Recrystallization Annealing Soaking Time Effect on the Tribological Behavior of Cold Upsetted Low Carbon Steel

Cássio Aurélio Suski^a* 💿, César Edil da Costa^b, Júlio César Giubilei Milan^b

^aInstituto Federal de Santa Catarina, Departamento de Mecânica, Ver. Abrahão João Francisco, 3899, Ave, Itajaí, SC, Brasil.

^bUniversidade do Estado de Santa Catarina, Departamento de Engenharia Mecânica, Paulo Malschitzki, St, 200, Joinville, SC, Brasil.

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This article evaluates the effect of recrystallization annealing soaking time on the tribological behavior of cold upsetted low carbon steel. The 40% cold formed steel was subjected to annealing at 900 °C, for 10, 20, 30, 40, 50 and 60 minutes. The mechanical properties of hardness, tensile strength, yield strength and ductility, measured through specific deformation, as well as the precipitation of carbides through microstructural analysis were examined. In order to analyze the volume of removed material, the wear test was performed using a pin-on-disk tribometer according to ASTM G99. The results showed that the combination of lamellar perlite precipitation and pearlitic interlayer spacing reduction increases the mechanical strength of 1020 steel, reduces ductility, increases friction coefficient and increases wear observed by the volume of removed material increase.

Keywords: low carbon steel; wear; annealing; microstructure; mechanical properties; precipitation.

1. Introduction

Annealing heat treatment of cold formed low carbon steel is used in order to eliminate defects and change the mechanical properties of the material. These properties include hardness, tensile strength, yield strength and ductility. In this treatment, the cold-formed material is heated to temperatures above the total recrystallization temperature, where carbon enters in a solid solution in the austenite phase (total austenitization of the grains), then the material is cooled in the oven promoting the full annealing.

The annealing of cold-formed steels has been the object of investigations¹⁻⁸. These studies have been concerned with the resulting mechanical properties, recrystallization kinetics, microstructure and texture development, individual and combined effects of composition, thermal history before cold forming, heating rates and annealing soaking time.

However, there are few works that analyze the wear resistance of low carbon steels and its relationship with the parameters of the heat treatment process. Tribological studies are generally carried out for high wear resistant steels with hard layers, and low carbon steels are less explored in these studies.

On the other hand, there are several researches about the effects of microstructural parameters on mechanical properties of tensile and wear resistance of steels. It is commonly accepted that the yield and strength limits will increase, and the wear rate will decrease with decreasing prior austenitic grain size, perlite colony size and interlamellar spacing. However, some studies also show that samples with high ductility and similar hardness present a lower wear rate than samples with low ductility^{1,9,10}. There is, therefore, a gap to be investigated regarding the relationship between ductility and the predominant wear mechanisms, so that microstructures can be designed for better performance. The aim of this work is to evaluate the effect of the annealing soaking time on the tribological behavior, associated with ductility, of cold upsetted low carbon steel.

2. Methods

A commercially available SAE 1020 low carbon drawn steel with 8 mm diameter was selected. Figure 1a shows the material's microstructure as received. The chemical composition analysis (Table 1) of low carbon steel was carried out in an Optical Emission Spectrometer - SPECTROMAXx M - SPECTRO. Six samples of each condition of the carbon steel considering: without forming, with 40% forming and without annealing, with 40% forming and annealed in a muffle oven at 900 °C for 10, 20, 30, 40, 50 and 60 minutes were investigated.

Cold forming was carried out in a 45-ton hydraulic press in order to obtain 40% reduction. Figure 1b shows the microstructure of the 40% cold upsetted material.

A Muffle oven, JUNG Model - LF2313, was used for the annealing process. All samples were cooled in the oven to room temperature, with a cooling rate of 0.006 °C/s and then removed for testing. Hardness tests, tensile tests, and wear tests were performed at six samples of each condition. Optical microscopy, confocal microscopy and scanning electron microscopy were used in order to evaluate the samples after the tests.

