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Microstructure and mechanical properties during annealing of cold-drawn pearlitic steel wire

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Abstract: The main of this work was to evaluate the microstuctural evolution and the mechanical properties of SAE 1070 steel subjected to a thermomechanical process. The steel was wire drawn up to 12 passes with average reductions between 15 and 21%. The accumulated deformation was 2.52. Following, the material was annealed in ferritic field at the temperatures of 400 ° C, 500 ° C, 600 ° C and 700 ° C, during 5, 10, 15, 20, 25 and 30 min. The microstructural characterization was performed with scanning electron microscopy (SEM) and atomic force microscopy (AFM). The mechanical behavior was analyzed from the results of the tensile test and hardness Vickers test conducted in both, deformed and annealed, material. The microstructural characterization of the deformed material showed the formation of the curling structure. The transition from the pearlitic structure to a composite structure of globular cementite particles dispersed in a ferritic matrix was observed in the annealed material. The spheroidization process during the annealing was intensified by the fragmentation and partial dissolution of the cementite resulted of the drawing process. The material strength increased from 1028MPa to 2045 MPa after the drawing process and at the end of the annealing at 700ºC/30 min the value was 694 MPa. The average reduction in the hardness values for the same annealing time was 7% between 400°C and 500°C, 25% between 500°C and 600°C, and finally, 28% between 600°C and 700°C. The changes in the microstructure during the heat treatment reflected in the mechanical properties.

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I. INTRODUCTION

Pearlitic steel wires produced by cold-drawn are often applied as structural materials due to their high strength associated with acceptable levels of ductility and toughness [1]. This combination of properties is attributed to the lamellar structure where the constituent with high hardness is incorporated in the ductile constituent [2]. The most common applications of this material are cables for suspension bridges, steel wire rod for tires and piano strings [3]. For this reason, the microstructural evolution during the processing of the pearlitic steels has been, for many years, a subject of considerable scientific research [4].

The control of the parameters that influence the thermomechanical processing has a fundamental importance in guaranteeing the final characteristics of the product. The complex relationship between microstructure, texture and chemical composition determines the mechanical properties of the material during its processing [5]. Pearlitic steel subjected to the wire drawing with high accumulated deformation presents a structure called "curling" [8]. The curling structure is attributed to the deformation under stress condition which is associated with the texture <110> development during wire drawing [9]. The non-deformed perlite structure, the cementites lamellae are relatively straight and tend to rotate around the wire axis during the initial stages of the deformation process. It results in a curvilinear microstructure and a defined crystallographic texture [10]. This curvature results in a radial orientation in the direction <001> [11].

During the annealing treatment of a deformed material, the phenomenon of recrystallization nucleates preferably in specific regions of the microstructure. During the annealing of a drawn pearlitic steel, the restoration phenomena are directly influenced by the microstructure and texture characteristics of the curling structure, that is, by regions with certain orientation. The growth capacity of the nuclei is also influenced by the orientation of the adjacent regions, thus, nucleation and growth characteristics ensure the formation of a re-crystallization texture [7].

In this context, the objective of this work was to characterize the microstructural evolution of the SAE 1070 pearlitic steel when subjected to a thermomechanical processing. The tensile test and hardness test were conducted to evaluate the mechanical properties.

II. MATERIAL AND METHODS

The material used in this study consisted in SAE 1070 steel wires with chemical composition of 0.712C, 0.01S, 0.489Mn, 0.007P, 0.225Si, 0.003Al, 0.016Cr, 0.009Cu, 0.006Ni, 0.005Mo, 0.01N, expressed in wt%. The as received material with 5.50 mm diameter was forming by hot rolling with continuous cooling. The steel wire was cold drawn in 12 passes with reduction rate of 15 to 20% reaching a diameter of 1.55 mm. Following, the samples were annealed in a tube furnace at 400ºC, 500ºC, 600ºC e 700ºC during 5, 10, 15, 20, 25 e 30 minutes without atmosphere and cooling rate control. The microstructural characterization was carried out by scanning electron microscopy (SEM) and atomic force microscopy (AFM). The Vickers hardness test was executed in a Shimadzu Micro Hardness Tester (HVM-2T). The test force was 300 gf during 30s. Tensile tests were conducted in a 1 ton universal testing machine EMIC at a tensile speed of 1mm/s.

III. RESULT

Figure no1 shows the microstructure of the deformed material.

