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Study of Short Times Tempering for AISI D2 Cold Work Tool Steel

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Short time tempering has been applied to steels to improve toughness keeping the hardness at the same level. For AISI D2 tool steel, typical thermal cycles are quench and temper. The austenitizing temperature is around 1040 ºC and the subsequent double tempering at temperatures close to 500 ºC, for 2 hours each, in order to get the maximum secondary hardness. The aim of the present work is to verify the effects of short time tempering in the AISI D2. For such purpose the Hollomon-Jaffe tempering parameter was used to determine the equivalent time at 600 and 700 °C, being 500 °C for 2 hours the standard temperature and time. The thermal cycles were carried out at a dilatometer. All the specimens were austenitized at 1040 °C followed by double tempering at: 500 and 700 °C for 2 hours each; and 500 °C, 600 °C, and 700 °C for 10 s and 1 min each. The dilatometric curves were analysed. Scanning electron microscopy, X-ray diffraction and Vickers hardness were carried out to characterize the microstructure and mechanical properties. The short time tempering conducted at high temperatures led to the formation and growth of tempering carbides and to matrix recrystallization, which were responsible for greatly reducing the hardness of the material. The short time tempering up to the intermediate temperature of 600 ºC, for 1 minute or 10 seconds, produced results like the conventional tempering in microstructural and hardness terms, denoting that the Hollomon-Jaffe tempering parameter predictability close to the secondary hardness peak.

Keywords: *AISI D2 Cold Work Tool Steel, Hollomon-Jaffe Parameter, Short Time Tempering.*

1. Introduction

Short-term tempering of martensite has been suggested as a viable approach to achieve an optimal balance between corrosion resistance and obtaining the desired mechanical properties of martensitic carbon steels[1]. Validation of computer simulation results revealed the potential for optimizing short tempering heat treatment processes for steel as a reliable approach to save time, resources and energy[2]. Short time tempering, at high temperature also claims to decrease the temper embrittlement, resulting in mechanical properties not obtained by conventional tempering[3].

Rapid induction tempering of high speed steel produced mechanical properties which are approximately equal to those resulting from conventional long-time tempering treatments. At maximum secondary hardness, high speed steel which is induction tempered by rapid heating exhibits a slightly greater proportion of retained austenite[4]. In secondary hardening steels the precipitation of dispersed carbides taking place at the elevated tempering temperatures caused martensite ageing[5].

Tempering in short times can also be understood as an overtempering and is justified by using treatment attributes such as tempering and partitioning or intercritical treatments, where higher treatment temperatures are used and, in turn, reduced treatment times, with the intention of acting on austenite levels, either through its stabilization and retention or even through a possible reversion from martensite, as occurs with martensitic and supermartensitic stainless steels, combined with other phenomena such as carbide precipitation and recovery of martensite, responsible for better properties[1,4,6,7,8,9].

Temperature and time in quenching and tempering treatments are the variables with the greatest influence on the microstructure and properties of steels. Some parameters are used in an attempt to estimate different tempering time-temperature relationships that produce similar effects, with emphasis on the Hollomon-Jaffe parameter[10].

The dimensions and distribution of the carbides are the most important factors in terms of impact toughness. When the material was over-tempered, either for a long period of time or by substantially raising the treatment temperature, the resulting broadening of bainitic ferrite and precipitation of grain boundary carbides led to embrittlement of the material by tempering[11].

In a study on the tempering and partitioning, used process responsible for increase retained austenite, usually at slightly higher temperatures was observed that five events take place during heating, probably initially involving the segregation and grouping of carbon, followed by precipitation of ε/η transition carbides, decomposition of retained austenite and formation of cementite and finally recrystallization[6,7]. One or another

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stage of tempering can overlap, or even some step may be omitted due to the characteristics of each material[12]. In highly alloyed steels such as AISI D2, cementite is replaced by the precipitation of alloy carbides and the secondary hardening can ocours[13,14]. An increase in tempering temperature leads to a change in the composition of precipitated carbides and to the corresponding microhardness[15].

