

THE INFLUENCE OF HEAT TREATMENT IN MECHANICAL PROPERTIES AND MICROSTRUCTURE OF THE SAE 1045 STEEL

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Abstract: *The aim of this study is to verify through tensile, hardness and metallography tests the influence of heat treatments of martempering, quenching in brine, normalizing and annealing in the mechanical properties and in the microstructure of the SAE 1045 steel. The results found an alteration in microstructural composition, with an increase of over 350 % in hardness and virtually double the necessary strength to break the test specimens that had been treated. Normalizing didn't show significant increases in hardness nor in the necessary strength to rupture the pieces when compared with the treated ones. Annealing increased the toughness, thus making the annealed more ductile. The metallography was able to correlate the change in the microstructure with the mechanical properties. The result obtained also supports the statement that heat treatment is an excellent method for the alteration of mechanical properties, which justifies the wide range of usage of such material in several kinds of projects.*

Key-words: *heat treatment, mechanical tests and metallography, 1045 steel*

1. INTRODUCTION

Materials are probably more ingrained in our culture than we can imagine. They are closely linked to the emergence and rise of man, from the Stone Ages to the future, with nanotechnology and the appearance of "smart" materials. According to Van Vlack, (1984, p. 15) "materials are undoubtedly, the work substance of our society; they develop an essential function not only in our natural life development, but also, in the welfare and safety of nations".

According to Askeland and Phulé (2012), the comprehension of composition and microstructure of materials, the use of synthesis and the proper processing have enabled the existence of the enormous range of applications for metals and its alloys; always looking for the optimization of the cost-performance ratio, thus the correlation microstructure-composition-property-processing is extremely important in order to obtain a better performance. Ample research allowed the materials to obtain high mechanical resistance, low weight and cost, and to be able to conduct or isolate heat and electricity, being able to work in several kinds of projects.

The main objective of this study is to verify the influence of heat treatment in the mechanical properties and the microstructure of the SAE 1045 steel.

The experimental research is based upon a qualitative and quantitative approach, and at first the study will not deal with the numerical representativity, but with perfecting the compression, and later on to express the relationship between the variables found and through correlation and verification of test results, raising the largest amount of information possible.

The research was divided into five parts, including this one: the literature review was made in chapter 2, where were reported the important concepts in relation to mechanical, metallography e heat treatment tests. In chapter 3 the methodology of the project was introduced, defining and characterizing how the experimental research was structured. In chapter 4, the results collected from the performed tests were introduced, defined and discussed, ending with the conclusion of the study in chapter 5.

2. LITERATURE REVIEW

For better understanding of the terms used, according to the nomenclature here introduced, it is possible to describe shortly some of the important microconstituents of the steel:

Ferrite: is the pure iron or alpha iron (α) structured BCC. It has a property known as allotropy; between 1,391°C and 1,536°C it becomes iron (δ) FCC.

Austenite: also known as gamma-phase iron (γ) structured FCC. It has interstitial solution of carbon in room temperature obtained through fast cooling.

Cementite: it is a compound made out of carbon and iron (iron carbide, Fe_3C). It is the hardest stage of the steel, and, in consequence, very brittle.

Pearlite: consists of alternating layers of ferrite and cementite. Commonly obtained through slower cooling speed. There is a great variety of pearlites by changing the cooling, alloying elements, size of the austenite grain among other factors.

Bainite: aggregates of ferrite and cementite formed in a temperature range located between the formation of the pearlite and the formation of the martensite.

Martensite: supersaturated solid solution of carbon in iron, obtained through fast cooling, with a body-centered tetragonal structure (BCT). It has grains shaped as needles that occur due to the shearing of the austenite lattice and carbon capture, due to high cooling speed.

In order to fully appreciate the nature of the many properties that are sensitive to the microstructure it is essential to know how it is developed. An important tool used is the phase diagram.

2.1 Mechanical tests and metallography

The determination of the mechanical properties of the materials is made through several tests, that may or may not be destructive tests, that causes the sample to break or impossible to use; or non-destructive ones, that aim to point out the quality of the pieces. Among the means to categorize the destructive tests are the tensile, flexural, torsion, fatigue, impact and compression tests, among others. The hardness test, despite not destroying the test specimen is included into this category by many Authors. Among the nondestructive tests, are ones such as X rays, ultrasounds, dye penetrant inspections, and others (SOUZA, 2012).