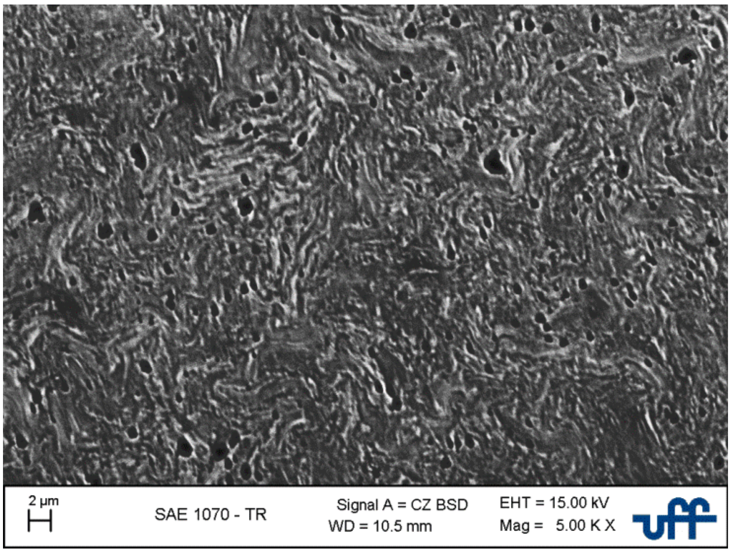

Figure no1: SEM. Cross section of SAE 1070 steel cold-deformed with accumulates strain of 2.52.

Figures no 2- no 5 show the microstructure of the annealed material at 400°C, 500°C, 600°C and 700°C, respectively, during the times of 5 to 30 min.

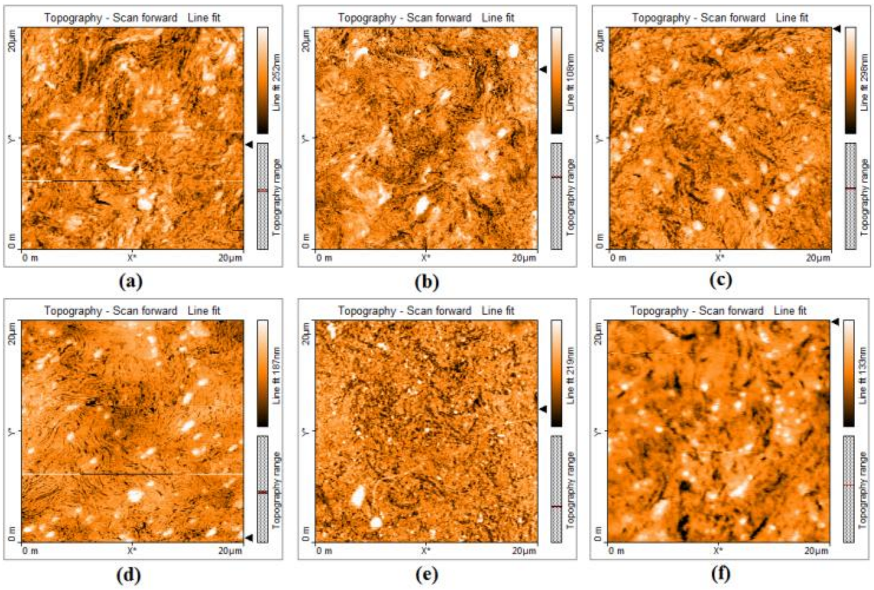

Figure no 2: AFM. Micrograph of SAE 1070 steel annealed at 400ºC during (a) 5min, (b) 10min, (c) 15min, (d) 20min, (e) 25min, (f) 30min. Analyzed area of 400 μm^2.

