10º Congresso Brasileiro de Engenharia de Fabricação
05 a 07 de agosto de 2019, São Carlos, SP, Brasil

MICROSTRUCTURAL STUDY AND HOLLOMON-JAFFE CONSTANT BEHAVIOR OF QUENCHED AND TEMPERED SAE 4140 STEEL

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Abstract. *This article aims to investigate the microstructure and hardness behavior of SAE 4140 steel after tempering processes at times ranging from 15 minutes to 24 hours and temperatures between 200 and 700ºC. It also intends to investigate changes on the average grain size after austenitizing at different temperatures and times during the quenching process. Microstructural analysis was performed using an optical microscopy and Electron Backscatter Diffraction, X-ray diffraction technique was used to identify the phases fractions on the material. Hot rolled SAE 4140 steel showed a ferritic-pearlitic microstructure containing rod-shaped iron and chromium carbide. After water quenching, an austenitic grain boundary was obtained in a matrix of martensite, as expected. The average grain size remained practically constant after quenching at temperatures between 900 and 1000 ° C in times of austenitizing ranging from 1 to 4 hours. At temperatures above 1050°C there was a significant increase in the average grain size. The mathematical modeling for hardness prediction after the tempering process was based on the Hollomon-Jaffe parameter. The Hollomon-Jaffe constant presented a mean value of 14.36, but at high temperatures it was observed a variation in the C-value due to spheroidization and coalescence of iron-carbide.*

Keywords*: Quenching. Tempering. Hollomon-Jaffe. Mathematical modeling.*

1. INTRODUCTION

SAE 4140 is a low alloy steel widely used under quenched and tempered conditions in the production of shafts, gears and molds for the automotive, aeronautic and oil industries due to the properties it can achieve after surface treatment. (Rios *et al.*, 2016; WU *et al.*, 2016). After quenching and tempering, SAE 4140 steel has austenitizing temperature range sufficiently high to homogenize the austenite, but low enough to avoid coarse-grained microstructure which may promote intergranular cracks and retained austenite increase (Tavares *et al.*, 2017).

The main purpose of the tempering treatment is to improve the insufficient toughness of the martensitic steels, but a decrease in the mechanical strength occurs during the process. The properties obtained after the tempering process are related to the temperature and time of the treatment and also to the presence and the amount of alloying elements that the steel contains. The choice of these parameters may result in temper embrittlement, residual stress and microstructural heterogeneity. Microstructural heterogeneity is caused by the complex evolution of the microstructure during tempering such as transition metals carbides precipitation, decomposition of retained austenite into ferrite and cementite, precipitation and spheroidization of cementite and recovery and recrystallization of martensite (Saglitz *et al.*, 2008).

With the purpose of correlating tempering time and temperature with hardness for quenched steels, Wan, *et al*, 2005 developed a mathematical equation (2) based on the tempering parameter proposed by Hollomon-Jaffe. The tempering parameter (TP) is dependent on the temperature, time and the steel used, as shown below:

$$TP = T\,(\log \tau + C)\; x\; 10^{-3} \tag{1}$$

Using a single parameter to estimate tempering time and temperature for all alloy steels can lead to serious errors (Canale *et al.*, 2008). The C=20 value for the SAE 4140 steel was not constant according to other authors, and also, the model did not fit the tempering of tool steels. According to Thomas, *et al*, 2008 the fact that the C-value might not be constant as proposed by Hollomon-Jaffe is a concern regarding the use and precision of the tempering parameter model. This paper aims to mathematically model the tempering heat treatment for SAE 4140 steel at temperatures ranging from 200 to 700ºC and holding times varying from 15 minutes to 24 hours, and, it also aims to estimate the C-value and to correlate it with the microstructural evolution along the process.

