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# ANALYSIS OF HEAT TREATMENTS AND MECHANICAL STRENGTH IN A HSLA STEEL

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**Abstract.** *The present study was carried out on the analysis of the behavior of a steel alloyed to Niobium ASTM A572 after being subjected to normalization and tempering heat treatments, in this sense the general objective of this work sought to validate the performance of the steel in mechanical resistance tests and analyzes microstructural, the manipulation of the variables found by the laboratory tests were treated in a quantitative and qualitative way with theoretical basis, the behavior of the steel under treatment was positive, surpassing in all aspects the steel under normal conditions having the microscopically proven with the material structure, thus listing the importance of this study as a basis for future research. For the tensile test, there was an increase of 5.56% of the normal steel for the tempered and tempered, and an increase of 13.16% for the yield limit, the hardness was greater, however it remained in balance, already for energy of impact there was an increase of 93.54% for tempered and tempered steel, the predominant microstructures in the specimens were a combination of tempered martensite with cementite and ferrite for tempering and fine perlite with ferrite for normalization, thus justifying the results obtained in the tests.*

**Keywords:** *niobium steel; thermal treatments; mechanical resistance.*

## 1. INTRODUCTION

Brazil has a wide variety of minerals in its subsoil, which can be said to be a privilege, as it has great potential in the production of ores, providing the country with a highlight in the world scene in the field of extractive activity (MELFI *et al.*, 2016). Among the various ores extracted from Brazilian lands such as iron, tin, aluminum, manganese, gold, titanium, Brazil also has Niobium, being that it holds the largest reserves of this mineral, thus contributing to a privileged position vis-à-vis other countries in the world (SILVA, 1994).

According to Carmo (2017, p.15), “Niobium is a naturally occurring element. It is a soft, safe and available metal, in addition to being ductile, malleable and highly resistant to corrosion. For its characteristic of improving properties and functionality”. Niobium has several applications today, especially in the steel industry. Its use has been increasing in the technological environment, and it can be noticed its use in the aerospace, rail and automotive sectors, in addition to its applications in civil construction and in the areas related to energy (SILVA, 1994).

The study was justified, therefore, new technologies are emerging all the time, as in the case of steel microalloyed to Niobium, which ends up generating a fierce competitiveness between industries. As the market is increasingly demanding, products need to have as much confidence and quality as possible. When thinking about the future, what is the importance of having a study of new materials that are more resistant and lighter as a solution for audacious projects with lower costs? For this reason, the search for new materials such as Niobium microalloyed steel has been growing more and more.

This research aimed to validate the performance of the micro alloyed steel to Niobium after receiving heat treatments being possible to reach this objective by performing the mechanical resistance tests and microstructural analyzes, thus creating knowledge about the material, which can be used in future researches that can add value to the Niobium metal.

Proposing such a scenario, the present work was an experimental and bibliographic research.

According to Macedo (1995) a bibliographic search is defined as the search for information, it is the first step looking for research-related subjects, this work was based on books, articles and publications in the field of materials science, providing the basis for an experimental research, being defined by Chizzotti (2018), as the part of the research where tests are carried out in order to manipulate the study variables, understanding how the phenomenon is produced, in this case the specimens of steel microalloyed to Niobium under heat treatment were subjected to mechanical tests using the equipment available in the laboratories of materials science, material resistance and manufacturing processes at the University Center of Patos de Minas-UNIPAM, thus resulting in parameters that were treated through a quantitative and qualitative approach.

## 2. THEORETICAL REFERENCE

### 2.1 Nióbio

Niobium was discovered in the early 19th century by the British Charles Hatchett, he named the ore primarily as "columbium", but after a while the International Chemical Union recommended the use of the name Niobium, in honor of the Greek goddess Niobe, daughter of Tantalus, which also named another chemical element in the periodic table (FERNANDES, 2012).

Niobium (symbol Nb) is a clear, shiny, ductile silver metal, resistant to corrosion and high temperatures, is a transition element with atomic number 41 in the Periodic Table, has a density of 8.57 g / cm<sup>3</sup>, melting point at 2,468 °C and boiling point of 4744 °C, being highly resistant to heat and wear and can be considered a refractory metal (SILVA, 1994).