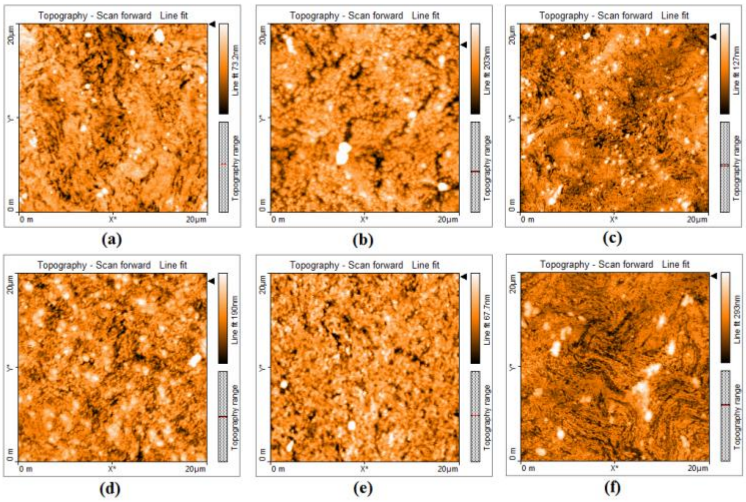

Figure no 3: AFM. Micrograph of SAE 1070 steel annealed at 500ºC during (a) 5min, (b) 10min, (c) 15min, (d) 20min, (e) 25min, (f) 30min. Analyzed area of 400 μm^2.

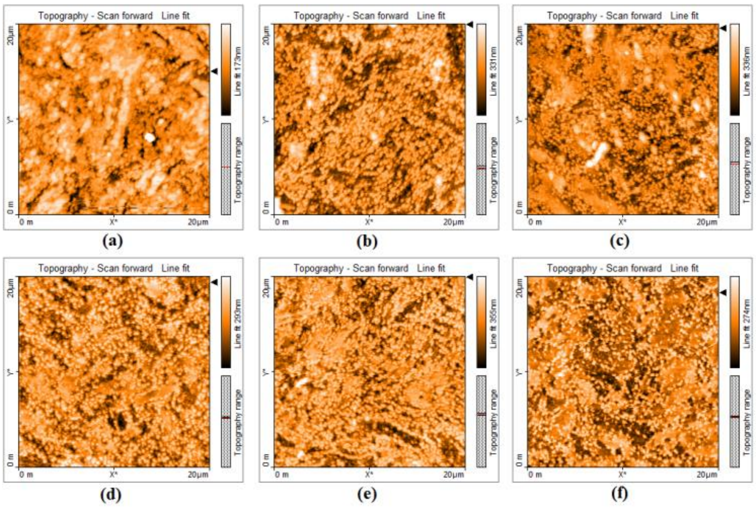

Figure no 4: AFM. Micrograph of SAE 1070 steel annealed at 600ºC during (a) 5min, (b) 10min, (c) 15min, (d) 20min, (e) 25min, (f) 30min. Analyzed area of 400 μm^2.

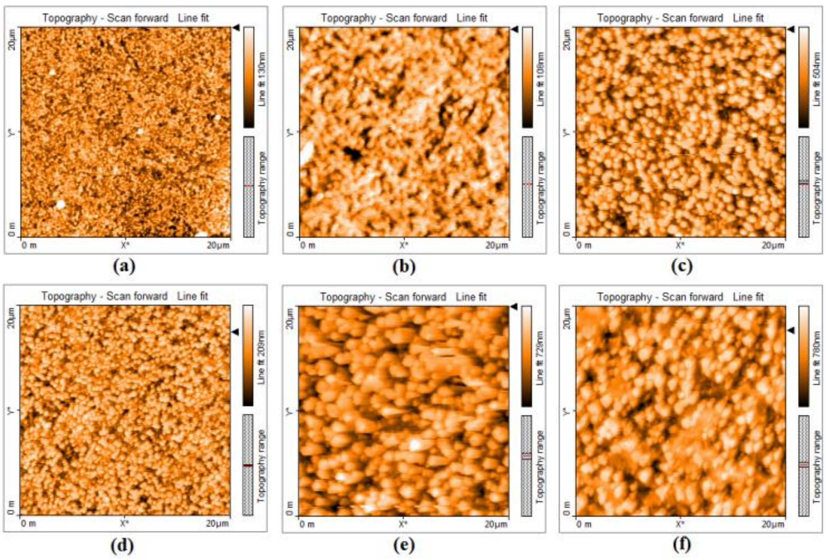

Figure no 5: AFM. Micrograph of SAE 1070 steel annealed at 700ºC during (a) 5min, (b) 10min, (c) 15min, (d) 20min, (e) 25min, (f) 30min. Analyzed area of 400 μm^2.

Figure no 6 shows the micrograph of the annealed material at 600 ° C for 30 min at a magnification of 5000x.