The embrittlement associated with secondary hardening is caused by non-coherent carbide particles. Being the resistance increase, obtained in tempering in the secondary hardening range, produced by an increase in tensions (when the carbides of the alloy are coherent) and hardening (when the carbides are non-coherent)[13].

Short time tempering (STT) thermal treatments were carried out in order to preliminarily study the indicators of microstructural changes obtained in comparison to conventional cycles. The objective of this work was to investigate the feasibility of carrying out a thermal cycle with tempering treatment in short times.

2. Procedures – Experimental Techniques

This study was carried out with the high carbon, high chromium tool steel, AISI D2 (1.5%C and 12%Cr). The steel, produced by Villares Metals S.A., was supplied in bars with diameter of 25 mm, in the annealed condition. For the dilatometric tests, specimens with 4 mm in diameter and 10 mm in height were manufactured from the half radius of the bars, being extracted by wire EDM machining.

In order to simulate the effects of short time tempering, the Hollomon and Jaffe parameter was used to determine the thermal cycles. The Hollomon-Jaffe parameter is determined by the expression $P = T (C + \log t)$, where T is the temperature, t is the time of the tempering heat treatment and C is a material-related constant.

The standard tempering condition was set up as a double tempering at 500 °C, for two hours each. The equivalent P for 600 °C and 700 °C is near 1min and 1 s, respectively. Based on that, the thermal cycles were defined, Table 1.

The thermal cycles were simulated at a Bähr quenching dilatometer DIL805A, available at Phase Transformation Laboratories – LTF – USP. All the specimens were austenitized at 1040 °C, with a heating rate of 10 °C/s, dwell time of 10 min, and quenched to room temperature at a cooling rate of 50 °C/s. Just after quench, the specimens were submitted to the different temper treatments, with a heating rate of 10 °C/s and cooling rate of 50 °C/s, the dwell time is according to Table 1.

Table 1. Specimens identification and respective thermal cycles.

HEAT TREATMENT CYCLES	
DESIGNATION	Conventional Thermal Cycles
QT500	Double Tempering at 500ºC for 2 hours
QT700	Double Tempering at 700ºC for 2 hours
Short Time Tempering Thermal Cycles - 10 s and 1 min.	
QSTT500-10 s	Double Tempering in Short Time at 500ºC for 10 s
QSTT600-10 s	Double Tempering in Short Time at 600ºC for 10 s
QSTT700-10 s	Double Tempering in Short Time at 700ºC for 10 s
QSTT500-1 min.	Double Tempering in Short Time at 500ºC for 1 min.
QSTT600-1 min.	Double Tempering in Short Time at 600ºC for 1 min.
QSTT700-1 min.	Double Tempering in Short Time at 700ºC for 1 min.

The dilatometry curves were analyzed using the first derivative method and the cross-section of the specimens were characterized to confirm the microstructure and hardness.

The diffraction data collections were carried out on the X-ray diffractometer Malvern PanAlytical Empyrean X'Pert, available at the Laboratory of Technological Characterization – LCT – USP. With the diffractometer in reflection-transmission mode, using a copper tube in rotation with a spinner of 3 rps, with a pitch of 0.02°; time of 300 s and voltage of 45 kV. The volumetric fractions of the austenite and martensite phases were carried out with Malvern Panalytical's HighScore Plus XRD analysis software.

The SEM (Scanning Eletron Microscopy) was carried out at the Microscopy Laboratory – LabMicro – USP.

The average size and distribution of carbides observed in each microstructure was statistically estimated, as recommended by the ASTM E975-13 standard, with the support of the image analysis program (ImageJ)[16,17].

To determine hardness, measurements were taken using a Vickers Microdurometer, Shimadzu DHT, model HVS -1000. Load: 300 g or 2.94 N, Time: 10 sec. Values of hardness correspond to the average of 3 determinations.

