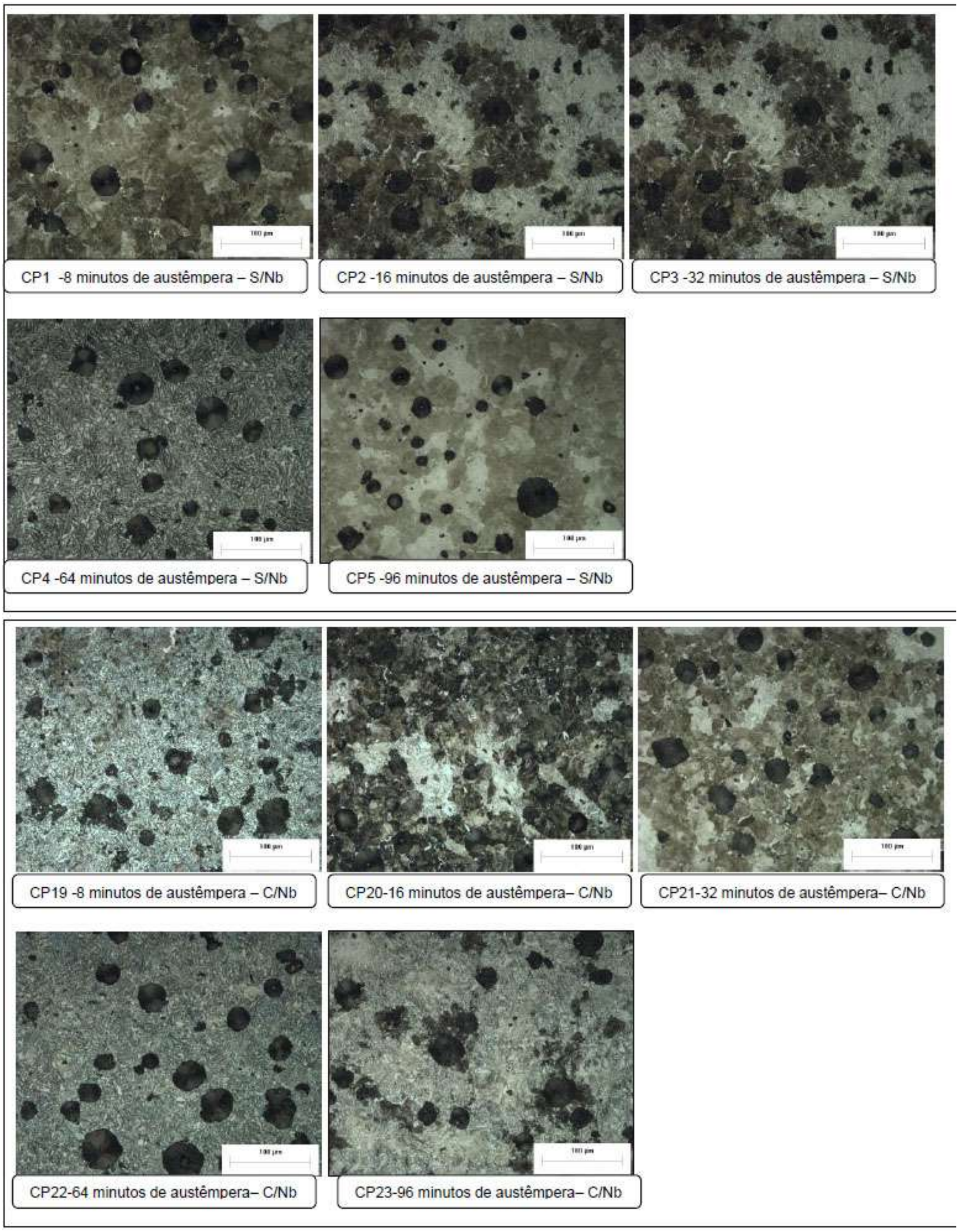


Fig. 7: Metallographic analysis of the samples without and with addition of niobium and with chemical attack, 500x of magnification.

Figure 8 shows in detail niobium particles dispersed in the sample matrix with niobium addition, using 500x magnification and without chemical etching:

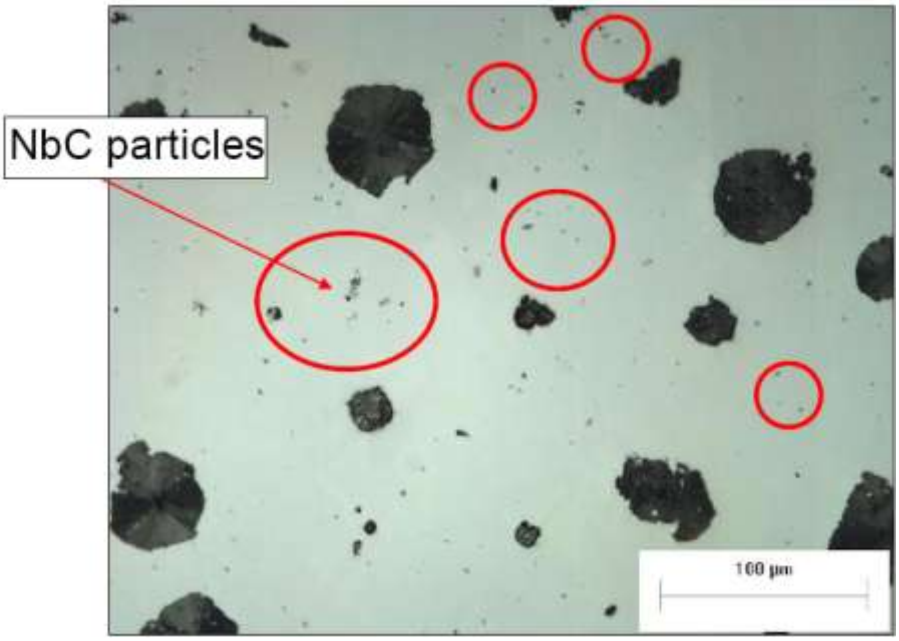
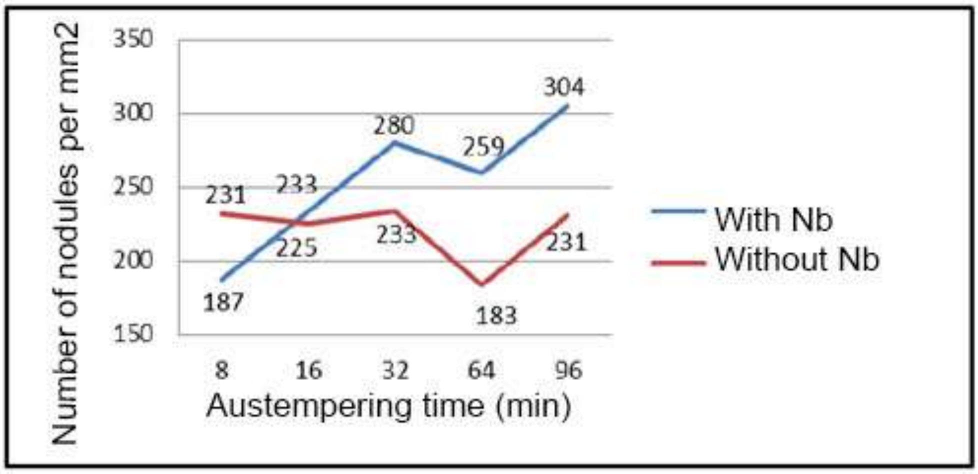


Fig. 8: NbC particles dispersed in the sample matrix.

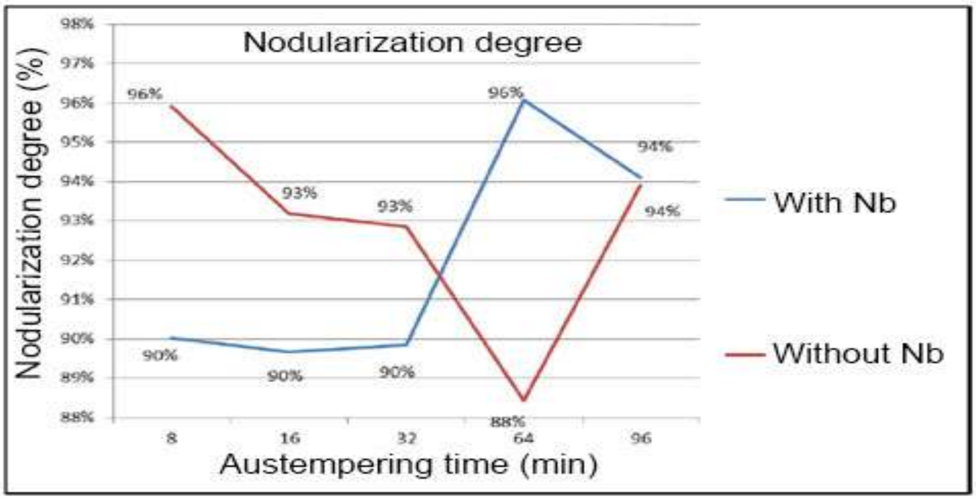
The number of nodules and the degree of nodularisation directly interfere with the mechanical properties of the ausferritic material, obtained after the treatment of austenoma. The nodules act as a carbon reservoir during austenitization, ensuring the enrichment of austenite in carbon and, in the crude state of fusion, to better distribute possible segregations [9].