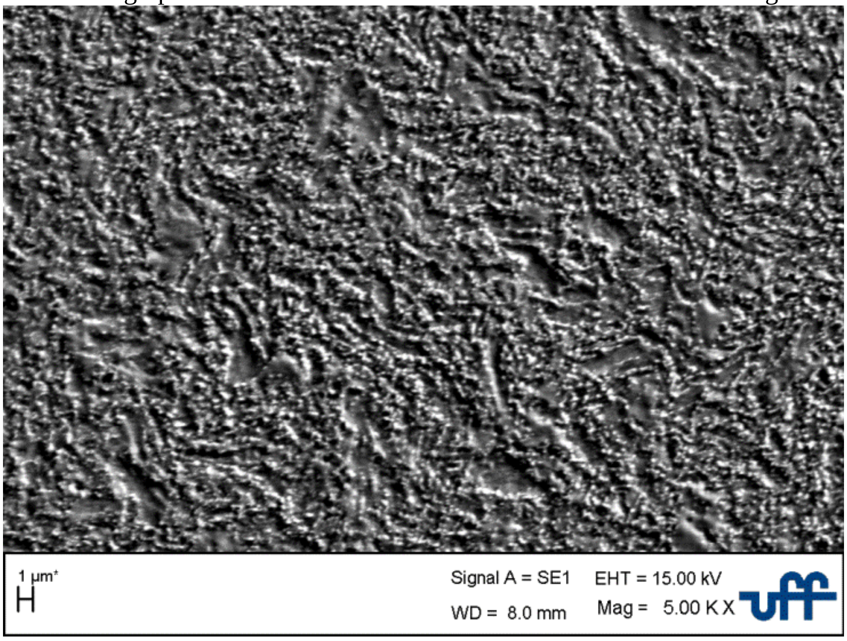

Figure no 6: SEM. Cross section of SAE 1070 steel annealed at 600ºC for 30 min.

The mechanical properties of the deformed material was an ultimate tensile strength of 2045 MPa, yield strength of 1886 MPa and elongation percentage of 0,98%. Table no 1 and Figure no 7 show the results of the tensile tests conducted on the annealed material at the times of 10, 20 and 30 min.

Table no 1: SAE 1070 steel mechanical properties after annealing.

Temperature (ºC)	Time (min)	Ultimate tensile strength (MPa)	Yield strength (MPa)	Elongation (%)
400	10	1995	1863	3,3
	20	1951	1837	2,4
	30	1809	1671	2,9
500	10	1603	1503	2,1
	20	1530	1436	1,8
	30	1487	1389	2,0
600	10	1124	1000	1,7
	20	1093	980	1,9
	30	1007	912	1,5
700	10	811	745	2,1
	20	738	676	2,4
	30	694	500	3,2