3. Results and Discussion

AISI D2 tool steel, in the annealed condition, has a ferritic matrix plus eutectic and secondary carbides, Figure 1a. In the as quenched condition, the steel has a martensitic matrix with some retained austenite, large eutectic carbides and dispersed secondary carbides, Figure 1b.

The microstructures of AISI D2 cold work tool steel specimens in the condition quenched and double tempered at (a) 500 °C and (b) 700 °C are shown in Figure 2.

In the heat treatment condition shown in the image in Figure 2a, the specimen has a tempered martensite structure with retained austenite and fine precipitated carbides in the tempering at 500 ºC. Figure 2b referring to double tempering conventionally applied at 700 ºC for 2 hours, presents the exacerbated tempering carbide precipitation and the steel matrix starts to present a condition similar to the annealed ferritic matrix and austenite is not observable due to the transformation into ferrite and carbides in the tempering.

The results of the dilatometry tests provided information on phase transformations that occur in each cycle, as shown in Figure 3.

The images in Figure 3 show the (a) dilatometry graph, highlighting the austenitizing and tempering stage at 1040 ºC. The Ac1, Ac3 and Ms temperatures are indicated. Also represented, the (b) derivative of change in length from the dilatometry graph in the region close to the critical zone during heating for austenitization, as an aid for the identification of temperatures Ac1 and Ac3, with values of 830 ºC and 905 ºC, respectively, and Ms

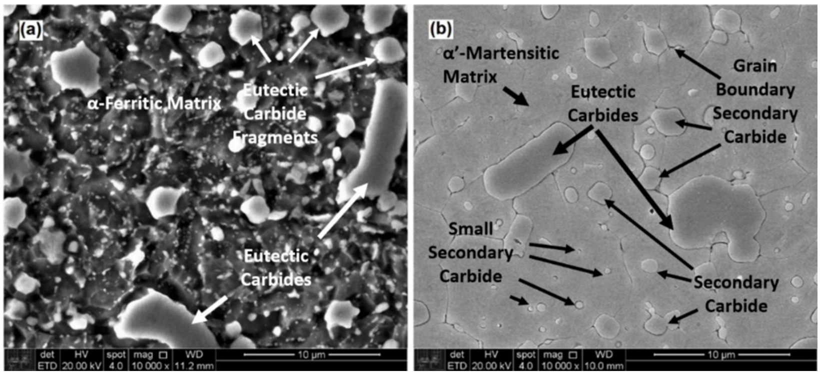

Figure 1. AISI D2 tool steel microstructures: (a) annealed and (b) as quenched. SEM. Nital etched.

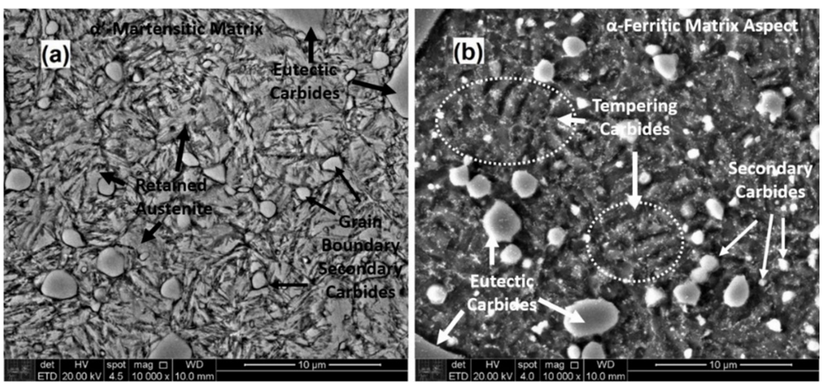

Figure 2. Microstructures of AISI D2 steel: Quenched and double tempered at (a) 500 °C and (b) 700 °C. SEM. Nital etched.