The tensile test is one of the most common mechanical testing of materials. The machine applies a stress load over the sample at a constant speed, indicating precisely the value of the load and the elongation caused by it (CALLISTER JR., 2008).

The typical result from this test is a stress-strain curve, where the stresses are located in the ordinates and the strains in the abscissas. The stress-strain curve can also be split into two distinct regions: (1) elastic deformation, there is initially, in the curve, the existence of a linear (elastic) region that corresponds to Hooke's law. (2) Plastic deformation, which is the permanent deformation. It is not recovered when the load is removed. The plastic region is a non-linear part generated when the tension exceeds the elastic limit, defined as the material yield strength (YS or σ_y). When this point is reached, and the stress continues to be applied, a phenomenon called work hardening occurs, where the sliding of the dislocations upon the other begins and its cross-section area begins to decrease. The decreasing of the section continues until the fracture (CALLISTER JR., 2008).

The hardness test measures the resistance of the surface of a material to the penetration by a rigid object, which can represent the resistance to risks and can also be a qualitative resistance measurement of the material. The indenter can be rounded or pointed and is made of a material much harder than the sample, for example, hardened steel, tungsten carbide, or diamond (SHACKELFORD, 2008).

According to Van Vlack (1984), even though a material can only contain only one phase, its constitution may imply the existence of different crystallographic orientations. Such single crystals are called grains, and the grain boundaries occurs when there are different orientations. To visualize grain boundaries in metals, firstly the metal is polished, followed by a chemical attack, making the grain boundary visible under the microscope. This technique is called metallography.

2.2 Heat treatments

Heat treatments can be considered the set of operations performed to alter mechanical properties, without altering the geometry of the final product. The main purposes are: internal stress removal, refinement and homogenization of the microstructure, increase or decrease in hardness, increase of mechanical resistance, improvement of ductility, machinability, resistance to wear, corrosion, heat and the electrical and magnetical properties (ASKELAND & PHULÉ, 2012).

Tempering consists of heating the steel until a temperature over the critical zone (austenitization), keeping it for a certain amount of time and next suddenly cooling it in water, oil or air. The cooling rate of a sample depends on the extraction of thermal energy that is closely linked to the size and geometry of the piece, always obtaining martensite in the steel structure. Through fast cooling it is possible to avoid the formation of ferrite, bainite and microconstituents that are softer than martensite. (TSCHIPTSCHIN, [S.D] and SHACKELFORD, 2008). The quenching in brine is performed with fast cooling, aiming to obtain martensite in a solution of unrefined natural salt (NaCl) in room temperature.

According to the aforementioned Authors, after the austenitization is made in a similar manner to tempering, the steel

is immersed in a bath which consists of molten salts. This technique is known as martempering. The steel part is kept in a bath, with a solution whose temperature is located between 150 and 425 °C, until the temperature of the surface and the center of the piece are equal. This treatment aims to minimize the distortions and the residual tension resulting from the differences of temperature in the surface and in the interior of the piece. The final microstructure is martensitic (high hardness e low toughness).

Normalizing is used to refine the grains and produce a more even size distribution. It is obtained through heating to a temperature of at least 55°C over the critical temperature kept during enough time so that the alloy turns completely into austenite. After the process is finished, it is cooled at air-to-room temperature (TSCHIPTSCHIN, [S.D]).

During intermediate and full annealing, a cold-worked piece is made less resistant and more ductile. It is used when an extensive plastic deformation is needed. In this treatment it is allowed that the recovery and recrystallization occur. A microstructure with fine grains is usually desired. After being kept in the furnace at a certain temperature, the steel is cooled inside the furnace, what demands several hours of process (CALLISTER JR., 2008).

3. MATERIALS AND EXPERIMENTS

The method used to carry out the study will be an experimental research, where the data shall be collected from tensile, hardness and metallography tests, before and after heat treatment.

The hardness was found with the help of a durometer model F-26/1 RNS y RNSB produced by OSHMA S.R.L.. Based on the Rockwell scale ABNT NBR NM ISO 6508-1 (2008), the hardness was found in Rockwell C, with a preload of 10 kgf and actual load of 150 kgf, using a diamond cone (120°) as the indenter.

The tensile test was based in the ABNT NBR ISO 6892-1 (2013) standard. The used test machine has a capacity of 100 kN, from the brand EMIC, model DL10000. The elevation speed used in the tests was of 10 mm/min.