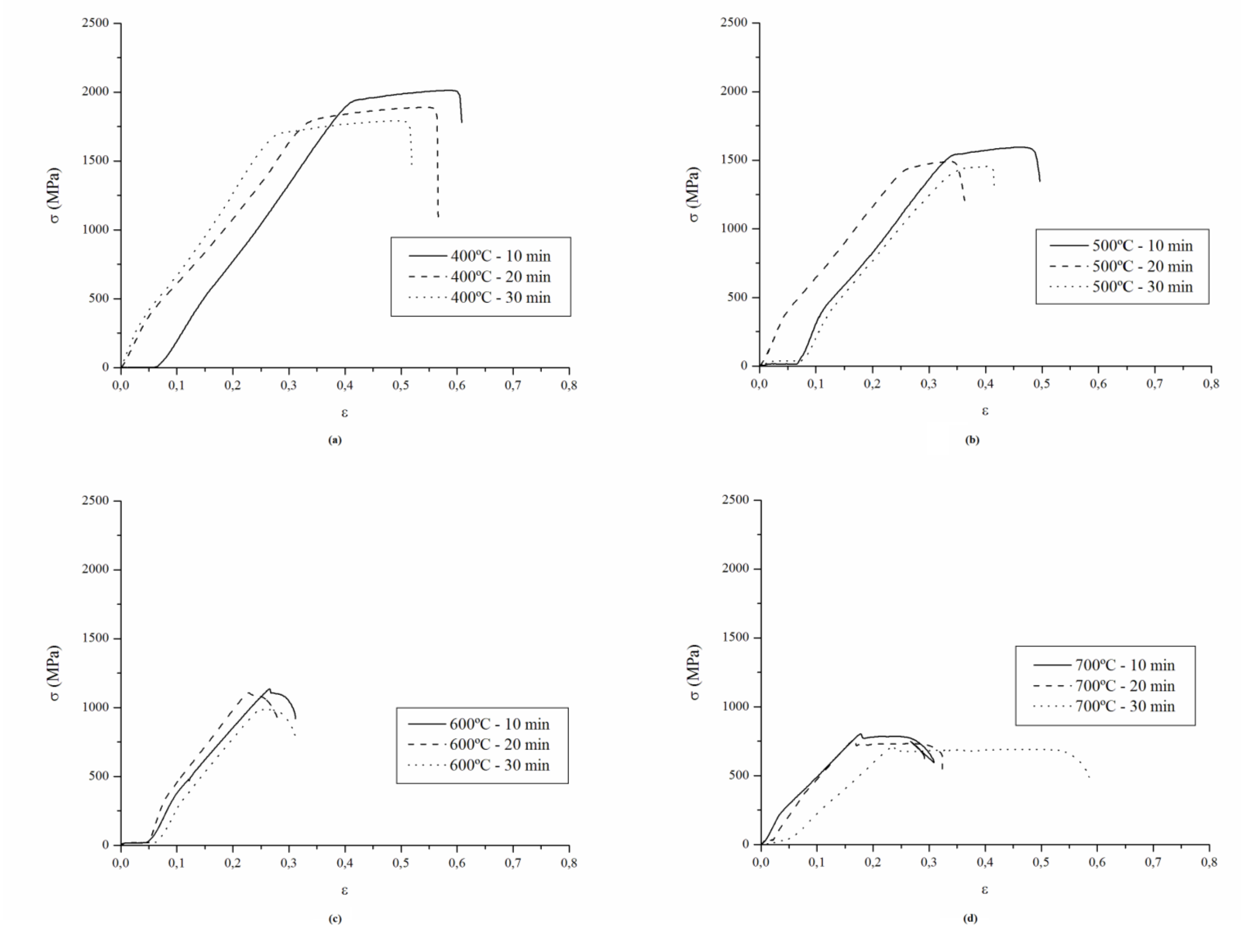

Figure no 7: Stress – strain curves of SAE 1070 steel annealed at (a) 400ºC, (b) 500ºC, (c) 600ºC and (d) 700ºC.

The hardness of the wire drawing material was 561 Vickers. Table no 2 and Figure no 8 show the Vickers hardness values of the material as a function of time and annealing temperature.

Table no 2: SAE 1070 steel Vickers hardness.

Temperature (ºC)	Annealing time (min)					
	5	10	15	20	25	30
	Vickers hardness					
400	542 ± 11	547 ± 10	538 ± 8	535 ± 5	531 ± 11	532 ± 7
500	526 ± 14	480 ± 5	504 ± 6	493 ± 7	493 ± 9	505 ± 6
600	432 ± 9	362 ± 14	353 ± 6	376 ± 7	365 ± 12	351 ± 11
700	328 ± 13	304 ± 12	244 ± 4	280 ± 11	234 ± 7	226 ± 4