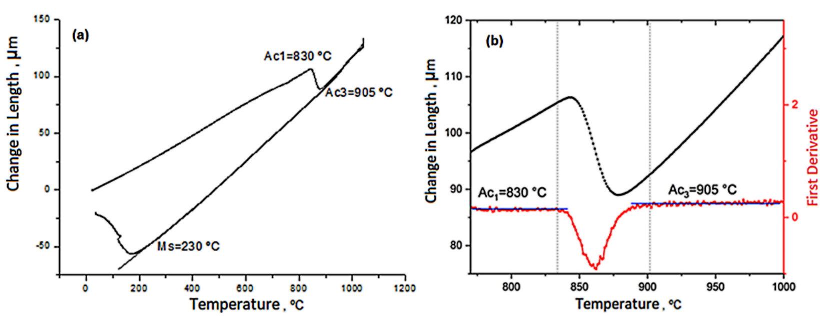

Figure 3. (a) Dilatometric graph indicating the critical transformation temperatures Ac1, Ac3 and Ms after austenitization at 1040 °C. (b) Detail for determination of Ac1 and Ac3 temperatures.

with mean value of 230 °C. The images in Figure 4 are of the microstructures of the AISI D2 tool steel referring to the double tempering cycles in short times at temperatures of 500 ºC(Figure 4a and 4b), 600 ºC (Figure 4c and 4d), and 700 ºC(Figure 4e and 4f), for times of 10 seconds or 1 minute, respectively.

In the detail shown in Figure 5, of the secondary carbide of the specimen treated in the QSTT700-1min cycle, it is noticeable the dissolution of the carbide edges. Such behavior is responsible for the supply of alloying elements, including carbon, to the matrix, favoring the precipitation of tempering carbides. Under the prevailing conditions, these carbides manifest as intricate formations, leading to the depletion of the matrix.

The hardness tests results of the specimens in the as received annealed condition, quenched, and quenched and tempered condition; the temperatures and times are indicated in the label of the chart, in Figure 6. The results of the hardness measurements show a marked reduction in hardness in the tempering applied at highest temperatures (700 °C), regardless of treatment time (10 s, 1 min. or 2 h).

The Vickers hardness results, Figure 6, indicates the maintenance of hardness levels for treatments carried out up to 600 ºC and a sharp drop in hardness for treatments carried