The metallography was carried out based in the ABNT NBR 15.284 (1995) standard and followed the following steps for the determination of the microconstituents that were present: cutting, embedment and mounting of the pieces, sanding, polishing, chemical attack and microscopy. A sequence of pieces of emery paper of the following grit sizes: 100, 220, 320, 400 and 600 was used. The surface was later polished using Alumina as an abrasive. To enhance and verify the different stages in microstructure, the boundary contrast is increased through the application of the chemical attack. The surface was submerged in a solution of 2% Nital (2 ml of nitric acid in 98 ml absolute alcohol) being in contact with the surface for about 30 seconds. After the attack, the microstructures are visible through the microscope. The microscope used was produced by Nikon - model Optiphot.

The procedure performed is simplified in the flowchart of Fig. 1:

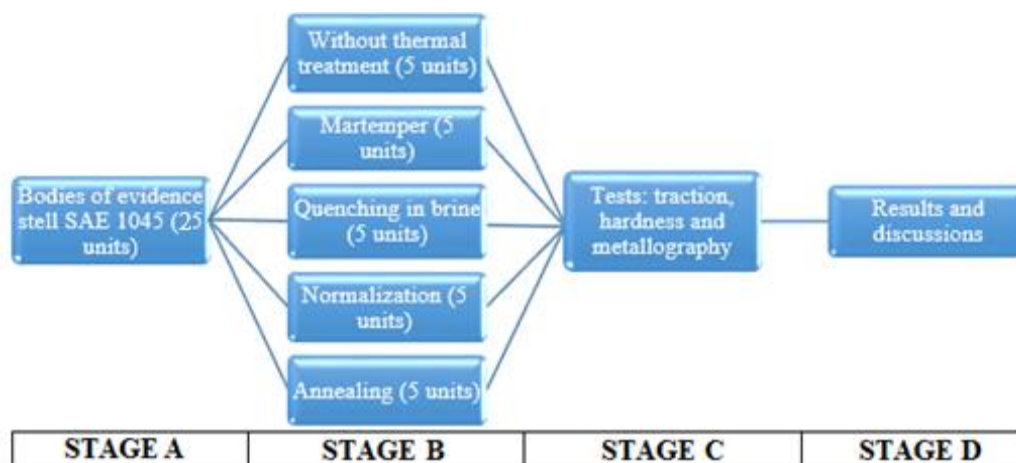


Figure 1 – Flowchart of the methodology used. Source: Author (2015)

According to the flowchart, stage A consists of acquiring and cutting, following the ABNT NBR ISO 6892-1 (2013) standards, the units used in the study. Next, in stage B, heat treatments will be performed, with the purpose of altering the mechanical properties, all following safety and quality standards. The items were divided into 5 batches of 5 units each. Batch 1– no heat treatment; batch 2– martempering; batch 3 – quenching in brine, batch 4 – normalizing and batch 5 – annealing. Stage C consists of performing the tensile, hardness and metallography tests. The tensile test provides us with the curve ($\sigma \times \epsilon$) and the main information necessary to characterize a material. The hardness test will be performed in

Rockwell C, which will allow us to know the hardness of the steel, which is directly linked with the material resistance. In the metallography the pieces are duly polished and a chemical attack is performed to see, with the help of a microscope, the present microstructure. Stage D consists of defining, relating and discussing all the variables and information from the tests.

To start the procedures, the SAE 1045 steel measuring $\frac{1}{4}$ " was bought in one company in Franca, SP. The samples were cut in oxy-fuel cutting by the same company that provided the material. The dimensions of the samples were taken from the ABNT NBR ISO 6892-1 (2013). They are shown in Fig. 2.

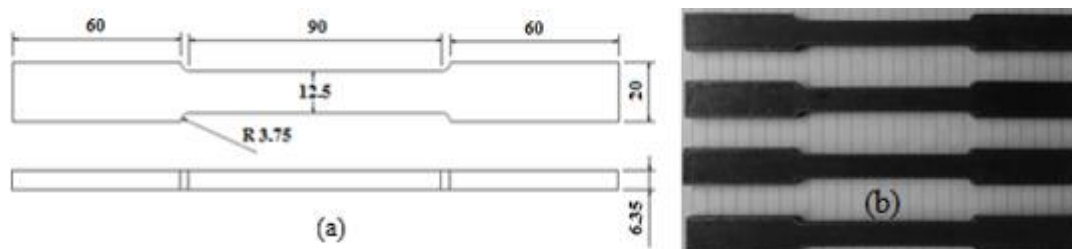


Figure 2 – (a) Picture of specimens and (b) samples used in the project. Source: Author (2015)

In order to analyze the percentage of the chemical elements that were present in the acquired steel, an optical emission spectrometer was used. Table 1 shows the values obtained through the analysis. Several components were found, in small quantities. It is believed that some of these impurities have appeared due to the storage time and the exposure of the sample to the contaminated environment.