Hardness was measured through a Digimess HRC. Tensile tests were performed at room temperature in a

^{*}e-mail: cassio.suski@ifsc.edu.br

(a) (b)



Table 1. Chemical composition of SAE 1020 low carbon steel (wt%).

С %	Mn%	Si %	Р%	S %	Al %	Cr %	Cu %	Ni %	Mo %	Co %	В %	Ti %	Sn %	V %
0.2	0.49	0.09	0.021	0.016	0.009	0.03	0.01	0.005	0.005	0.05	0.0016	0.003	0.006	0.005

10 kN Quanteq universal testing machine. The specimens produced according to ASTM E8M standard. The mechanical properties of tensile strength, yield strength and ductility, measured through specific strain were analyzed and a statistical analysis (ANOVA, α =0.05) was performed on the mechanical properties results in order to verify the reliability.

The wear test was performed without lubrication in a pin-on-disk tribometer (Figure 2a), model CZ1000 according to ASTM G99, considering that the alumina ball/pin did not suffer material removal. Four trials were performed for each study condition. Table 2 shows the initial parameters used during the wear tests on the 9 x 30 mm flat-faced samples, sanded with sandpaper up to 600 mesh (Figure 2b). The test environment was kept at a temperature ranging between 22 and 24 °C and relative air humidity ranging between 60 and 66% to minimize the influence of these variables on the tests results.

Optical microscopy was performed in an Olympus BX53MHFU microscope in order to observe the microstructure before and after deformation (Figure 1) and to evaluate the ASTM grain size. The Leika DCM 3D confocal microscope was used to obtain the surface topography for later calculation of the volume of removed material using the Mountains Map software. Scanning electron microscopy (SEM) was performed with the Field Emission JEOL equipment, model JSM-6701F, with the aim to verify the amount of precipitates and the pearlitic interlamellar spacing, as well as to identify the predominant wear mechanisms.

ASTM grain size was determined by the linear intercept method (ASTM 112) performed at six samples of each condition.

The calculation of the volume of removed material (VRM) was performed in Mountains Map software, by multiplying

Table 2. Wear test parameters.

Alumina ball diameter	6 mm				
Speed	0.1 m/s (6 m/min)				
Sliding distance	200 m				
Track radius	2.5 mm				
Load	5 N				

the test perimeter with the cross-sectional area of the wear track obtained through confocal microscopy. Through the software, it is possible to obtain the average profile of the topographic layer series and, consequently, the wear area and the VRM.

The quantification of precipitates was performed using scanning micrographs in Digimet Plus 5G software and the pearlitic interlamellar spacing was determined by the method of random intersection in three different regions for each test condition. A line was drawn on the pearlite with smaller interlayer spacing, dividing the total size of the line by the number of intersections with the cementite lamellae and obtaining the average interlayer spacing of the pearlite.

The predominant wear modes were observed and analyzed using the wear tracks micrographs obtained by scanning electron microscopy.

3. Results

Figure 3 shows the variation in tensile strength, yield strength, hardness and ductility of 40% cold-formed steel annealed at 900 °C for the different soaking times studied. A drop in tensile strength, yield strength and steel hardness is noticed, as well as an increase in ductility between the soaking times of 30 and 40 minutes. Considering the soaking



(a)

(b)

Figure 2. Pin-on-disk tribometer CZ1000 (a). Flat-faced sample measuring 9 x 30 mm (b).



Figure 3. Tensile strength and ductility (a), yield strength and ductility (b) and tensile strength, yield strength and hardness (c) of 40% cold-formed steel, annealed at 900 °C and with soaking times from 10 to 60 minutes.

times of 50 and 60 minutes, there is an increase in tensile strength, yield strength and steel hardness and a drop in ductility compared to the soaking time of 40 minutes. Although the difference in ductility is small, it can be seen from the statistical analysis that there is a difference and that this influences the results and properties. Table 3 and Figure 4 show the average friction coefficient for the various annealing soaking times and Figure 5 shows the friction coefficient according to the sliding distance in the pin-on-disk type tribometer test. A higher friction coefficient average can be observed for the 50 and 60 minutes soaking times.

