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Study of the Effect of Multiple Tempering on the Impact Toughness of Forged S690 Structural Steel

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Abstract: During the production of forged metal components, the sequence of heat treatments that are carried out, as well as hot working, remarkably influences mechanical properties of the product, in particular impact toughness. It is possible to tailor impact toughness by varying tempering temperature and soaking time after hardening treatment, widening the application range of structural steels. In this work, we consider the effects of a second tempering treatment on the microstructural properties and impact toughness of a structural steel EN 10025-6 S690 (DIN StE690, W. n: 1.8931). The steel was first forged and quenched in water after austenitization at 890 °C for 4 h. After quenching different tempering treatments were performed, at 590 °C in single or multiple steps. The effect of these treatments was evaluated both in microstructural terms, by means of optical microscopy, scanning and transmission electron microscopy and X-ray diffraction, and in terms of impact toughness. The mechanical behavior was correlated with the microstructure and a remarkable increase in impact toughness was found after the second tempering treatment due to carbide shape change.

Keywords: tempering; impact toughness; carbides; heat treatment; bainite; steel

1. Introduction

S690 is a low-alloy high strength steel mainly employed in structural applications. One of the typical uses of this steel grade is offshore applications, where high impact toughness even at low temperatures is required. In these applications, the steel is supplied with a mainly bainitic microstructure. Moreover, considering the low carbon content this steel is also characterized by a relatively good weldability [1,2]. In more detail, in order to obtain the desired properties, high strength steels are formed by forging and thereafter undergo a quenching and tempering treatment [3–5]. One of the key mechanical properties of steels employed in structural applications is impact toughness. However, impact toughness is not related only to the chemical composition of the metal but can be significantly modified by deformation route or/and heat treatments [6]. Among the different heat treatments, quenching and tempering are used extensively in the forging industry. Tempering efficiency in forging is important both for mechanical properties achievement as well as for environmental and economic reasons. Since treated parts and equipment are commonly huge, the related time

for the treatment and natural gas consumption is high and onerous, hence any optimization in the treatment cycle (and of course on furnaces insulation) can reduce atmospheric emissions and produce economic saving. The tempering treatments performed for long soaking times seem to significantly increase the impact toughness of different steel grades [7,8]. This is due to bainitic carbides spheroidization, that produces an increase in mechanical properties by reducing stress concentration [9,10]. The modification of carbides morphology in fully bainitic steels depends on the carbides type. For example, as evidenced by Depinoy et al. [11], for a 2.25Cr-1Mo steel four types of carbides can precipitate depending on tempering temperature and time: M_3C , M_2C , M_7C_3 and $M_{23}C_6$. M_3C is the least stable carbide and is characterized by lenticular-globular morphology, M_2C and M_7C_3 are characterized by a needle like and rhombus shaped morphology, $M_{23}C_6$ is the equilibrium carbide and possesses a rod-like morphology. Generally, a more spherical shape of the carbides can lead to an increase in the impact toughness [12].

Impact toughness of quenched and tempered components can be strongly decreased also by the presence of retained austenite after tempering due to: (i) precipitation of interlath cementite aided by partial thermal decomposition of interlath films of retained austenite; (ii) subsequent deformation-induced transformation on loading of remaining interlath austenite, which has become mechanically unstable due to carbon depletion as a consequence of carbides precipitation as demonstrated by Horn et al. [13]. This problem can also affect S690 steel, as observed by Yen et al. [14] who proposed a new steel composition for offshore applications to overcome the problem. Another possible approach that can solve the problem is to decompose retained austenite using multiple tempering treatment [15,16].

However, even if S690 steel is a commonly used structural steel, very few works regarding the effect of heat treatments on its impact toughness and the microstructure can be found in literature. In particular, one work by Duan et al. regarding the effect of quenching temperature on the microstructure [17], one by Dong et al. [18] about the effect of the quenching and partitioning treatment, and one by Quin et al. [19] where different heat treatments, with an orthogonal experiment, have been performed, were found in literature.

The aim of the present work is to study the difference between a single long-lasting tempering treatment compared to multiple tempering treatments on forged and quenched S690 steel and to verify whether a connection exists between the microstructural changes and the variations, aiming to an improvement in the impact toughness.