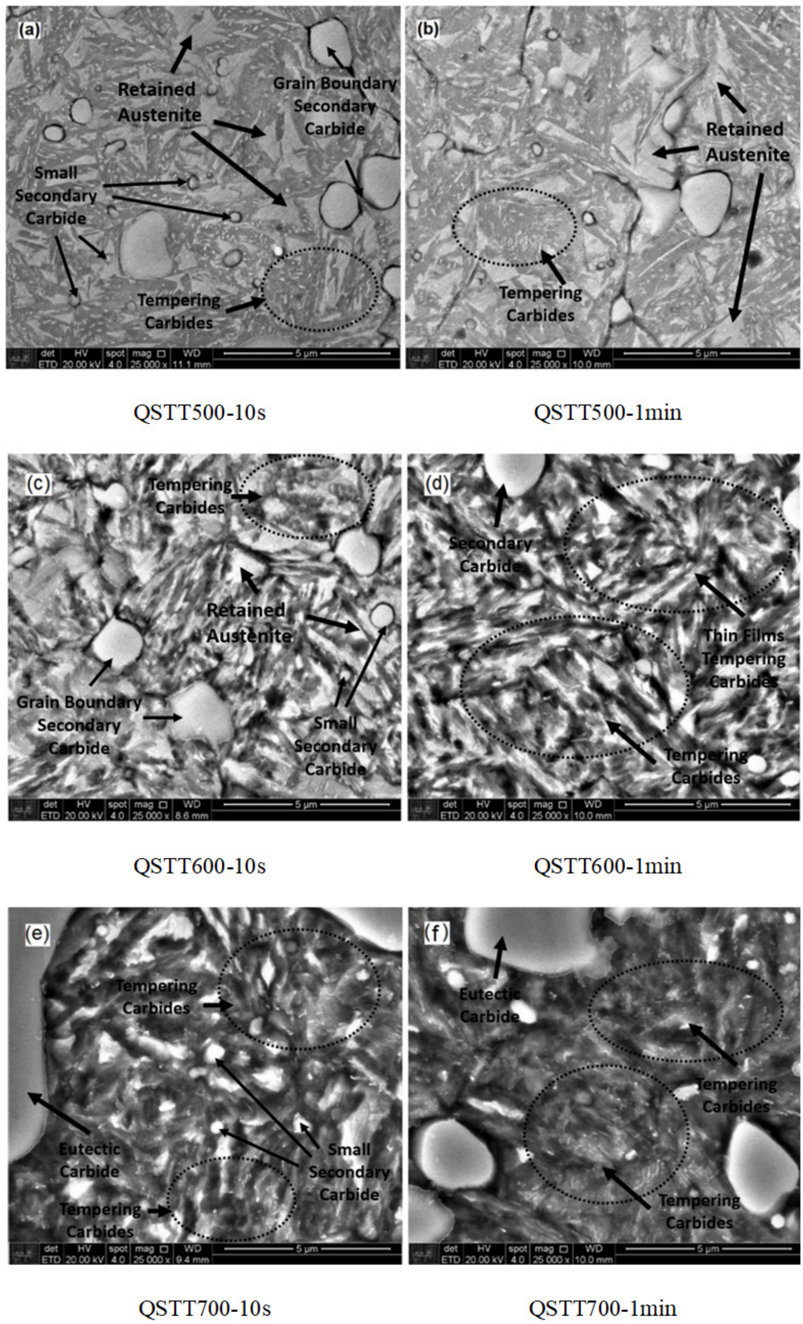

Figure 4. AISI D2 tool steel microstructures referring to double tempering cycles in short times at 500 ºC, 600 ºC and 700 ºC for 10 seconds or 1 minute: (a) QSTT500-10s; (b) QSTT500-1min.; (c) QSTT600-10s; (d) QSTT600-1min.; (e) QSTT700-10s and (f) QSTT700-1min. SEM. Nital etched.

out at 700 ºC, where hardness decreases with increasing treatment time. The maintenance of hardness levels for treatments carried out up to 600 ºC, when compared to the conventional tempering treatment carried out at 500 ºC for 2 hours, demonstrates that such tempering temperature is, possibly, within the predictability field for the parameter of Hollomon-Jaffe[1]. A sharp drop in hardness occurs for treatments performed at 700 °C, regardless of the treatment time. Hardness decreases with increasing treatment time, when comparing short tempering (for 10 s or 1 min.), due to precipitation and growth of tempering carbides and the associated matrix recrystallization that occurs in all cases when tempering is carried out at 700 °C. Differences in hardness values for treatment conditions referring to cycles with tempering at 700 ºC after 10 s and 1 min. are subtle and are due to the small difference in the maintenance of treatment time[11,13,15].

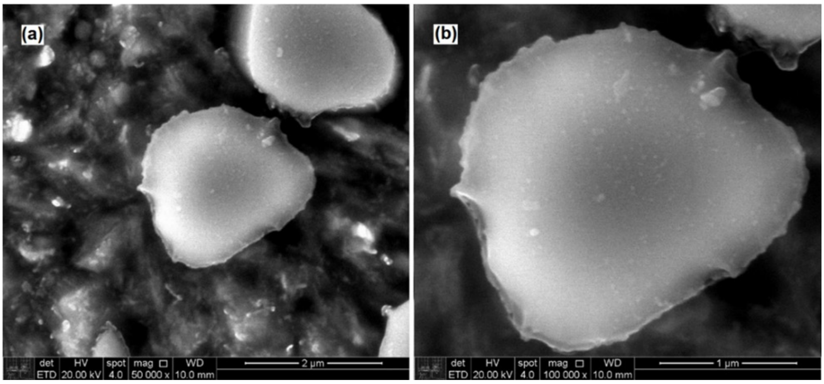

Figure 5. Microstructural details of the secondary carbides of the AISI D2 tool steel of the QSTT700-1 min. cycle, quenched and tempered at 700 ºC for 1 minute. SEM. Nital etched.