2. METHODS AND MATERIALS

SAE 4140 steel bars were acquired with 5/8" and 3" in diameter and length of 1000 mm. Chemical analysis of the bars was performed using x ray fluorescence technique. In order to know the phases in the austenite region, a simulation was run using the Thermo-Calc software. The samples were austenitized in a muffle furnace with chamber dimensions of 30x15x15cm at 900, 950, 1000 e 1050ºC for 1, 2, 3 and 4 hours respectively, and after that, they were quenched in water at ambient temperature. The furnace temperature control was made by digital controllers containing electronic relay with accuracy of ± 0,1ºC. The tempering treatment was also performed using a muffle furnace in the samples that were previously austenitized at 900ºC. These samples were heated at 200, 250, 300, 350, 400, 450, 500, 550, 600, 650 and 700ºC for times ranging from 15 minutes to 24 hours. Hardness tests for SAE 4140 steel were carried out on a Rockwell Hardness Testing Machine using the Rockwell C scale. The samples were sanded, polished and etched with a 2% nitric acid solution or picric acid for measurement of austenitic average grain size according to Hilliard method. Afterwards, the specimens were analyzed by Optical and Electron Microscopy using a ZEISS brand optical microscope and a JEOL brand scanning electron microscope equipped with EDS. A Shimadzu brand X-Ray diffractometer was used to identify the phases of SAE 4140 under hot rolled, quenched and tempered conditions.

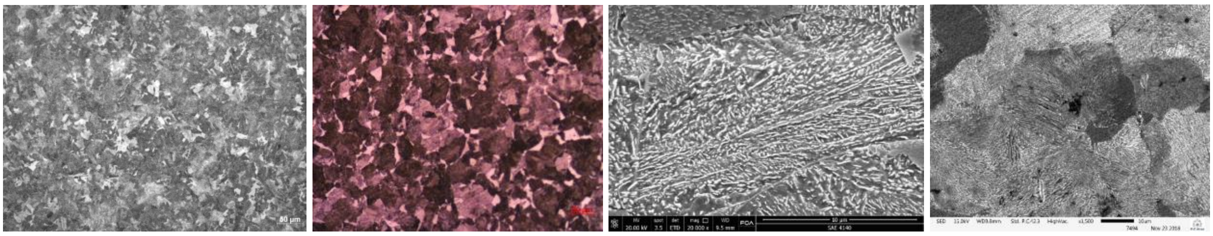
3. RESULTS AND DISCUSSION

Table 1 shows the chemical composition in percent by weight and the hardness for the SAE 4140 steel under hot rolled condition with 5/8" and 3" in diameter.

Table 1 – Chemical composition of SAE 4140 Steel

%C	**%Mn**	**%Si**	**%P**	**%S**	**%Al**	**%N**	**%Cr**	**%Ni**
0,405	0,92	0,236	0,009	0,0014	0,0276	0,0043	0,999	0,035
%Mo	**%Ti**	**%Nb**	**%V**	**%B**	**%Cu**	**%Sn**	**%Ca**	***Hardness(HRC)***
0,22	0,0047	0,003	0,004	0,0005	0,04	0,003	0,0012	33+-1

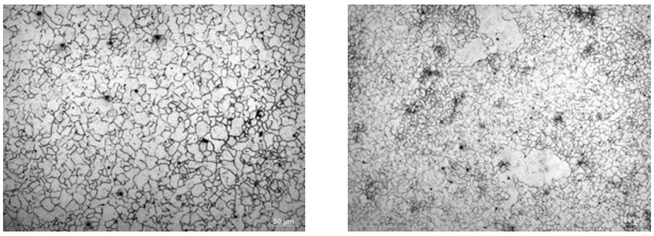
The SAE 4140 steel microstructures at the conditions referenced above respectively showed ferritic microstructure containing rod-shaped iron and chromium carbides and a ferritic-pearlitic microstructure (Fig. 1).