2. Materials and Methods

Test samples came from a production forging Bush (forging ratio of >4:1 from starting ingot), with dimensions $\Phi 2520\text{ mm} \times \Phi 2130\text{ mm} \times H 1417\text{ mm}$, and weighing about 16 tons. A test ring was cut after heat treatment of the forging bush (austenitization at $890\text{ }^{\circ}\text{C}$ for 4 h, quenching in water, cooling rate approximately $115\text{ }^{\circ}\text{C/s}$ and tempering at $620\text{ }^{\circ}\text{C}$ for 6 h) as reported in Figure 1A, and cut again to obtain samples $60\text{ mm} \times 40\text{ mm} \times 50\text{ mm}$ (Figure 1B) to perform heat treatments in lab scale. Standard V-notch impact test samples were machined transversally to the major forging direction, as reported in Figure 1C.

Composition of the steel is reported in Table 1. Considering the different heat treatments performed on the laboratory scale samples three parts of the test ring were austenitized at $890\text{ }^{\circ}\text{C}$ for 4 h and quenched in agitated water at $30\text{ }^{\circ}\text{C}$. Two parts of the test ring were tempered at $590\text{ }^{\circ}\text{C}$ for 6 h or at $590\text{ }^{\circ}\text{C}$ for 10 h. Another part of the test ring was tempered at $590\text{ }^{\circ}\text{C}$ for 6 h and tempered for a second time at $590\text{ }^{\circ}\text{C}$ for other 4 h. In this way, the behaviors of samples with the same total tempering time but performed either in a unique treatment of 10 h or in two treatments of 6 h + 4 h were compared.

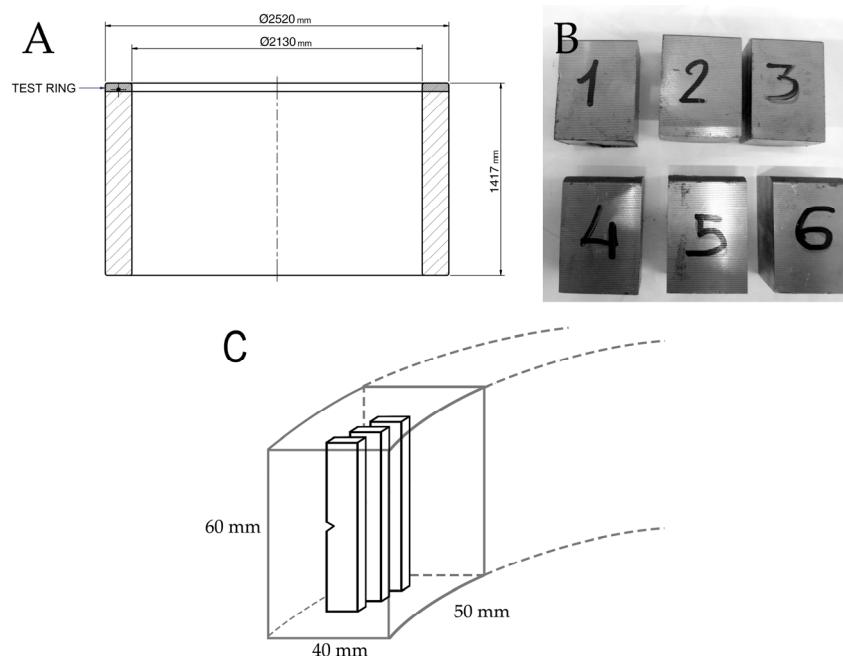


Figure 1. Drawing of the forging bush and of the position of the test ring (A); test ring sections used for the laboratory scale heat treatments (B); sketch of the extraction of the Charpy specimens from the test ring (C).

Table 1. Composition of the S690 steel (wt.%).

C%	Mn%	Si%	Cr%	Ni%	Mo%	V%
0.14	1.29	0.31	1.11	1.11	0.27	0.044

The sequence of heat treatments performed in the case of the double tempering of laboratory scale test ring sections is summarized in Figure 2.