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According to Mekkawy et al (1991), the extracted niobium is currently used as an enhancer in various media in the steel industry, its addition in small quantities (0.01 to 0.10%) to low carbon steels leads to improved properties and also in the emergence of new ones. These metals from the addition of Niobium are called microalloyed steels or High Strength Low Alloy *Steels* (HSLA).

### 2.2 Microalloyed steel

Steels are considered metal alloys consisting basically of iron and carbon, and may contain other elements in their composition, the so-called alloy elements. The mechanical properties of steels are directly related to the carbon content in their compositions, thus being classified into categories: steel with low, medium, or high carbon content (CALLISTER; RETHWISCH, 2016).

Low carbon steels are those produced in greater quantity because they are cheaper to produce than other steels, normally have a carbon content of less than 0.15%, resulting in low hardness and low resistance. According to Callister and Rethwisch (2016), low carbon steels seek to fill this gap with low hardness and resistance with the addition of alloy elements, thus emerging the subcategory of microalloyed steels or High Strength Low Alloy *Steels* (HSLA), these they can be formed by elements such as copper, vanadium, nickel, molybdenum, niobium, titanium, among others. These elements can be added in a combined or isolated way, their development is briefly based on: (i) Refining the grain to increase strength and toughness; (ii) Improved toughness and weldability for low carbon steels; (iii) Hardening by precipitation (DINIZ, 2005).

According to Diniz (2005), Niobium metal has been used as a microalloy normally in low carbon steels (less than 0.15% carbon) both structural and for mechanical construction, since the main modification in the final properties of micro alloyed steels to Niobium is the increase in the material's resistance without the need to increase the carbon concentration, this benefit is due to the growth of the grain during reheating due to Niobium carbonitrides. Even with the addition of Niobium, there is a small improvement in grain refining, which can be further intensified by making surface heat treatments to steel (MEKKAWY et al, 1991).

### 2.3 Heat treatments

For Ferraz (2005), heat treatments would be a cluster of heating and cooling procedures, always having a detailed control of time, atmosphere, temperature, and cooling speed. Heat treatments are mostly used in steels with high carbon content or with binder elements. For him, the main objectives of treatment are:

- Increased mechanical strength; decrease or increase in hardness;
- Improved resistance to corrosion, heat and wear;
- Modification of magnetic and electrical properties;
- Removal of internal tensions;
- Improved workability and ductility.

The main parameters that have total influence on the final result of the heat treatment are:

- Heating: being carried out above the critical temperature of the material (723°C), thus obtaining the austenization of the steel, with the formation of austenite, which would be the starting point for the heat treatments;
- Heating time: it is necessary to obtain a uniform temperature throughout the steel, taking into account the size of the specimens;
- Speed and cooling: it will be the cooling speed that will determine the structure of the steel and its desired properties.

According to Ferraz (2005), the most used heat treatments are annealing, normalization, tempering and tempering. For Callister and Rethwisch (2016) “the term annealing refers to a heat treatment where a material is exposed to a high temperature for a prolonged period of time, and then it is cooled slowly”, the annealing seeks to relieve tensions, reduce hardness and increase ductility and toughness. The normalization treatment has the same purpose as annealing, however its cooling is faster because it is in contact with air, this is a treatment that aims to refine the grains seeking a uniform size distribution, normalization is achieved by heating the material to at least 55 °C above the upper critical temperature, the material is finally cooled to air temperature.

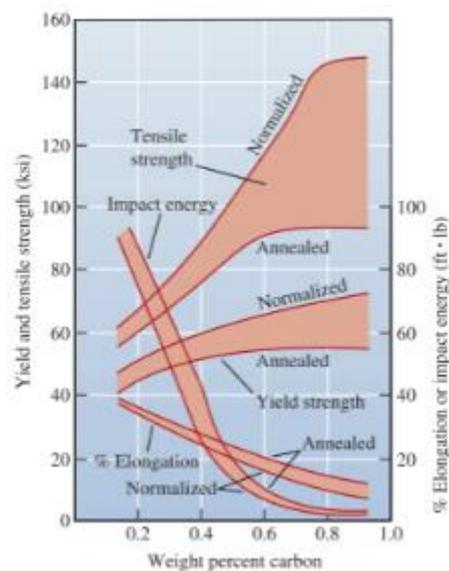


Figure 1. Effect of carbon in normalization and annealing treatments.