In addition, the largest volumes of removed material from the disks are observed for the soaking times of 50 and 60 minutes (Table 4 and Figure 6). The largest wear area of the track calculated by the Mountains Map software corresponds to $10420 \ \mu m^2$ for the sample with soaking time of 60 minutes and the volume of removed material corresponds to $0.1716 \ mm^3$.

Figure 7a shows the wear track observed by confocal microscopy and Figure 7b shows a profile obtained from the wear track of the sample with 60 minutes soaking time, which resulted in the highest wear observed in the study.



Figure 4. Average friction coefficient according to the annealing soaking times.

The micrographic analysis of the samples results in the observation of higher cementite spheroidization and smaller presence of lamellar pearlite with increasing soaking times up to 40 minutes and in a smaller spheroidization of cementite and higher presence of lamellar pearlite in soaking times from 50 minutes and on (Figure 8).

Figure 9 shows the precipitation of lamellar cementite for the soaking times of 50 minutes (a) and 60 minutes (b). Table 5 shows the average pearlitic interlamellar spacing for soaking times from 40 to 60 minutes and Table 6 shows the ASTM initial grain size increase with increasing soaking time between 50 and 60 minutes.

Figure 10a shows the smallest wear track width formed on the surface of the without heat treatment sample, due to its strain hardening. In addition, it can be seen through the wear tracks' images that there are two levels of abrasive wear.

The first level is represented by Figure 10b where the wear track width is smaller and the scratch depth is greater for the soaking times of 10 to 40 minutes. The second level is represented by higher wear tracks width, with greater quantity and smaller depth of scratches for the soaking times of 50 and 60 minutes (Figures 10c and d), resulting in a higher volume of material removed, as observed in Figure 6.

Compared with the 40 minutes sample (Figure 10b), the deformed material (arrows) that was removed from the track and is stuck in the interface of pin and disk of the 50 and 60 minutes samples are observed to be detached more frequently and easily from the surface of the disk (Figures 10c and d).

4. Discussion

Table 7 presents the results of the parameters analyzed in this study. Based on the results found, an increase in disk



Figure 5. Friction coefficient by sliding distance.

Table 3. Average of friction coefficient according annealing soaking times.

Soaking time (min)	WHT*	10	20	30	40	50	60
Friction coefficient average	0.22	0.21	0.25	0.25	0.24	0.29	0.27
Standard deviation	0.03	0.01	0.02	0.02	0.02	0.02	0.02

^{*}WHT - Without Heat Treatment

Table 4. Volume of removed material from the disks for the different soaking times.

Soaking time (min)	10	20	30	40	50	60
Disk volume loss (mm ³)	0.0560	0.0403	0.0581	0.0429	0.1690	0.1716
Standard deviation (mm ³)	0.0166	0.0041	0.0262	0.0083	0.0358	0.0246



Figure 6. Disk volume loss.

volume loss can be observed from a 50 minute soaking time and this increase can be explained by two sets of factors.

On the one hand, the reduction of the average pearlitic interlamellar spacing and the increase of the ASTM grain

Table 5. Average pearlitic interlamellar spacing.

Soaking time	Average pearlitic interlamellar spacing (µm)				
40 min.	0.60				
50 min.	0.52				
60 min.	0.34				



Figure 7. Confocal wear track micrography (a) wear track profile of 60 minutes soaking time sample.



Figure 8. SEM micrograph of (a) and (b) soaking time of 40 minutes and (c) and (d) soaking time of 60 minutes.

Table 6. ASTM grain size.