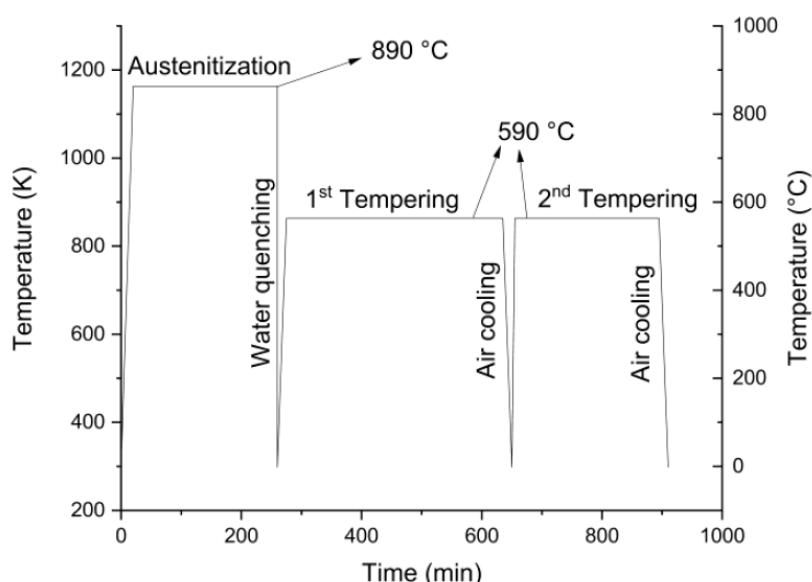


Figure 2. Heat treatment performed on the double tempered sample: austenitization at 890 °C for 4 h, first tempering at 590 °C for 6 h and second tempering at 590 °C for 4 h.

In the case of quenching and single tempering, carbides precipitation sequence was modeled with JMatPro® software (7.0.0, Sente Software Ltd., Guildford, UK).

Mechanical properties were evaluated by means of Vickers HV_{0.2} micro-hardness tests using a Leitz Miniload 2 (Leica Microsystem S.r.l., Milan, Italy) and Charpy-V impact toughness tests at room temperature and at -22 °C by means of a Charpy Wolpert pendulum (Wolpert Wilson Instruments, Aachen, Germany). In order to confirm the reproducibility of the results, for each heat treatments combination, five micro hardness and three impact toughness measurements were performed. Mechanical properties were linked to the microstructure that was analyzed by a LEICA DMRE optical microscope (Leica Microsystem S.r.l., Milan, Italy) and a Leica Cambridge Stereoscan LEO 440 scanning electron microscope (Leica Microsystem S.r.l., Milan, Italy). For the microstructural analysis, the samples were cut and mounted in phenolic resin, and then polished with standard metallographic preparation technique (grinding with abrasive papers 320, 500, 800, and 1200 grit, followed by polishing with clothes and diamond suspensions 6 µm and 1 µm). After the polishing step, the samples were etched with Nital 5% reagent immersing the sample for 5 s. Both the observations with Optical Microscope (OM) and Scanning Electron Microscope (SEM) were performed on the etched samples. For SEM analysis secondary electron mode was employed. The microstructure of the samples after different tempering treatment was compared with the one of the quenched sample and the one of the initial forging bush after heat treatment. Both micro-hardness and microstructural analysis were performed on Charpy specimens in regions of the samples far from the V notch. In order to investigate the possible presence of retained austenite, X-ray diffraction (XRD) measurements were performed. XRD analysis was carried out using a Siemens D500 diffractometer (Siemens, Munich, Germany) equipped with Cu tube radiation and a graphite-monochromator on the detector side with step size (2θ) of 0.05° and counting time of 5 s/step. The effect of possible texture was eliminated by a rotating fixture on the goniometer. To correlate more deeply the microstructure with mechanical properties, transmission electron microscope analysis was performed as well. The observations were performed with a JEOL 200CX transmission electron microscopy (JEOL Ltd., Tokyo, Japan). Sample preparation for transmission electron microscopy consisted in mechanical thinning to 50 µm, cutting of 3 mm diameter disk and perforation with Twin-Jet polishing technique, using a solution of 95% acetic acid and 5% perchloric acid as electrolyte at 50 V and room temperature.

3. Results

3.1. Micro-Hardness and Impact Toughness Evaluation

The results of the characterization on the different samples in terms of micro-hardness and impact toughness at room temperature and at -22 °C are summarized in Table 2.

Table 2. Micro-Hardness and Impact Toughness of the forged S690 steel after different heat treatments.

Treatment	Micro-Hardness HV _{0.2}	Impact Toughness Room T (J)	Impact Toughness -22 °C (J)
Water Quenched (after austenitization 890 °C 4 h)	400 ± 5	-	-
Quenched and Tempered 590 °C 6 h	276 ± 5	220 ± 10	48 ± 10
Quenched and Tempered 590 °C 10 h	275 ± 6	237 ± 9	60 ± 8
Quenched and Tempered 590 °C 6 h + second tempering 590 °C 4 h	272 ± 6	282 ± 9	128 ± 10

From the previously reported data, it can be clearly observed that the different tempering treatments do not affect the hardness of the steel. In fact, this property does not vary between the sample quenched and tempered and the samples with the second tempering treatment. Clearly, a reduction from the hardness of the quenched sample was observed for all the tempered samples.