The Graph 1 shows the number of nodules / mm^2 in both sample situations, with and without niobium addition:



Graph 1: Number of nodules/mm^2.

The Graph 2 shows the degree of nodularization in both situations:



Graph 2: Degree of nodularization.

In relation to the proportion of the phases, the amount of graphite with and without niobium addition varied. In the samples without niobium addition, the graphite was present in the average amount of 12% during all the periods of austempering (8, 16, 32, 64, and 96 minutes) and the amount of the austeferritic matrix remained in the average proportion of 89% in the same test periods.

In the samples with niobium addition, the average amount of graphite remained at 12% and the average amount of the austeferritic matrix remained at 88% for the same test periods.

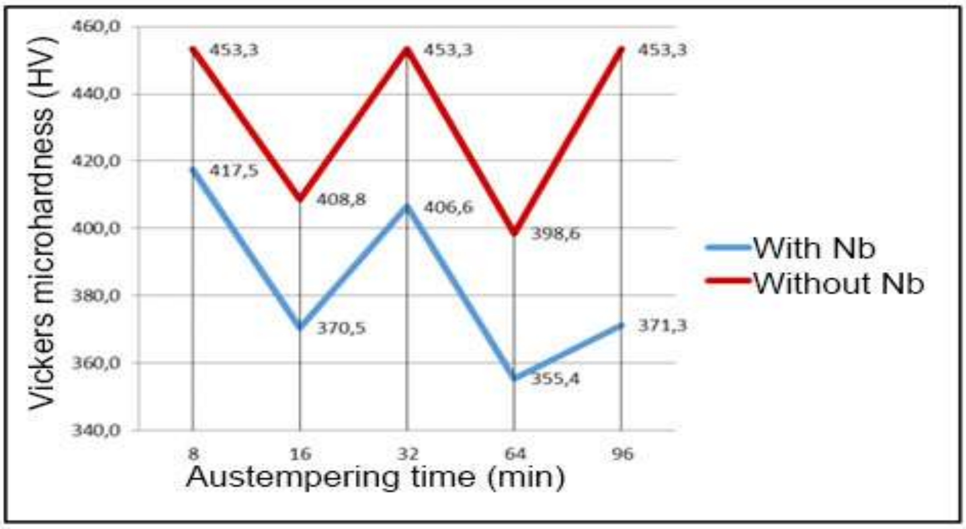
In relation to the proportion of the phases without niobium addition, the ausferrite remained in the proportion of 84% (8 min), 33% (16 min), 42% (32 min), 100% (64 min) and 29% (96 min). The amount of perlite remained 16% (16 min), 67% (32 min), 0% (64 min), and 71% (96 min).

In relation to the proportion of the phases in the samples with niobium addition, the ausferrite remained in the proportion 96% (8 min), 45% (16 min), 35% (32 min), 100% (64 min), and 52% (96 min). The ratio of perlite remained at 4% (8 min), 55% (16 min), 66% (32 min), 0% (64 min) and 48% (96 min).

High perlite content was observed in the samples without addition of niobium, this value due to problems in the cooling of the test specimens after their fusion. Samples with niobium addition showed an increase in perlite content, due to niobium acting as a perlitizing agent.

3.3 Vickers Microhardness result

Samples with Nb addition showed increase in hardness compared to the samples without Nb addition, which increase represented by the formation of hard particles of niobium (NbC) carbides present in the matrix. Graph 3 compares the microhardness obtained in the samples:



Graph 3: Result of Vickers microhardness test.

3.1 Tensile result

The mechanical properties obtained in the tensile test were: yield stress (σesc), maximum stress (σmax) and elongation, obtained in the T2 region, with and without the addition of niobium, respectively:

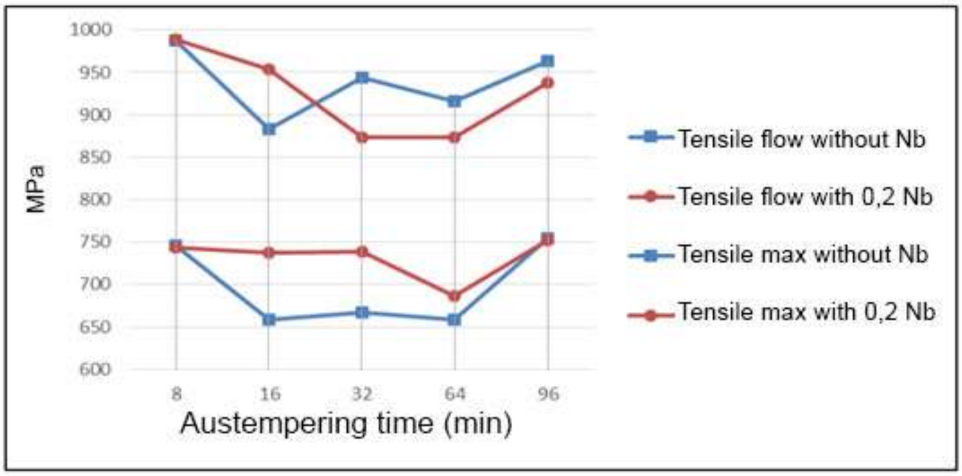
Table 2. Results obtained from the alloy without addition of Nb element.