According to Ferraz (2005), the quenching consists of the quick cooling of the part from a temperature higher than the critical, seeking to obtain a structure with high hardness, in addition it leads to an increase in the limit of tensile strength, but ends up having a reduction in malleability. and even the appearance of internal tensions. In order to mitigate these tempera inconveniences, a tempering heat treatment is sought, as it helps to relieve or remove internal stresses, in addition to correcting the excessive hardness and fragility of the material, thus improving malleability and resistance to shock, whatever material is used. subjected to tempering it must be heated below the eutetoid temperature (723°C), for a specific period of time. Normally, tempering is carried out at temperatures between 250°C and 650°C, this treatment allows the formation of tempered martensite. After carrying out the correct treatment, it is sought to analyze microscopically in order to reveal the microstructure of the material where it can be checked whether the correct properties were obtained.

### 3. METHODOLOGY

The material used in the present work was ASTM A572 GR 50 microalloyed steel with about 0.15% carbon and 0.023% Niobium. The specimens were subjected to heat treatments of quenching, tempering and standardization, using the Jung oven LF04212 of the materials science laboratory, which withstands temperatures up to 1200 °C. To perform the treatments, some essential parameters were followed to obtain maximum reliability.

With regard to normalization, the steel was first heated to at least 55 °C above the upper critical temperature, for 10 minutes, knowing that the Niobium microalloyed steel has 0.15% carbon determined the correct temperature for normalization was 905°C. After this heating, we remove the specimens from the oven in order to have a quick cooling in contact with the air. For the tempering, the steel was heated above the austenization temperature, that is, above 727 °C, for 10 minutes, immediately afterwards it was cooled in water. After tempering, the steel was turned to the oven to perform the tempering, seeking to mitigate the inconveniences of tempering. For tempering, the steel was heated to 500 °C for 10 minutes, immediately after being cooled in air.

After the heat treatment processes, these specimens were submitted to a microstructural analysis, where the structure of the metal, its constitution and alloys can be observed through a microscope, determining the various characteristics of the material (ROHDE, 2010). To prepare the sample, we had to cut it so as not to compromise the material structure, using a metallographic cutter. Subsequently, the sample was embedded, followed by sanding with 80,120,320,600 and 1200 sandpaper, then polishing with 9, 6, 3 and 1  $\mu\text{m}$  diamond paste. Finally came the chemical attack, made by immersion in Nital 2% for approximately 10 seconds, revealing the microstructure of the metal under the microscope.

The next step was to perform the mechanical tests on the specimens, the first performed was the hardness test. The test consists of measuring the depth of the impression left on the steel after application of the load, using a Mitutoyo HR-400 durometer, with tests on the Rockwell scale.

The next test was a tensile test using equipment from the material resistance laboratory, which resulted in data on tensile strength, yield limit and elongation that we can see through the generated strain strain graph. According to Pannoni (2002), microalloyed steels, which have high mechanical resistance, allow the relationship between the yield strength and the tensile strength limit to be raised without appreciable loss of ductility. In addition, these types of steels are useful when you want to improve the resistance to shock and the fatigue limit, as well as making it possible to proportionally decrease the section, in other words, due to their high mechanical resistance, it is possible to use sections thinner in structures.

Subsequently, the steel was subjected to the impact test where the Charpy method was standardized following the parameters of type A specimen, that is, a V-notch was made, with a depth of 2.0 mm, 55.0 mm long and 10.0 mm high and wide. The mechanical energy exerted by the pendulum hammer in the laboratory was 300 joules.

#### 4. ANALYSIS AND DISCUSSION OF RESULTS

This section presents the results obtained regarding the characterization of the material and after conducting the tests. Discussions about these results are also presented, which will lead to the later definition of the conclusions of this work.

The steel used in the tests was ASTM A572 GR 50 with the following chemical composition according to the supplier's data.

Table 1. Chemical composition (% by mass)

C	Mn	Si	P	S	Al	Cr	Nb
0.15	1.36	0.30	0.021	0.006	0.047	0.01	0.023

##### 4.1 Mechanical strength tests

For the first test, the steel was subjected to the impact test using the Charpy type A method. The tests followed the ASTM E23 standards for both specimens under tempering treatment and for normalization. The test aims to analyze the energy absorbed by the materials at room temperature after being subjected to the pendulum hammer, according to the values obtained in Table 2.