Considering the impact toughness, more significant variations between the various samples can be noticed. In the tests at room temperature, an increase of about 60 J in impact toughness can be observed, passing from the quenched and tempered samples (6 h or 10 h) to the sample with the second tempering treatment. In detail, the sample that showed the higher value of impact toughness is the sample treated with the second tempering at 590 °C for 4 h that exhibits an impact toughness about 60 J higher than the sample quenched and tempered in one step. The difference in the impact toughness between the samples with and without the second tempering treatment is even higher at –22 °C. In this case, the impact toughness raises from 48 J (60 J) of the samples quenched and tempered for 6 h (10 h), to 128 J of the samples which also underwent the double tempering (6 + 4 h) heat treatment sequence.

3.2. Microstructural Characterization

In order to correlate the evidences regarding the impact toughness with microstructural changes OM, SEM and TEM observations were performed on all the tempered samples. For comparison the OM microstructures of the initial forging bush after quenching and tempering (Figure 3A) and the test ring samples after quenching (Figure 3B) are also reported.

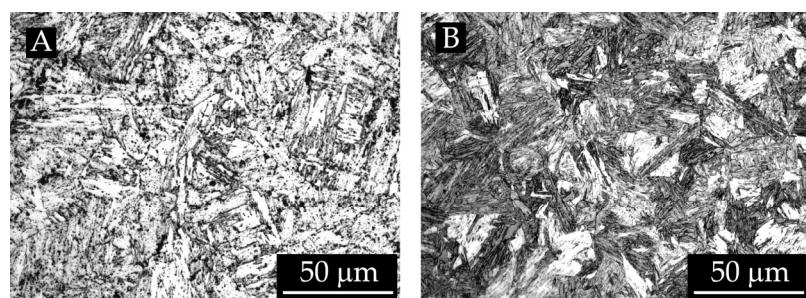


Figure 3. OM micrographs of the initial forging bush after austenitization at 890 °C for 4 h, water quenching and tempering at 620 °C for 6 h (A) and of test ring samples after austenitization at 890 °C for 4 h and water quenching in laboratory scale furnace (B).

From the micrograph reported in Figure 3A the expected microstructure composed by bainite and tempered martensite in the initial forging can be observed. After quenching (Figure 3B), the microstructure is composed by lath martensite and mixed bainite.

After water quenching, the test ring slices underwent different tempering treatments, as previously described, the microstructure of which is shown in Figure 4.

Comparing OM images of Figure 4, it appears that there is no significant microstructural difference among the samples. In fact, microstructure of all samples is substantially composed mainly by bainite with the presence also of low amount of tempered martensite.

In order to investigate in deeper detail possible microstructural differences that can explain the obtained improvement in impact toughness, SEM observations were performed on the three samples, whose results are reported in Figures 5–7.

From the SEM images, the mainly bainitic microstructure of all the samples is confirmed. In all the samples it can be also observed the presence of small carbides (white spots) located mainly along the prior austenitic grain boundaries and in-between bainitic plates. Comparing micrographs of Figures 5–7, no significant differences in the microstructure can be observed even in at higher magnifications.

One of the microstructural changes that can affect impact toughness in this steel, as previously described, is the presence of retained austenite, that is not visible in the OM images. To confirm the absence of retained austenite, XRD analyses were performed and the results can be observed in Figure 8.