(a) (b) (c) (d)

Figure 1: Hot Rolled SAE 4140 Microstructure (a)ferrite and carbides – 5/8" (b) ferrite and pearlite – 3"(c) ferrite and carbides - 5/8" (d) pearlite - 3"

After etched with picric acid solution, the microstructure of SAE4140 steel quenched in water showed austenitic grain boundaries in a martensitic matrix (Fig. 2)

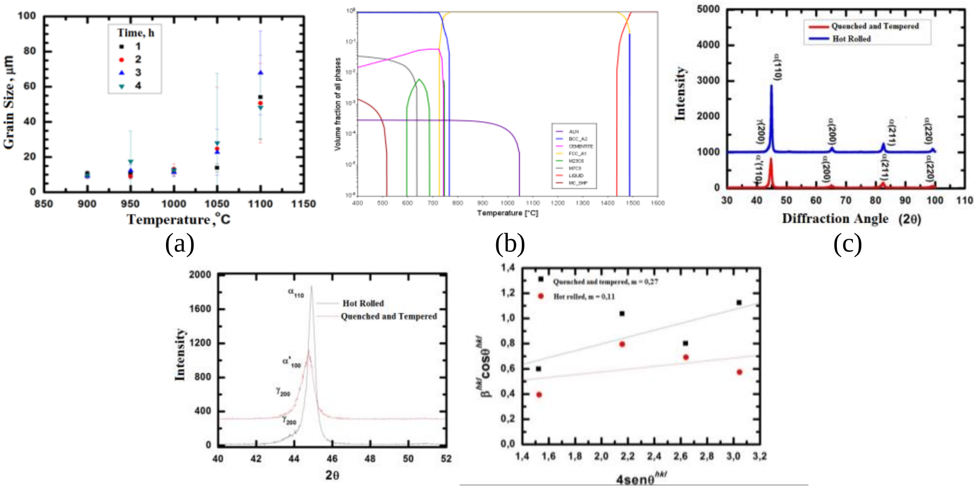


(a) (b)

Figure 2: Microstructures of SAE4140 steel after quenching in water showing uniform granulation (a) 1h and heterogeneous or mixed (b) 1100°C - 4h

The evolution of the average grain size in relation to austenitizing temperature, and the precipitation simulation run using Thermocalc software are shown in Fig. 3(a) and (b). Figure 3(a) shows that the austenite average grain size remains practically constant at austenitizing temperature of 1000 °C with holding times of 1, 2, 3 and 4 hours. The growth started at 1050ºC in times greater than 2 hours. It is observed that the dissolution of aluminum nitride (AlN) precipitates in the austenitic microstructure occurred in temperature range of 1000-1050°C which shows that the AIN precipitates might anchor the grain boundaries. The hardness of the SAE 4140 steel after austenitizing at 900°C and water quenching was 56.4 ± 3.4 HRC. Figure 3(c) shows the X-ray diffraction pattern of hot rolled and quenched SAE 4140 steel with 5/8” in diameter after tempering treatment at 580ºC for 2 hours. Hot rolled/Quenched and Tempered SAE 4140 steel had predominantly ferritic microstructures. Also, reflections of lower intensity corresponding to the austenitic phase were detected in both conditions, showing that this phase is present in smaller amounts. This is made clear by the peak deconvolution (Fig 3(c)). Figure 3(d) shows the region corresponding to the most intense ferrite and austenite phases reflections. By expanding the image, it was noticed that the SAE 4140 steel sample that was water quenched and tempered at 580ºC for 2 hours had a broadening of the diffraction peak. It was also noticed through spectral deconvolution using *Voigt* peak functions the presence of martensitic reflections in the same sample. (*e.g.* (HERNANDEZ-RIVERA, CAMPOS CAMBRANIS, RAMOS-MOORE, 2011)). Broadening of diffraction peaks is usually attributed to two main reasons, which are the reduction of the crystallite size (portion inside the grains from which coherent diffraction of the incident radiation is obtained) and heterogeneities in the crystalline network caused by defect densities increase. (RAMOS-MOORE et al., 2014). The difference in the peak broadening showed in Fig. 3(c) was also persistent for the other ferrite phase reflections, as observed by analyzing the parameters obtained using the profile of the diffraction peaks. (Full width at half maximum, FWHM, and integral breadth, β)