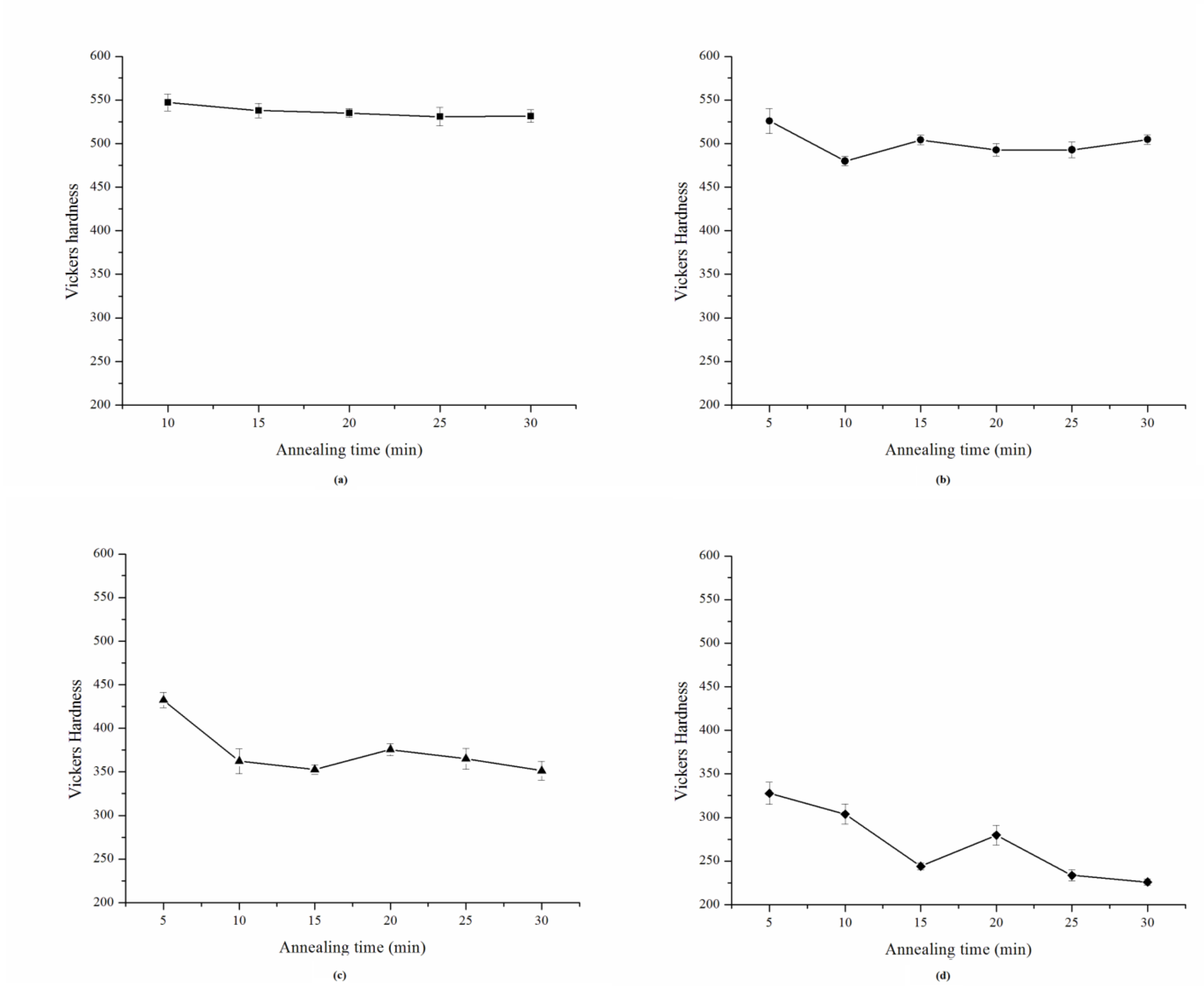

Figure no 8: SAE 1070 Vickers Hardness evolution annealed at (a) 400ºC, (b) 500ºC, (c) 600ºC and (d) 700ºC

IV. DISCUSSION

The deformed material in Figure no 1 is characterized by the curling microstructure. This structure is typical of pearlitic steels subjected to high levels of strain during de wire drawing. In this structure, the thicknesses of the ferrite lamellae are reduced, bended and/or twisted depending on their position relative to the drawing direction. While, the cementite lamellae are a hard and fragile constituent of the pearlite structure, the majority were fragmented, due to the level of deformation.

The annealing at 400ºC still in the lowest range was able to restore the microstructure of the material significantly, as observed in Figure no 2. However, some lamellar regions (fragmented and twisted) still remain, but with reduced thickness. The thin thickness of the lamellae is attributed to the low temperature and the short annealing time, which were not enough to make them thicker [12]. Temperature and annealing time are critical factors for interlamellar spacing. The diffusion phenomenon is the mechanism responsible for the growth of the lamellae. Thus, low temperatures reduce diffusion potential and short-term heat treatments prevent the process complete [13].

The temperature of 500°C was marked by the transition of the microstructure of the material. The morphology of cementite became globular, called spheroidized cementite. During this temperature range it was possible to observe the beginning of the change of the pearlite microstructure to a microstructure characterized by spherical particles of cementite dispersed in a ferritic matrix. It was verified that in this temperature range there are regions of the material that present perlite colonies, spheroidized cementite and the transition between them. This randomness in the microstructure during the annealing at 500 ° C is due to the fact that the heat treatment is not homogeneous throughout the entire thickness of the material, especially for short-term anneals such as those employed in this work.