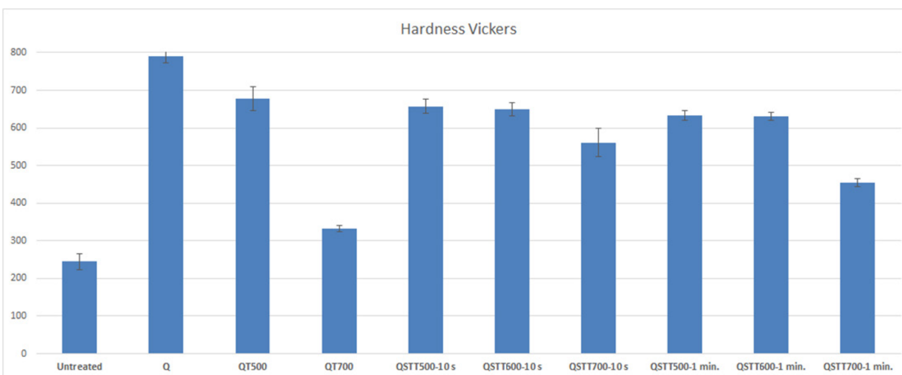

Figure 6. Vickers Hardness Measurements.

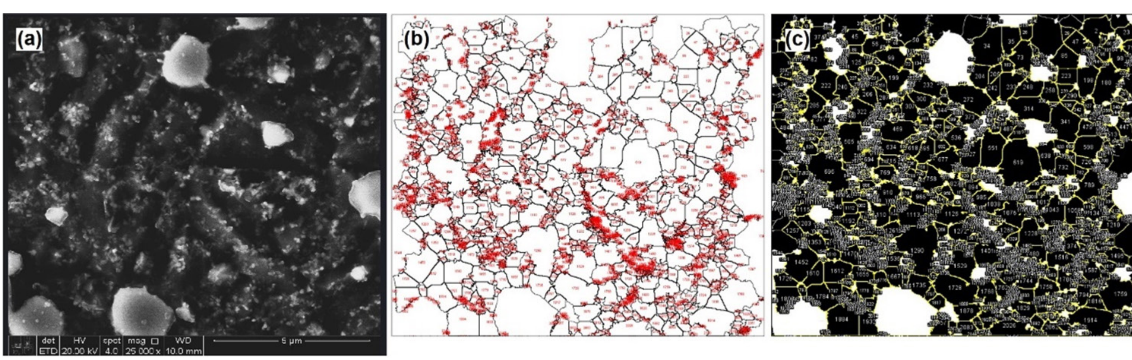

Figure 7. Distribution of carbides with the support of the image analysis program (ImageJ), (a) SEM image of the sample quenched and tempered at 700 ºC for 2 hours, images from the analysis program referring to the measurements of, (a) portion of tempering carbides and (c) matrix.

The result of the study of the average size and distribution of the observed carbides is represented in Figure 7, referring to the quenched and tempered specimen at 700 ºC for 2 hours (Figure 7a), with the aid of the image analysis program, the portion of the tempering carbides (Figure 7b) and the matrix as a whole (Figure 7c).

The results of the quantification of decomposed retained austenite and precipitation of carbides in tempering are shown graphically in Figure 8, where the AISI D2 tool steel presented 5% of retained austenite combined with the precipitation of 1.4% of carbides after tempering at 500 ºC and an amount of 2% of remaining austenite with 3.3% of carbides for tempering treatments carried out up to 600 ºC and an amount of only 1% of austenite associated with precipitation of 5.8% of tempering carbides for treatments carried out at 700ºC, in all cases, regardless of treatment time.