After the analysis of the energies absorbed by the material in different conditions, a great improvement in the toughness of the steel can be noticed when subjected to heat treatment, especially for the tempering condition where it was practically twice as much in relation to the steel in normal conditions without any treatment. , this is due to the fact that tempering seeks to mitigate the defects caused by tempering, one of its benefits is the increase in resistance to shock (ASKELAND; PHULÉ, 2008).

The second test consisted of the Rockwell A hardness test along the surface of the specimens for different types of conditions. The recommended and used load was 60 kgf with a diamond penetrator.

According to the analysis of the values, a slight increase in the hardness of the tempered and tempered steel can be noted, which in fact was expected, as according to Callister and Rethwisch (2016) the tempering increases the surface hardness, however it ends up becoming more fragile, for this reason a tempering was done, which ends up reducing the hardness and balancing the other properties, this can be noticed in the impact test where it obtained high values meaning that it had a correct tempering temper. The normalization, which is a pre-treatment aimed at refining the grain, obtained a value below standard steel.

The last test was the tensile test, which was carried out according to NBR 6892-1: 2002, at room temperature with a specific strain rate fixed at 0.0067 s<sup>-1</sup>. Observing the values obtained in the test, the tensile strength gain with the tempering quench condition is noted, a jump from 523 MPa in the untreated steel to 552.1MPa in the tempered and tempered steel, and the yield stress was from 395 MPa in untreated steel to 447MPa in tempered and tempered steel, according to Carmo (2017) the ASTM A36 standard specifies a minimum yield limit, ranging from 245 to 355MPa,

highlighting that tempered steel was above the minimum stipulated by standard, demonstrating that the heat treatment was really effective. However, for the normalization condition, a value well below the standard was obtained, according to Callister and Rethwisch (2016), both the yield strength and the tensile strength limit decrease with increasing temperature; just the opposite is observed for ductility, it increases with increasing temperature, justifying the strain values observed in Table 2 and also in figures 2, 3 and 4 of the tension X deformation curve.

Table 2. Values of the impact, Rockwell A Hardness and tensile test in different conditions

Conditions	Absorbed Energy (Joules)	HRA hardness (Kgf)	Tension strength max (MPa)	Flow tension (MPa)	Deformation (mm)
No treatment	62	51.63	523	395	31
Normalized	112	49.4	475.8	352	35
Tempering temper	120	52.8	552.1	447	28.4