Soaking time	ASTM Grain Size Number				
10 min.	6.33				
20 min.	6.25				
30 min.	6.33				
40 min.	6.29				
50 min.	6.37				
60 min.	6.42				

size number with the increase of the soaking time generate an increase in hardness, yield strength and tensile strength, which, consequently, reduced the disk volume loss.

Several authors¹¹⁻¹⁵ carried out studies confirming the effect of microstructural parameters on mechanical properties of tensile and wear resistance of steels, where the yield strength increases and the wear rate decreases with the reduction of the size of the prior austenitic grain, of the size of perlite colonies and interlamellar spacing.



Figure 9. Quantitative analysis of lamellar cementite precipitation for the soaking times of 50 minutes (a) and 60 minutes (b).



Figure 10. SEM micrograph of wear tracks for soaking times of (a) Without heat treatment, (b) 40 minutes, (c) 50 minutes, and (d) 60 minutes.

Soaking time (min)	10	20	30	40	50	60
Disk volume loss (mm ³)	0.0560	0.0403	0.0581	0.0429	0.1690	0.1716
Average pearlitic interlamellar spacing (µm)	-	-	-	0.60	0.52	0.34
ASTM Grain Size Number	6.33	6.25	6.33	6.29	6.37	6.42
Hardness (HRC)	52.2	52.1	51.9	50.3	51.0	51.7
Yield Strength (MPa)	240	238	236	205	220	228
Tensile Strength (MPa)	379.2	379	378	366.8	370	375
Ductility (mm/mm)	0.61	0.60	0.60	0.62	0.58	0.57
Friction coefficient average	0.21	0.25	0.25	0.24	0.29	0.27

Table 7. Master table of parameters studied.

Mao et al.¹⁶ showed that, for samples of low carbon steel with smaller grain size, there is greater wear resistance and that samples with larger grain size and lower density of dislocations have lower wear resistance and, as a result, the wear mode changed from abrasive wear to adhesive wear.

The tribological behavior shows a significant reduction in wear resistance (Figure 6) of samples with soaking times of 50 and 60 minutes. This fact differs from the results found in some previous studies by other authors, where steels with greater tensile strength have greater wear resistance. In the mentioned soaking times, the tensile strength and yield limit of the samples are greater than the for the 40-minute soaking time samples (Figure 3). Thus, these properties are not the only factors that enable the generation of different wear behaviors for the samples.

On the other hand, an increase in lamellar cementite with an increase in the soaking time reduces the ductility which, together with an increase of the friction coefficient average, results in a significant increase (about 400%) in the disk volume loss of the steel studied.

For samples with soaking times of 50 and 60 minutes was the precipitation of iron carbides (cementite) on lamellae form, unlike the shorter soaking times that presented spheroidal cementite (Figures 8 and 9), in addition to the pearlitic interlamellar spacing decrease with increasing soaking time (0.52 and 0.34 μ m, respectively, Table 5). This fact can be explained by the higher transformation kinetics in the coarse grain region (Table 6), which would shift the transformation curve in continuous cooling and, therefore, the austenite transformation would occur at a lower temperature. Thus, with longer soaking times (50 and 60 minutes) there is higher transformation kinetics and, consequently, occurs the formation of fine pearlite, which results in an increase in the yield limit and tensile strength of the steel, i.e., precipitation hardening occurs.

According to Kuziak et al.¹⁷ hardness increases as the interlayer spacing decreases, or as the fraction of cementite within the pearlite increases. It was observed by the authors that the interlamellar spacing reduced with increasing cooling rate from 0.25 to 5 °C/s, but between 5 and 10 °C/s the interlamellar spacing increased. The fine pearlite has a smaller interlayer spacing, added to the fact that it has a larger grain boundary area, which results, on the one hand, in a higher resistance compared to the coarse pearlite, however, on the other hand, results in less ductility.