The results of the quantification of decomposed retained austenite and precipitated tempering carbides shown in Figures 7 and 8, point to a decreasing amount of retained austenite combined with the increase and growth in tempering carbides precipitation with the elevation of the tempering temperature, that is, such events are mostly attributable to temperature and are less dependent on treatment time[11,12,13,15].

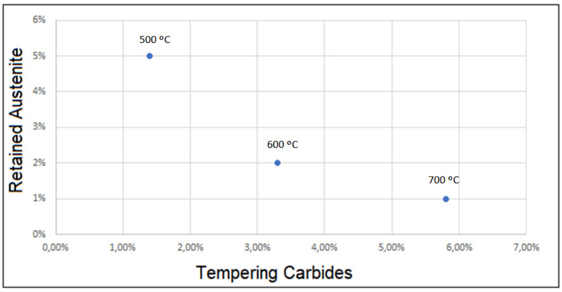

Figure 8. Retained austenite and precipitation of tempering carbides.

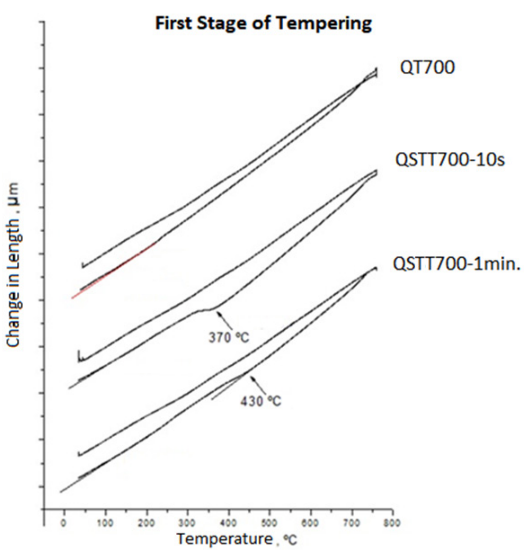

Figure 9. Dilatometry graphs of the first tempering at 700 °C at different times.

As shown in Figure 9, after the first tempering step, there was loss of linearity relative to the expansion on cooling at approximately 370 °C for treatments of 10 s and at 430 °C for treatments carried out for 1 min., all at 700 °C.

From another perspective, dilatometry results of the variation in length and the derivative of the variation in length with the temperature on cooling after the first tempering steps in short times at 700 ºC are presented in Figure 10.

As shown in the dilatometry study on the heat treatment of quenching and partitioning, in the same range and temperatures, more evidently demonstrated in Figure 10a, when short time tempering is performed for 10 s, it points to the occurrence of one or more events that happened during the heating, here it is believed that the decomposition of the retained austenite and formation of cementite or the precipitation and growth of carbides takes place. The XRD results indicate that in the tempering performed at 700 ºC, recrystallization occurred, in the same way as predicted in the tempering or quenching and partitioning studies, for higher temperatures[6,7,8,12].

Figure 11 shows the X-ray diffraction graphs, whose analysis suggests that there is a ferritization of the matrix in short tempering at higher temperatures.

The main qualitative interpretations of the diffraction graphs presented in Figure 11 are the increase in the diffracted peak relative to the ferrite phase in the samples submitted to treatment at higher temperatures at 700 ºC.

Comparatively, in all cycles where tempering treatments were carried out at 700 ºC, regardless of the time, the diffraction graphs tend to take the form of the diffractogram of the specimen without treatments, in the condition as received, denoting a tendency to ferritization of the matrix. Austenite is not stabilized in treatments at higher temperatures (600 ºC and 700 ºC), regardless of the time, contrary to expectations[7,8,9]. Additionally, it is possible to observe in the graph referring to the tempering treatments followed by tempering at 500 ºC for 2 hours and in short times also carried out at 500 ºC for 10 seconds or 1 minute, the remaining peak referring to the austenite phase, since, the treatment by 2 hours, or even a shorter time, at 500 ºC was not able to promote the removal of the austenite retained in the quench[4,5,9]. Higher tempering temperatures can lead to embrittlement with microstructural changes that may be