The first minutes of the treatment at 600ºC clearly showed the transition from the pearlite structure to the formation of spheroidized cementite. It was observed that the remaining lamellar regions are constituted of spherical nanometric cementite particles. Similar behavior was observed in the work of Lv [14], where cyclic thermal treatments were performed on a 0.8% p. He verified that the heat treatment at 710 ° C for 3 min it was possible to identify the spheroid formation process by means of fragmented cementite lamellae. These spheroidized cementite particles were adhered to the lamellae, others at their extremities, and some particles were isolated. During the heat treatment, the microstructure was completely spheroidized. However, it was observed a higher density of spherical particles in the samples annealed during 20 and 25 min, respectively.

To further observe this microstructure, Figure no 6 shows the micrograph of the annealed material at 600 ° C for 30 min at a magnification of 5000x. Note that, the ferrite, as it is the most ductile constituent, presents a greater depth in the image due to the metallographic preparation. The cementite, harder and more resistant, appears prominent in the images obtained with secondary electron. In this way, it is observed that the elevated regions are composed of small spheres which are represented in the image by the white color, while the ferritic matrix has a flat topography and gray color. In the treatment at 700ºC the material presented a microstructure constituted by the spheroidized cementite in all the annealing ranges performed. It was also observed that the increase in the annealing time caused a gradual increase in the size of the globular cementite.

The ultimate tensile strength and yield strength decrease linearly with the increasing of temperature and annealing time as we can note in Table no1. It was observed that, even with a lower annealing time, the higher temperature led a reduction of the tensile strength and yield strength compared to the lower temperature and longer annealing time. The same behavior was not observed in the elongation percentage, which presented an irregular reduction up to 600ºC and increased considerably to 700ºC, as the annealing time also increased.

According to Bruckner [15], the mechanical behavior of materials with a lamellar and spheroid structure is different, especially when they are subjected to tensile stress, due to the way cracks appear before the fracture. Complementarily, Song [16] verified that the morphology of the cementite has complex effects on the mechanical properties of the material. The cementite in the form of thick lamellas or spheroids is considered the preferred site for the occurrence of damages and worse microstructural characteristic to steels. This occurs as a function of the low adhesion of the cementite in the ferritic matrix and the difference between the elastic-plastic behavior presented by these two constituents [17].

Figure no7 allowed observe that the material presented low ductility at 500ºC and 600ºC. The transformation in the microstructure and the change in the morphology of the cementite, which interact differently with the dislocations and other microstructural elements, are some of the factors that make the material less ductile. In general, a gradual reduction of the level of the plastic deformation supported by the material was observed during the treatment. This result is the opposite to the expected one, since the increase of annealing time and temperature, potentiate the restoration phenomena, which are responsible for the reduction of the strength and increase of the ductility of the material. The ultimate tensile strength decreased with the increasing of temperature and time. Song [16] states that steels consisting of globular cementite particles dispersed in the microstructure show high ductility since these particles improve the hardening capacity. The high volumetric fraction of thin dispersed cementite particles effectively increases the rate of hardening as it promotes the accumulation of discordance around the particles.

Thence, it is possible to state that the spherical cementite particles dispersed in the material interact with the dislocations during a cold deformation in order to increase the hardening capacity and ductility of the material. When the material is subjected to a heat treatment, stress relief and rearrangement of discordance is enhanced by the presence of these particles, making the material less brittle and therefore more resistant.

At 700ºC, the curves present significant differences between them, mainly the 30 min curve compared to the others. It was observed that for this range, the material exhibited a high deformation close to the value of the material annealed at 400°C for 10 min. Comparing the two curves, 400ºC - 10min and 700ºC - 30min, that is, the two extremes of the heat treatment, we notice the same level of deformation at different stress. The material annealed at 400ºC during10 min presents an ultimate tensile strength of 1995 MPa and an elongation percentage of 3,3%, the one annealed at 700ºC during 30 min exhibits an ultimate tensile strength of de 694 MPa and an elongated percentage of 3,2.