Table 1 – Chemical composition of the steel used in the 1045 study (values in percentages). Source: Venturoso Valentini & Cia Ltda (2015)

C	Si	Mn	P	S	Cr	Ni	Al	Cu
0.473	0.157	0.77	0.014	0.0079	0.0016	0.027	0.039	0.01
Ti	Sb	Sn	As	Ca	La	Se	N	Fe
0.0013	0.0032	0.005	0.0058	0.0036	0.0012	0.066	0.017	98.39

The heat treatments were performed in the company Thermix – Sertãozinho, SP. For the subsequent processes, it was used a well-type electric furnace produced by Brasimet. Next are described the temperatures, heating time, cooling time and the conditions which the samples have undergone.

Martemper: The test specimens were heated at approximately 12.4 °C per minute, to the temperature of 930 °C and was kept within this temperature for an hour. Next, the pieces were hoisted and cooled in a solution (a mixture of water and 11% potassium cyanide (KCN), which was at 160 °C was used), for 40 minutes, then cooled in air-to-room temperature. Figure 3 (a) schematically represents the thermal cycle.

Quenching in brine: The pieces were heated to approximately 11.5 °C per minute, to the temperature of 830 °C and was kept within this temperature for an hour. Next, the pieces were hoisted and cooled in brine (a mixture of water, unrefined natural salt (NaCl) and 11% potassium cyanide (KCN), which was at room temperature was used), during 20 minutes. Figure 3 (b) schematically represents the thermal cycle.

Normalization: The samples were heated at approximately 12.4 °C per minute, to the temperature of 930 °C and was kept within this temperature during an hour. Next, the pieces were removed from the furnace and left to cool in still air to room temperature. The estimate time for the cooling was of 3 hours. Figure 3 (c) schematically represents the thermal cycle of normalizing.

Annealing: The samples were heated at approximately 10 °C per minute, to the temperature of 630 °C and was kept within this temperature for two hours. Next, the pieces were slowly cooled inside the furnace. The estimate time for the cooling was of 5 hours.