	Austempering		Sample				
Material	Temperature	Time (min)	N	Region	σ flow	σ max.	Stretching (%)
Without Nb addition	320 °C	8	1	T2	746	987	1.5
		16	2	T2	658	883	3.7
		32	3	T2	667	944	5,9
		64	4	T2	659	916	8.9
		96	5	T2	754	963	3,1

Table 3. Results obtained from the alloy with addition of Nb element.

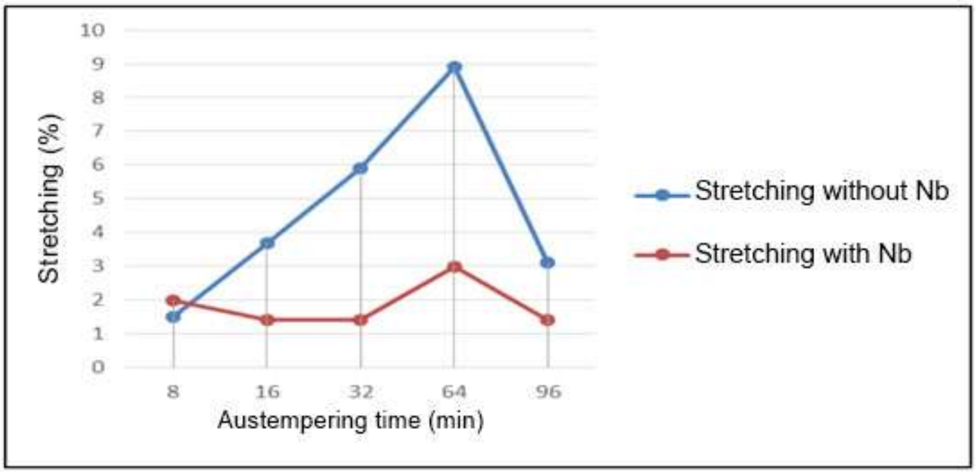
	Austempering		Sample				
Material	Temperature	Time (min)	N	Region	σ flow	σ max.	Stretching (%)
0,2% Nb	320 °C	8		T2	743	989	2
		16		T2	737	953	1,4
		32		T2	739	873	1,4
		64		T2	687	874	3,0
		96		T2	752	938	1,4

Graph 4 and Graph 5 shows the variation of the mechanical properties with respect to the sample time of the samples:



Graph 4: Comparison of the results of yield stress and maximum stress for both samples.

As shown in Graph 5, in the T1 and T3 regions of the samples because they were at the ends of the Y block, presented solidification problems such as porosities and microchips, the results of these regions were disregarded.



Graph 5: Comparison between stretching results for both samples.

It is observed in the last graph that with the time of austempering of 64 minutes there was the highest result in elongation, still below the result for a quality ADI, reflecting the solidification problems of the pieces.

From the data it is observed that samples with niobium addition showed gains in yield stress and losses in elongation.

The elongation of samples with addition of Nb should be less than that of samples without addition, because Nb is a perlitizing agent [9].

The values obtained in Tables 2 and 3 refer to the properties found in the center of the samples, that is, the most critical part region (thermal center), where imperfections, defects, structural heterogeneity and other defects are usually concentrated, due to being the last region of solidification and thermal transformation [10]. As a result of these defects the mechanical properties obtained were lower than expected for a quality ADI.

IV. CONCLUSION

The heat treatment of austempering in 64 minutes showed a higher degree of nodularization in the samples with and without addition of niobium, also only ausferrite was observed in the matrix. By adding niobium there was a reduction in the amount of graphite nodules, provided by the reduction of the carbon content.

The addition of niobium form hard particles of niobium carbides (NbC), these samples had higher microhardness when compared with samples without addition of Nb. The microstructural analysis revealed that there were no significant changes in the graphite ratio when adding the Nb element to the alloy.

By the analysis of the equivalent carbon, the samples without addition of niobium presented as a hypereutectic alloy, facilitating the formation of micro-refractions and porosities mainly inside the part, interfering mainly with the elongation value obtained in the tensile tests. Samples with niobium addition showed lower carbon equivalent contents, due to the maintenance time of the temperature in the furnace, in order to dilute the entire nipple in the alloy.

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