Besides the difference between the resistance values, the graphs presented in the Figures no 7A and 7D allow inferring that the deformation levels are close. However, for annealed material at 400 ° C for 10 min, most of the deformation is elastic, while for the material annealed at 700 ° C for 30 min the elastic deformation is much smaller than plastic. That is, at 400ºC the deformation level is due to the high stress load that the material supports, already at 700ºC this same deformation is attributed to restored ductility. The results of the tensile tests are in agreement with those presented in the literature. In the pearlite structure, the ferrite is separated by the brittle and hard cementite which prevents cooperation during deformation. Because of this, the lamellar structure is unable to offer high plasticity [14]. On the other hand, the material with a spheroidal structure may have higher values of plasticity and elongation, as shown in the studies in [18,19]. The size of the spheroids also influences the mechanical properties of the spheroid steels, where the elongation increases proportionally with the growth of the spheroids [14].

It was observed in Table no 2 and Figure no 8 that the hardness does not present a linear decrease behavior as a function of the annealing time at any of the temperatures. Also, some samples presented higher hardness at a higher annealing time, when what were expected was the reduction of the hardness with the increase of the annealing time. This reduction was expected due to the restoration phenomena that occur gradually as the annealing time and temperature increases. However, by comparing hardness values between temperatures, it is noted that there is always a reduction. The average reduction in the hardness for the same annealing time is 7% for 400°C and 500 ° C, 25% for 500°C and 600°C, and finally, 28% for 600°C and 700°C. In this way, it is concluded that temperature is the predominant factor responsible for the reduction of hardness in relation to the annealing duration, and that the greatest reductions in hardness values occur at temperatures of 600ºC and 700ºC.

The hardening mechanisms for the materials with perlite and spheroidized structure present some differences. According to Zelin [11], the hardening of the pearlitic steels is caused by the reduction of the thickness of the lamellae, the increase in the volumetric fraction of the interfaces and the increase in the density of dislocations that form walls of dislocations of the same width of the inter-lamellar spacing. The hardening of spheroid steels is characterized by a set of factors that mutually contribute to the increase in hardness as the level of stress increases [14,20]. In agreement to Xiong et al. [20], the combined effect of cementite particles, grain boundaries, and subgrain boundaries influence, directly, the mechanical properties of spheroidal high-carbon steels.

Correlating the results obtained with the tensile strength and the microstructural evolution, it was observed that at 400ºC, temperature which the material presents a pearly microstructure, the hardness remained close and a slight tendency to decrease along the time. Still at this temperature, the tensile tests presented a constant drop in the tensile strength and yield strenght. These results indicate restoration phenomena, which reduce strength, hardness and increase the ductility of the material, since in this temperature range there are no changes in the mechanical properties due to the transition influence of the microstructure. For the range of 500ºC and 600ºC the microstructure was characterized by a transition of the pearlite structure to a structure composed of globular cementite dispersed in a ferritic matrix. Thus, the random behavior of the percentage elongation as a function of the annealing time observed corroborates the hardness values for the same temperature range, as well as the significant drop in ductility. Similar results were reported by Saha [21], who subjected pearlitic steel to cyclic thermal treatments in order to obtain a spheroidized structure. It was verified that the inicial reduction of the elongation and ductility was due to the formation of a fine structure and then the growth was a result of the elimination of perlite in the microstructure. At the temperature of 700ºC, it was observed a growth of the cementite globules sizes, as well as an increase of the percentage elongation and the ductility of the material, accompanied by a sharp decrease of the hardness during the annealing time.

It is believed that the spheroidization process occurred quickly due to the previous deformation suffered by the material. According to Baranova and Sukhomlin [22], cold deformation generates defects in the ferrite and cementite structures that affect the mechanism and kinetics of spheroidization of pearlite. Under the influence of the previous cold deformation, the process of separating the cementite particles occurs more easily during annealing of the steel. This spheroidized microstructure is responsible for increasing both the ductility of the material and the toughness [16].

V. CONCLUSION

The pearlite microstructure evolved into a structure composed of globular cementite particles dispersed in a ferritic matrix. The spheroidization process was potentiated due to fragmentation and partial dissolution of the cementite during wire drawing. The tensile tests revealed a gradual reduction of the ultimate tensile strength and yield strength resistance as a function of time and annealing temperature due to restoration phenomena. The elongation percentage presented similar values at 400ºC and 700ºC. However, the deformation was predominantly elastic at 400ºC and plastic at 700ºC. The hardness of the material decreased as a function of the annealing temperature. Nevertheless, it did not vary uniformly respecting to the duration of the isothermal annealing.

It was concluded that the microstructural changes occurred in the material during the heat treatment reflected in the mechanical properties. And, the mechanisms of hardening and restoration of this material are directly influenced by the cementite morphologies.

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