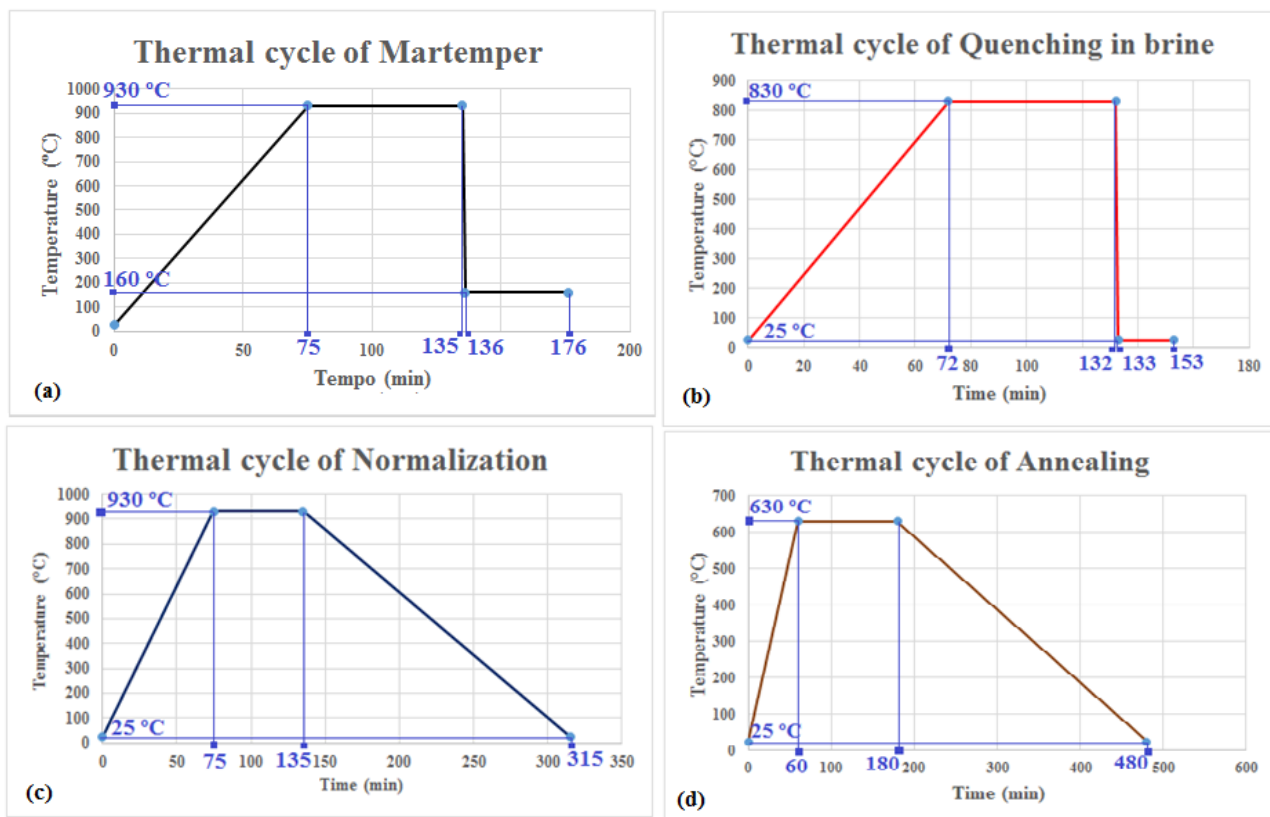


Figure 3 – (a) Martemper. (b) Quenching in brine. (c) Normalization. (d) Annealing. Source: Thermix (2015)

4. ANALYSIS OF RESULTS

4.1 Hardness

Three hardness measurements were performed in each sample. Table 2 shows the average values found. All the values were obtained in HRC, except for the samples that have undergone annealing, because these presented an extremely low hardness. To measure the hardness of the annealed, it was used Rockwell B scale, indenter used: diamond sphere with 1/16" in diameter, preload of 10 kgf and load of 100 kgf.

Table 2 – Average hardness values. Source: Author (2015)

Test specimen	No treatment	Martemper	Quenching in brine	Normalization	Annealing
1	17.2	64.9	59.8	19.8	49.7
2	17.5	65.4	59.2	22.1	58.8
3	16.5	67	54.7	21.5	67.6
4	18.3	67.3	59.4	25.8	63.8
5	14.6	66.8	54.3	21.4	54.6
General Average	16.8 ± 0.43	66.3 ± 1.18	57.5 ± 2.90	22.1 ± 1.84	58.9* ± 2.20

* Value in HRB.

The tempering performed were the ones that obtained significant amounts in hardness when compared to the items with no treatment. Martempering and quenching in brine have increased approximately 395 and 342 %, respectively.

Normalizing obtained a hardness similar to the items with no heat treatment. A small enlargement of it was also

noticed.

Annealing made the pieces extremely low in hardness, making it impossible to make a comparison of the hardnesses. According to the hardness conversion tables and the standards used to perform the test, the value would be below zero in the HRC scale.

The results show that tempering adds great hardening to the steel. It is also noticed the importance of the treatments in improving and changing the properties of the materials.

4.2 Traction

An average of the five test specimens used in each heat treatment was made. Table 3 shows the average of the values of maximum strength (F) % in elongation (defined by Eq. (1)), ultimate strength (US) e yield strength (YS) obtained in the tests.

$$\Delta l = \frac{l - l_0}{l_0} \times 100 \quad (1)$$

Where l is the final length and l_0 the initial length. In average, to break the martempered and the quenched in brine samples it was needed, approximately, 83.5 kN and 92.3 kN. It was noticed 65% and 84% more in strength than the items without treatment, respectively. Consequently, the elongation was lessened in half when compared with the pieces with no kind of treatment, which made the items to become brittle. For the normalized specimens the average maximum strength was of approximately 61.8 kN, an increase of 22.3 %. The elongation percentage was of 16.6, needing 23.8% less when compared with the ones with no heat treatment. As for the annealed test specimens the average of maximum strength was approximately 37.3 kN. The elongation percentage was of 23.2, making the pieces more ductile than all the others.