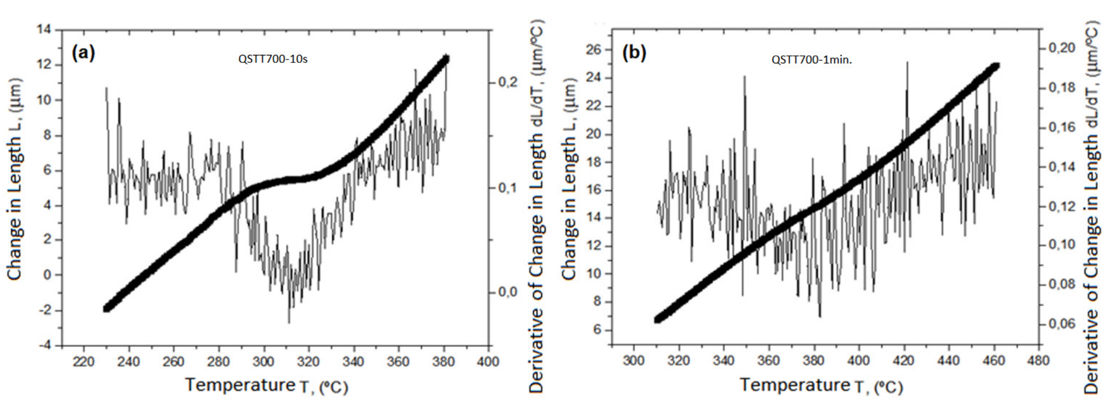

Figure 10. Dilatometry results: variation in length and derivative of the variation in length with temperature – Cooling after the first steps of Tempering of short-time tempering cycles at 700 ºC, for 10 s or 1 min. highlighting the regions at (a) 370 ºC or (b) 430 ºC, respectively.

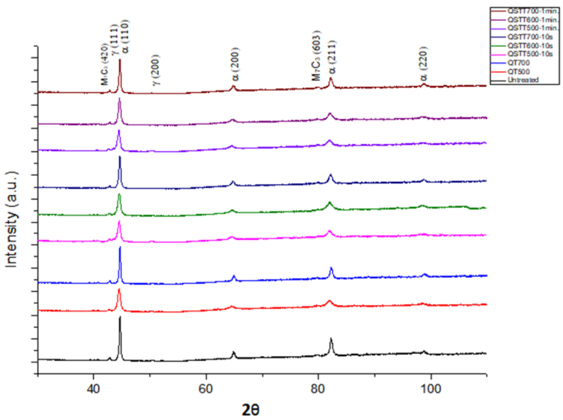

Figure 11. Diffractograms of the samples in conventional tempering and in short times tempering.

associated with either tempered martensite embrittlement or tempering embrittlement[3]. Short-term tempering of AISI D2 tool steel is recommended for temperatures close to secondary hardening peak[1].

4. Conclusions

Dilatometric analyzes allowed the identification of critical temperatures Ac1, Ac3 and Ms, with mean values of 835 °C, 905 °C and 230 °C, respectively. After the first tempering steps in short times at 700 °C, there was expansion during cooling at 370 °C for treatments of 10 seconds and at 430 °C for treatments carried out for 1 minute. The results showed the maintenance of hardness levels for tempering performed up to 600 °C for 1 min. or 10 s and a sharp drop in hardness for tempering at higher temperatures (700 °C) for 2 hours, 1 minute or 10 seconds. Short tempers at higher temperatures induce matrix ferritization, possibly due to the decomposition of retained austenite and carbide precipitation, depleting the carbon matrix, producing an annealing-like effect or subcritical recrystallization. The results point to a limitation on the use of the Hollomon-Jaffe parameter, for temperatures around the hardening peak in steels that present the phenomenon of secondary hardening, such as the AISI D2 tool steel, and open up a field of possibilities for the application of short times tempering in this temperature range.

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