Williamson-Hall plots (Fig. 3(e)) were generated to compare the level of micro deformations in the samples, which are related to crystalline defects density (UNGAR, BORBELLY, 1996). Conventional Williamson-Hall diagrams consider only the contributions to the peak broadening related to the crystallite size, which is a function of the intercept of the linear regression represented by the vertical axis and the micro deformation which is a function of the angular coefficient (“m”). The analysis performed in this paper is only semi-quantitative, since the contribution of the measuring instrument (XRD) to the peak broadening was not considered. Both samples presented positive angular coefficients in the Williamson-Hall diagrams which would be indicative of traction micro deformation, with a higher level of defect density observed in the quenched and tempered SAE 4140 sample (m = 0.27 compared to m = 0.11 for the hot rolled SAE 4140 sample). This may be due to the presence of a remaining portion of martensite from the water quenching performed before tempering



(a) (b) (c)

(d) (e)

Figure 3: SAE 4140 – (a) Average grain size x austenitizing temperature and time(b) Precipitation simulation (c) X-ray diffraction of hot rolled and quenched SAE 4140 steel tempered at 580ºC for 2 hours (d) Peak deconvolution of Hot rolled/Quenched and tempered SAE 4140 steel (e) Williamson-Hall plots for SAE4140TR and SAE4140HR.

The microstructure of quenched SAE 4140 steel tempered at 700ºC for 4 and 24 hours showed the presence of fine carbides (a) and spheroidal carbides after 24 hours (b) (Fig 4)

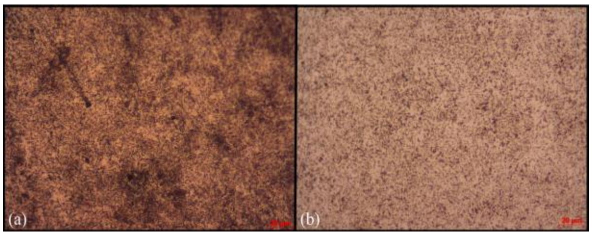


Figure 4: SAE 4140 tempered at 700ºC 500x a) 4h b) 24h

3.1 Mathematical Modeling for Tempering

The equations developed by Wan, *et al*, 2005 based on the Hollomon-Jaffe parameter (TP) are shown below:

$$TP = T\,(\log\tau + C)\ x\ 10^{-3} \tag{1}$$

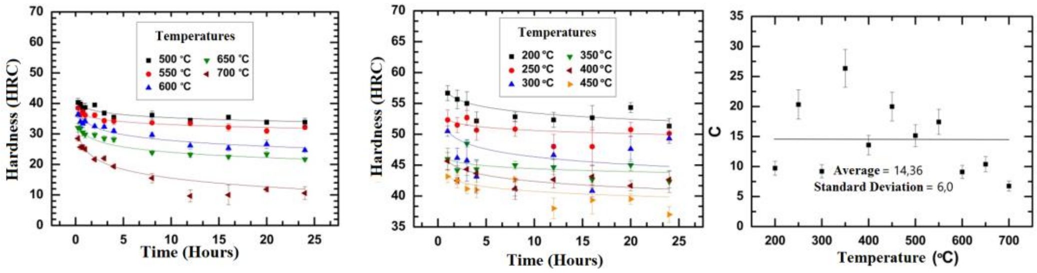
$$H = a + b\log\tau \tag{2}$$

$$a = H_1 \tag{3}$$

$$b = \frac{T}{C}\left(\frac{dH_1}{dT}\right) \tag{4}$$

Where H is the steel hardness after tempering treatment, $H1$ is the tempering hardness after 1 hour, T is the tempering temperature in K, τ is the tempering time in hours for an isothermal condition and C is the Hollomon-Jaffe constant which is related to the steel.