Table 3 - Average results of tensile tests for different heat treatments. Source: Author (2015)

Test specimens	Max. strength (N)	Elong. (%)	US (MPa)	YS (MPa)
Without treatment	50,556 ± 2,175.2	21.8 ± 0.8	636.9 ± 27.3	473.1 ± 27.3
Martemper	83,584.4 ± 5,802.2	10.2 ± 0.5	1,053.2 ± 73.1	1,017.8 ± 72.8
Quenching in brine	93,247 ± 14,507.2	11.3 ± 2.2	1,174.8 ± 182.8	961.4 ± 232.9
Normalization	61,803 ± 1,844.2	16.6 ± 2.1	779.1 ± 23.2	422.3 ± 15.9
Annealing	37,288 ± 2,640.3	23.2 ± 2.8	469.8 ± 32.2	295.3 ± 20.7

The YS/US ratio was calculated as one more indication of how close the yield strength is from the ultimate strength. The higher this value, the lower the elongation. Figure 4 shows the relation of the results of the YS/US ratio. For the test specimens with no treatment the ratio was ≈ 0.629 . For the tempered samples the ratio was the highest of all, confirming one more statement that the material lost ductility. For the normalized pieces the ratio was ≈ 0.542 , the lowest ratio compared to all processes. However, those were not the ones that have elongated the most. For the annealed samples, the ratio was practically the same found in the samples without any kind of heat process and these were the materials which have elongated the most.

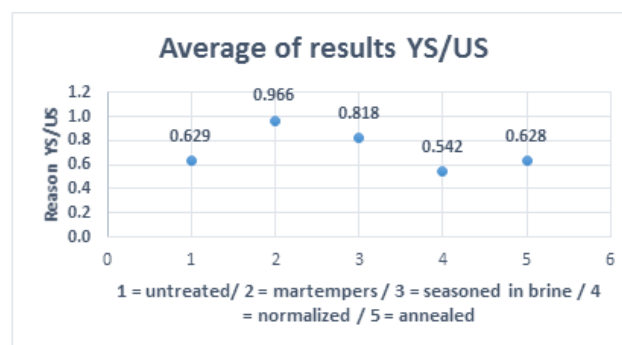


Figure 4 – Average results from the YS/UTS ratio found in the tensile tests. Source: Author (2015)

4.3 Metallography

The metallography was performed in the company Qualyservices: Soluções em Soldagem e Ensaios Tecnológicos Ltda, located in Sertãozinho-SP. The qualitative and quantitative analysis of the pieces was carried out. The microscopy was executed in a magnifying of ten times (100 x) so that the comparative method in the evaluation of the grain sized based on the ASTM E112 and NBR 11.568 (1990) standards can be used.

In Figure 5 are the pictures of the microstructures that were present in each sample, following the respective order: untreated, martempered, quenched in brine, normalized and annealed; all according to the references.

Figure 5 (a) is the microscopy of the piece with no heat treatment. There can be noticed the presence of a microstructure which consists of pearlite (dark part) with small lamellae and the small presence of proeutectoid ferrite in the grain boundaries (light part). In Figures 5 (b) and (c) the presence of martensite is clear. It shows that the cooling in martempering and quenching in brine happened in a sudden manner, enabling the formation of martensite, which is the hardest structure. The shape of needle occurs due to the shear of the austenite lattice and the carbon capture, due to the high cooling speed.

In Figure 5 (b) is represented the picture of the microstructure of the sample treated with martempering. It can be observed that the microstructure consists of martensite in a very acicular aspect (needle shape). In Figure 5 (c) there is the test specimen quenched in brine. It can be noticed that the microstructure is constituted by fine martensite.

In Figure 5 (d) it can be observed the microscopy of the normalized sample, where the microstructure consists of proeutectoid ferrite (light part) + pearlite with fine lamellae (dark part) with grains of size 5 and 6 predominantly.

In Figure 5 (e) it is possible to view the microscopy of the annealed sample, with a microstructure which consists of ferrite and pearlite decomposed in globular cementite. Ferritic grains size 7 predominantly.

Annealing is a cooling process slower than normalizing. Thus, there was more recrystallization of grains, in other words, the rearrangement and increase of the pearlite grain. Normalizing had a faster cooling time. Less recrystallization time shows smaller grains. The difference in the sizes of grain can be noticed in the normalized sample (5 and 6 predominantly) and annealed (7 predominantly). The lower the grain size, the bigger the hardness. Consequently, it will be a less ductile material. It is a plausible result because it thus shows the lowest hardness in the annealed sample, and consequently, more ductility.