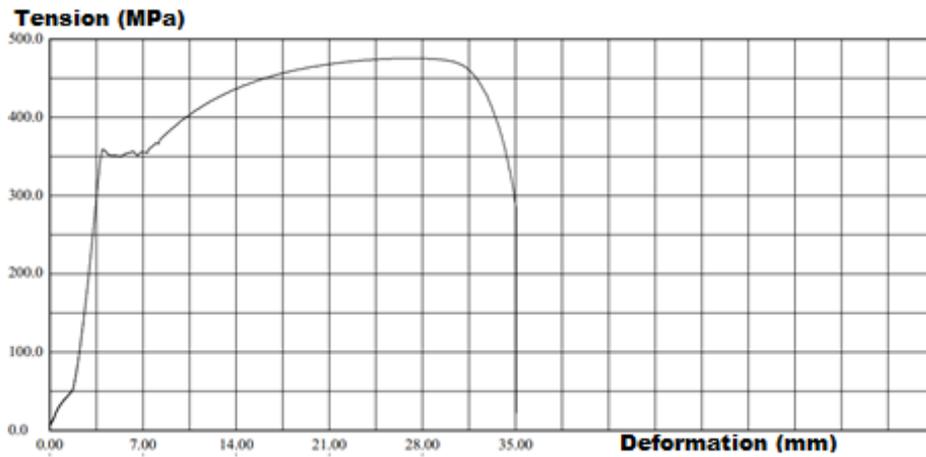


Figure 2. Tension X Deformation Curve for Standard Steel

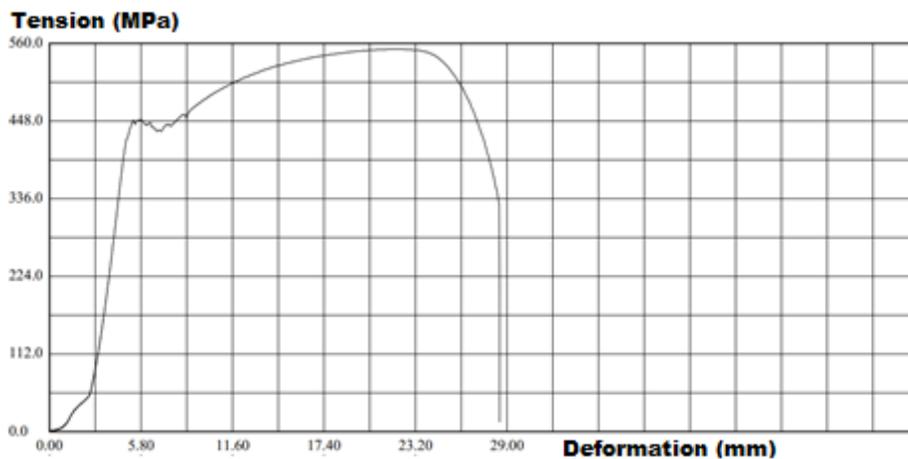


Figure 3. Tension X Deformation curve for tempered and tempered steel

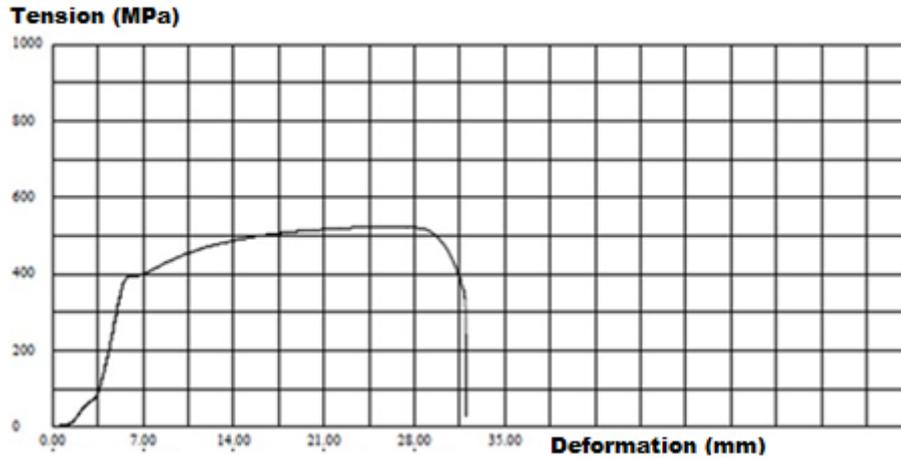


Figure 4. Tension X Deformation Curve for untreated steel

#### 4.2 Microstructural analysis

In order to understand the properties acquired by the specimens, they were subjected to a microstructural analysis, observing microscopically their entire structure in different points of the material. Below figures 5 to 8 are the microstructures in different magnification scales.