Other authors^{1,9,10,18} show that, for pin-on-disk wear tests in carbon steels, the sample with high ductility presents a lower wear rate than the sample with low ductility.

Thus, on the one hand, for the soaking times from 50 minutes, it can be seen the reduction of spheroidized cementite, the addition of lamellar perlite and the reduction of the pearlitic interlamellar spacing, which generates higher hardness and mechanical strength.

On the other hand, for steels with a ferritic matrix and hard regions (cementite) that have low ductility, it can be expected that they also have low wear resistance¹⁹.

In this study, an increase in lamellar cementite after annealing was observed at soaking times above 50 minutes, which helps to accumulate dislocations, increase the storage capacity of dislocations and the slip resistance, which results in samples hardening and ductility reduction. One of the factors that may have contributed to the lower wear resistance or a higher amount of removed material from samples with longer soaking times is their lower ductility (Figure 3), together with the presence of carbides.

In addition, the friction coefficient (Figure 4) increases for samples with longer soaking times, which increases the wear amount, consequently, observed by the volume of removed material. Andrade et al.²⁰ show in their study that, in a moderate wear regime, a lower friction coefficient was observed than in a severe regime. The authors obtained a coefficient of friction of 0.25 for a wear test on 1020 steel with a load of 5N. The article also shows a wear rate of $2.46 \times 10^{-4} \text{ mm}^3/\text{mm}$, which is equivalent to 0.0615 mm³ for a 250 m wear test, ie similar to the values found in this study.

Micrographs (Figure 10) confirms that the subsurface strain accumulates and plate-like wear debris begin to appear (Figures 10c and d), some of which are shown to be partly detached from the surface. The plate-like wear debris of the 40 minutes sample seem to be more ductile than that observed on the surface of the 50 and 60 minutes samples. Larger plate-like wear debris formation was observed in the sample that showed higher wear rates and a higher amount of wear debris remaining on the track were observed in the 50 and 60 minutes samples compared to the 40 minutes sample track surface. The micrographs indicate that the plate-like wear debris formation and its detachment, together with flake-like debris formation, predominate in the steel under the given test conditions. During dry sliding wear, surface deformation layers are formed by strain accumulation through repeated contact of the counterfaces and wear occurs when the layers are detached from the surface. The detachment

proceeds when cracks are initiated along regions where the accumulated strain exceeds its failure or critical strain, forming flake-like wear debris. The wear tests results and the micrographs reveal that the samples with higher ductility form the delaminating surface platelets slowly and show lower wear rate, indicating that mechanical properties would not be the only wear control parameters under the given test conditions.

5. Conclusion

The tribological behavior of 1020 steel with 40% cold forming, annealed at 900°C with soaking times from 10 to 60 minutes were investigated by means of a pin-on-disk wear test, in addition to the mechanical properties and microstructure observations. The main conclusions are as follows:

- The volume of removed material from samples with soaking time up to 40 minutes was lower than the volume observed for samples with soaking times higher than 50 minutes, although the fraction of lamellar pearlite of samples with soaking times higher than 50 minutes was higher than the observed for samples with a shorter soaking time. In samples with longer soaking times, the lamellar pearlites presented higher hardness than the observed in 40-minute samples;
- The combination of lamellar pearlite precipitation and pearlitic interlayer spacing reduction increases the mechanical strength of 1020 steel, reduces ductility, increases friction coefficient and generates greater wear observed by the increase in the amount of volume of removed material;
- The steel presented hard regions (cementite), but in a ferritic matrix, which have low ductility and low wear resistance, thus one of the factors that may have contributed to the lower wear resistance or higher volume of removed material from samples with larger soaking times could be its lower ductility, combined with the presence of carbides.
- Micrographic observations of the wearing surface showed that the formation and detachment of the wear sheets were slower in the sample with higher ductility. The delayed formation directly resulted in the lowest wear rate and ductility appears to be an important material parameter in determining the wear rate under the given test conditions.

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