Tempering hardness x tempering time curves were plotted considering the best-fit value for the b parameter for quenched and tempered SAE 4140 steel (Fig. 5(a) and (b)). After finding the best-fit for b at different temperatures, a linear regression was executed in order to find $\left(\frac{dH}{dT}\right)$ values. Subsequently, Hollomon-Jaffe constant values (C) were calculated (Fig. 5(c))



(a) (b) (c)

Figure 5: SAE 4140 Mathematical Modeling (a) Tempering Hardness x Tempering Time - 500 to 700ºC (b) Tempering Hardness x Tempering Time - 200 to 450ºC (c) Evolution of C-value in relation to tempering temperature

The mean value of the C parameter was recalculated considering two different tempering temperature ranges. (200 to 550ºC; C = 16,5 and 600 to 700ºC; C = 8,75). Using the obtained values for C and $\left(\frac{dH}{dT}\right)$ it was possible to calculate the estimated tempering hardness for SAE 4140 steel at tempering temperatures ranging from 200 to 700ºC. Table 2 displays the standard errors between the calculated and measured values.

Table 2 – Standard errors for each tempering temperature for the studied models

SAE 4140, C = 20; 1st. hour $\left(\frac{dH}{dT}\right)$				**SAE 4140, Changeable C and $\left(\frac{dH}{dT}\right)$**			
Temp. (ºC)	**Error (%)**	**Temp. (ºC)**	**Error (%)**	**Temp. (ºC)**	**Error (%)**	**Temp. (ºC)**	**Error (%)**
200	3,57 ± 2,65	500	2,66 ± 2,04	200	2,83 ± 2,17	500	1,42 ± 1,47
250	1,79 ± 2,13	550	2,02 ± 1,68	250	1,92 ± 1,37	550	1,66 ± 1,24
300	8,60 ± 7,31	600	8,95 ± 8,99	300	8,14 ± 6,64	600	3,27 ± 2,82
350	2,49 ± 1,95	650	7,65 ± 2,04	350	2,72 ± 2,65	650	5,96 ± 5,36
400	2,41 ± 2,45	700	42,70 ± 48,80	400	2,07 ± 2,18	700	17,49 ± 22,07
450	3,30 ± 3,71	-	-	450	2,11 ± 2,37	-	-

The mathematical modeling of the tempering process using equations based on the Holommon-Jaffe parameter showed low errors using C=20 at temperatures below 600ºC, but between 600 and 700ºC it presented standard errors that reached up to 42%. Spliting the model into two range of temperatures, it was found a mean value for the C parameter of 16.5 for temperatures up to 600ºC and 8.75 for temperatures above 600ºC. This variation in the C-value may be linked to microstructural changes that can occur at higher temperature ranges, such as coalescence and spheroidization of cementite particles. The corrected model had a maximum standard error of 17.49% at 700ºC, which is much lower than the 42% found when considering C=20 as a constant value for all temperatures.

4. CONCLUSION

In both hot rolled and quenched/tempered conditions, SAE 4140 steel presented ferrite as predominant phase with presence of austenite in small amounts. The quenched microstructure presented austenitic grain boundaries inside a martensitic matrix.

The average grain size for quenched SAE 4140 steel was approximately 10μm and remained practically the same when the austenitizing treatment occurred between 900 and 1000°C at times ranging from 1 to 4 hours. At temperatures above 1050ºC, a significant increase was observed in the average grain size, reaching up to 60μm at austenitizing temperatures of 1100ºC. This might be caused due to the dissolution of aluminum nitride that occurs at temperatures higher than 1050ºC.

It was noticed that the C-value behavior for the mathematical model proposed by Wan, *et al*, 2005 diverges from what was presented by Thomas, *et al*, 2008 by analyzing the original tempering parameter proposed by Hollomon-Jaffe.

The authors also concluded that any tempering parameter/model based on the Hollomon-Jaffe equations that considers C-value as constant should be used with extreme caution, since several researchers have reported a non-constant behavior for this parameter, and as seen in this paper, it can lead to critical errors in some specific situations.

5. REFERENCES

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6. RESPONSIBILITY FOR INFORMATION

The authors are solely responsible for the information included in this work.

7. ACKNOWLEDGEMENTS

J.R.G. Carneiro and the authors would like to thank the financial support of grant n° 2016/308964-0, Brazil Conselho Nacional de Pesquisa CNPq.