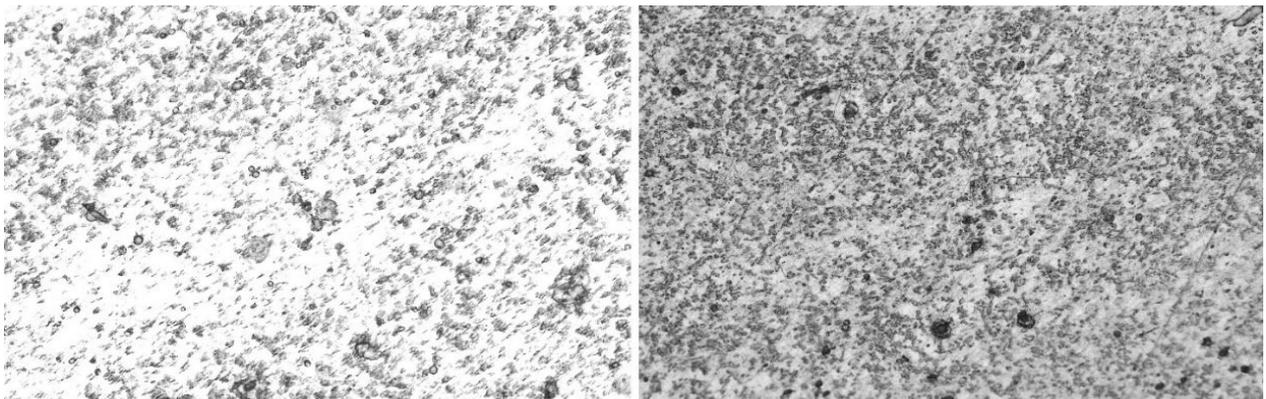


Figure 5. Microstructure of the tempered and tempered specimen 20X

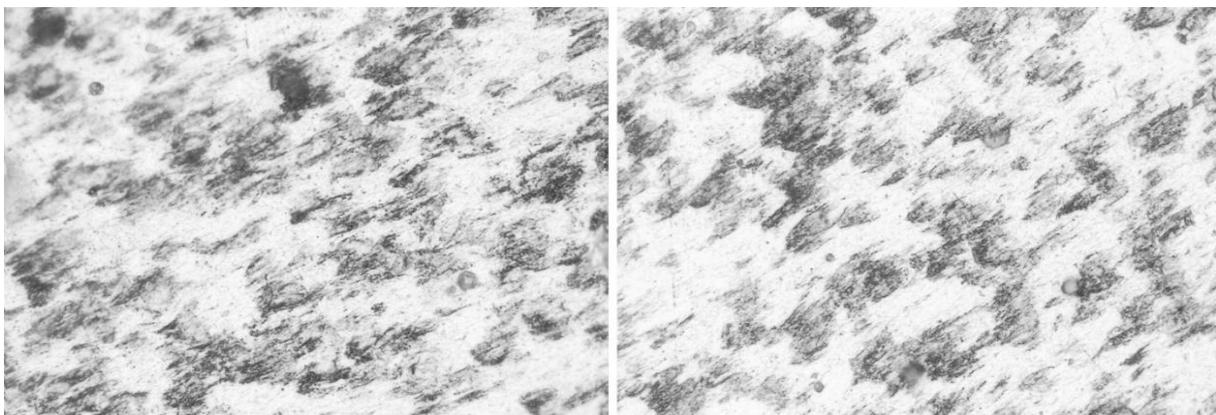


Figure 6. Microstructure of the 50X tempered and tempered specimen

After analyzing the microstructures, we can see that for tempering temper, figures 5 and 6, the predominant structure was tempered martensite, accompanied by small particles of cementite and continuous ferrite matrix, according to Callister and Rethwisch (2016) tempered martenite attenuates the high hardness acquired by tempering substantially

increasing toughness and ductility, the size of the cementite particles influences the mechanical behavior of the tempered martensite, the smaller the better the results will be, and the temperer determines its size.