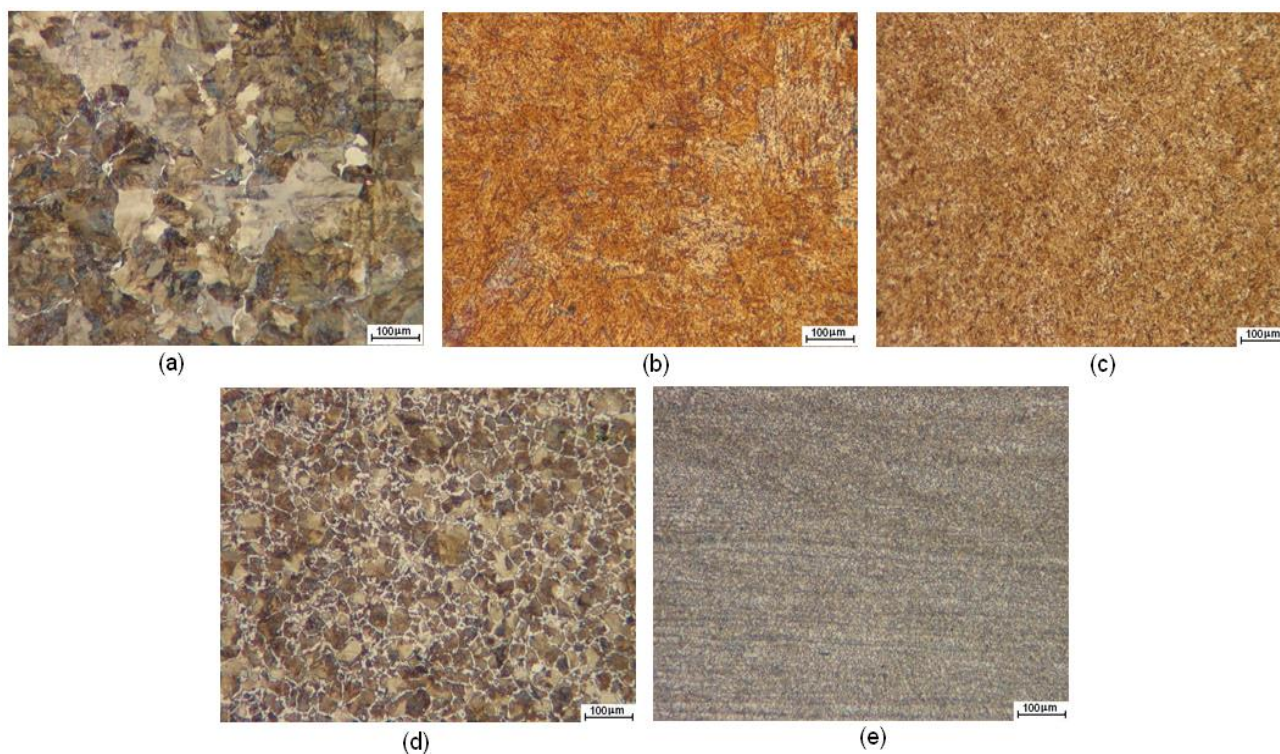


Figure 5 – Micrographs. Nital 2%. (a) no heat treatment, (b) martemper, (c) quenched in brine, (d) standard and (e) annealed. Source: Qualyservices (2015)

5. CONCLUSIONS

The results found show an alteration in the microstructural composition as a result of the treatments, with an increase of over 350 % in hardness and virtually double the necessary strength to rupture the test specimens treated with tempering. The processes of martempering and quenching in brine made the samples brittle, therefore, the pieces that needed more strain to be fractured, consequently, had less elongation. Normalizing has homogenized the microstructure of the samples and did not show significative results nor in hardness nor in the necessary strain to rupture the pieces, when compared with the tempered ones. Annealing has increased the toughness making the items more ductile, and consequently with extremely low hardness. Annealing is a slower cooling process than normalizing, and thus, had greater recrystallization of grains, so, one rearrangement and increase of pearlite grains. Normalizing had a faster cooling time, with less recrystallization time produces smaller grains, consequently greater hardness and therefore it will be a less ductile material. The metallography was able to prove the alterations in the microstructures with the mechanical properties resulting from each process. The result obtained also supports the statement that the heat treatment is an excellent method for altering the mechanical properties, thus justifying the wide range of usage of such material in several kinds of projects.

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