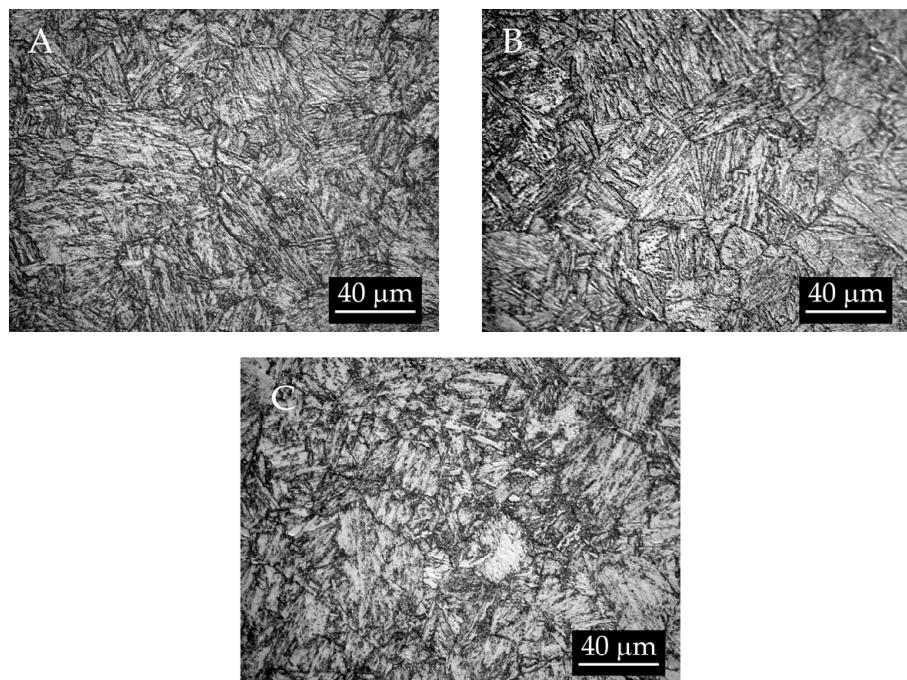


Figure 4. OM micrographs of the samples after different heat treatments: quenched and tempered at 590 °C for 6 h (A), quenched and tempered at 590°C for 10 h (B), quenched and tempered at 590 °C 6 h + 590 °C 4 h (C).

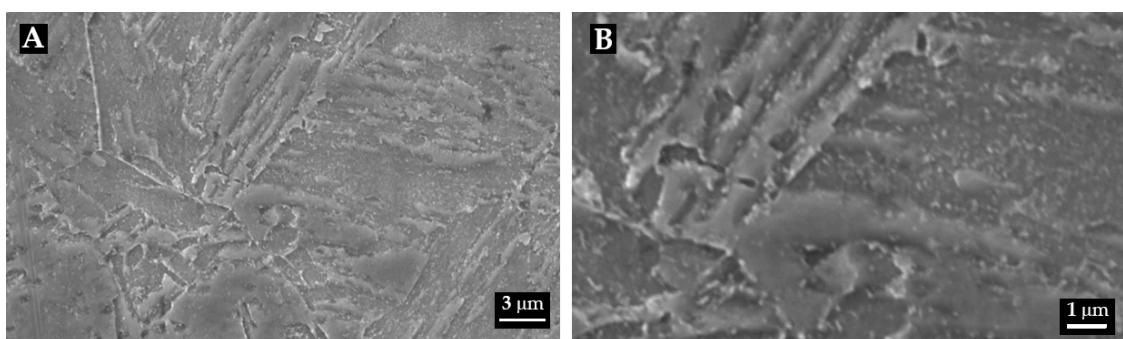


Figure 5. SEM micrographs of the sample quenched and tempered at 590 °C for 6 h at low magnification (A) and higher magnification (B).

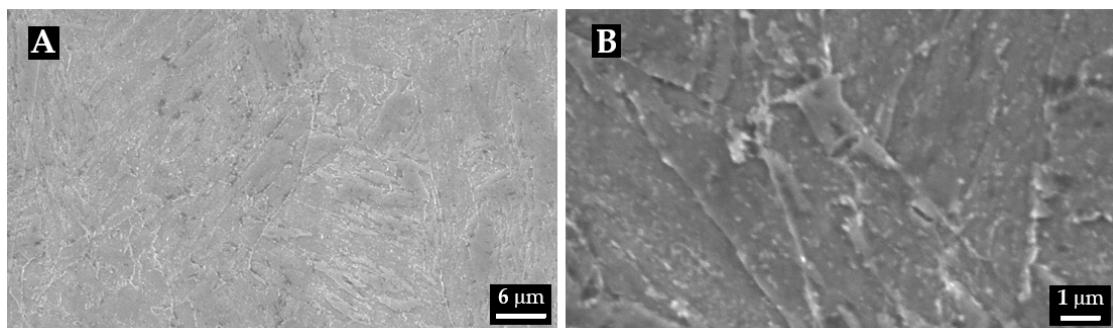


Figure 6. SEM micrographs of the sample quenched and tempered at 590 °C for 10 h at low magnification (A) and higher magnification (B).