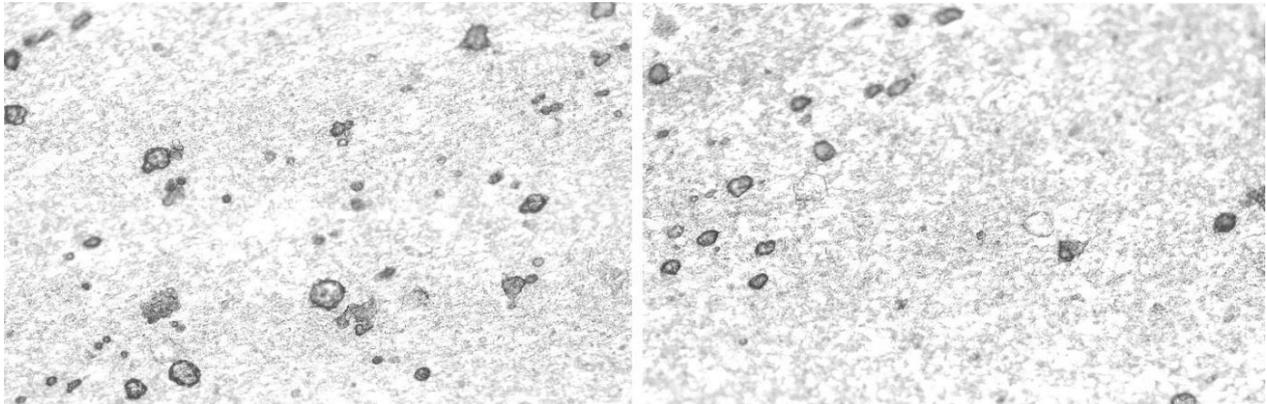


Figure 7. Microstructure of the 20X standardized specimen

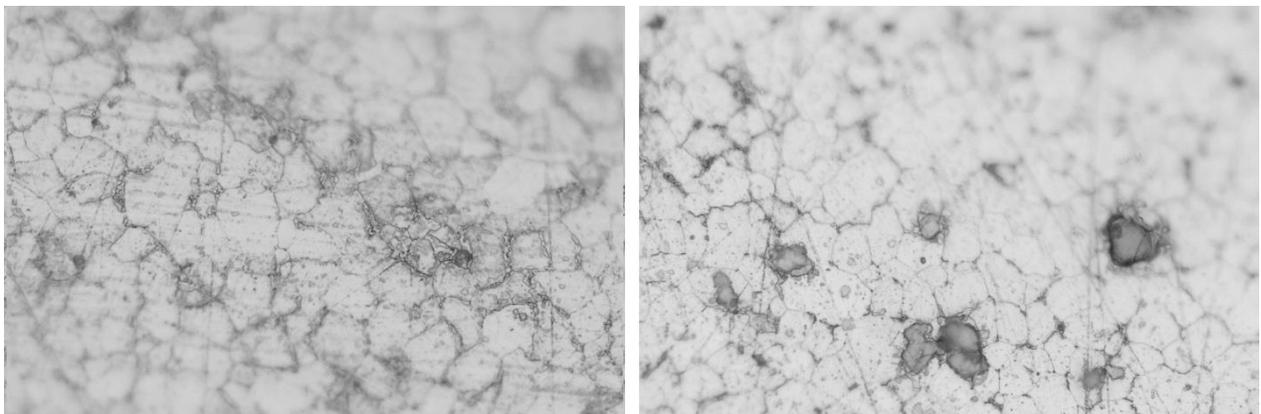


Figure 8. Microstructure of the 50X standardized specimen

For the standardized steel figures 7 and 8, the formation of fine pearlite with ferrite is observed, concretizing the results obtained in the tests, because fine pearlite, despite being harder than spheroidite and coarse pearlite ends up having a lower hardness however, it has better ductility, whereas ferrite is a malleable and more tenacious constituent (ASKELAND; PHULÉ, 2008).

## 5. CONCLUSION

The results of this work demonstrate that ASTM A572 GR 50 steel obtains a considerable gain in strength after being subjected to an appropriate heat treatment.

In the impact test, a great tenacity gain was observed, which can be given by the rearrangement that a correct treatment provides, be it stress relief, adjustments in the grain size and gain of mechanical properties. In the hardness test, the values remained well balanced, with a greater focus on tempering quenching, quenching only increases and the material's surface hardness increases, however the steel ends up becoming more fragile, for that the tempering was done, which seeks to mitigate these inconveniences generated by the material's temper, the hardness results with the tempering at 500°C maintained a medium hardness and balancing the other properties, as was seen in the tensile test, where the tempered and tempered steel obtained a gain on untreated steel.

Although satisfactory results were obtained for tempering treatment, the same was not true for normalization, which did well in terms of toughness and remained in balance in terms of hardness, however it lost a lot of resistance to traction and flow. This is due to the fact that it is basically a pre-treatment that aims at relieving stresses right after being laminated or machined. Another point that explains the results of the tests for normalization is the factor of the amount of carbon that interferes in the obtained properties.

In order to explain the results of the mechanical tests, microstructural analysis of the samples with treatment was performed, for tempering and tempering the tempered martensite microstructure was predominant, showing that the treatment was done correctly, in addition, there was precipitation of small particles of cementite with a matrix of ferrite,

for the treatment of normalization there was a predominance of fine pearlite with a ferritic matrix, structures that prove the mechanical properties lost and acquired by steel in these heat treatment conditions.

It is concluded that the micro-alloyed steel to Niobium further improves its mechanical properties when submitted to the tempering and tempering treatment. The results of the tests confirmed the theory showing that a steel with a small addition of Niobium, combined with a treatment process, obtains satisfactory results in terms of mechanical strength, further increasing its field of applications, in addition to being able to replace less resistant steels in projects, taking into account quantity X resistance.

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