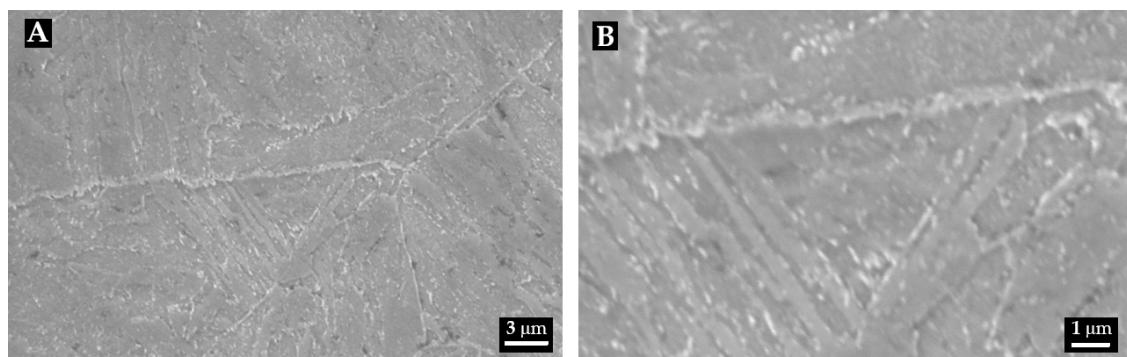


Figure 7. SEM micrographs of the sample quenched and tempered at 590 °C for 6 h + 590 °C 4 h at low magnification (A) and higher magnification (B).

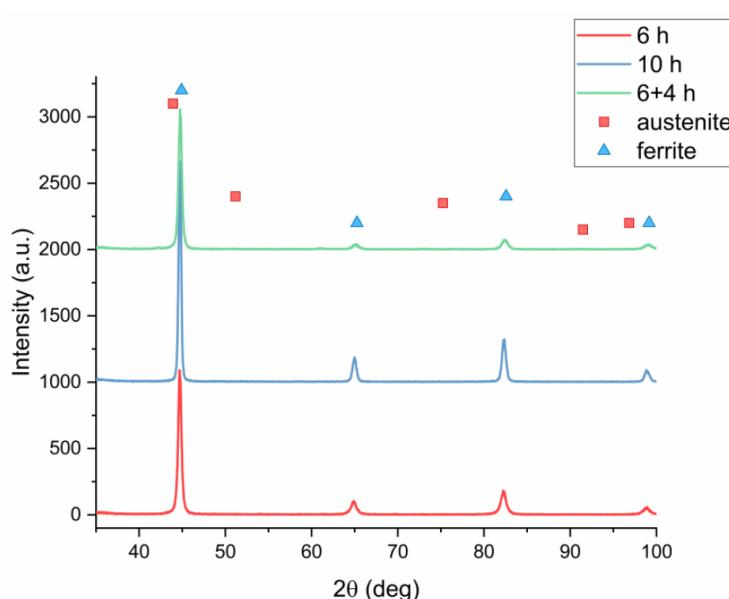


Figure 8. XRD analysis of the samples after different heat treatments: quenched and tempered at 590 °C for 6 h (red line); quenched and tempered at 590 °C for 10 h (blue line); quenched and tempered at 590 °C 6 h + 590 °C 4 h (green line).

From XRD analysis, retained austenite was not detected in any of the samples. In fact, only the peaks pertaining to ferrite can be noticed in the XRD patterns.

The shape and distribution of carbides can significantly affect the impact toughness. However, due to the small size of carbides, from SEM observation it was not possible to analyze and compare their shapes. In order to further investigate these microstructural features, TEM analysis on the three samples were performed and the results are reported in Figure 9. The images confirm the mainly bainitic microstructure with the presence of mixed upper and lower bainite, with carbides that can be found both inside and along the bainitic plates in addition to the one's observable at the grain boundaries. Significant variations in the shape of the carbides can be noticed. In detail, in the sample quenched and tempered at 590 °C for 6 h the carbides are lenticular-shaped, as shown in Figure 9A (red circles). Increasing the tempering time to 10 h (Figure 9B) produces an increase in the carbides average dimension, however, keeping an almost lenticular shape. With double tempering 6 h + 4 h (Figure 9C) a more globular shape can be noticed. The evolution in the shape of the carbides (i.e., from lenticular to globular) produces the observed increase in impact toughness. However, the hardness remains constant, despite the increased soaking time or number of steps in the tempering treatment.

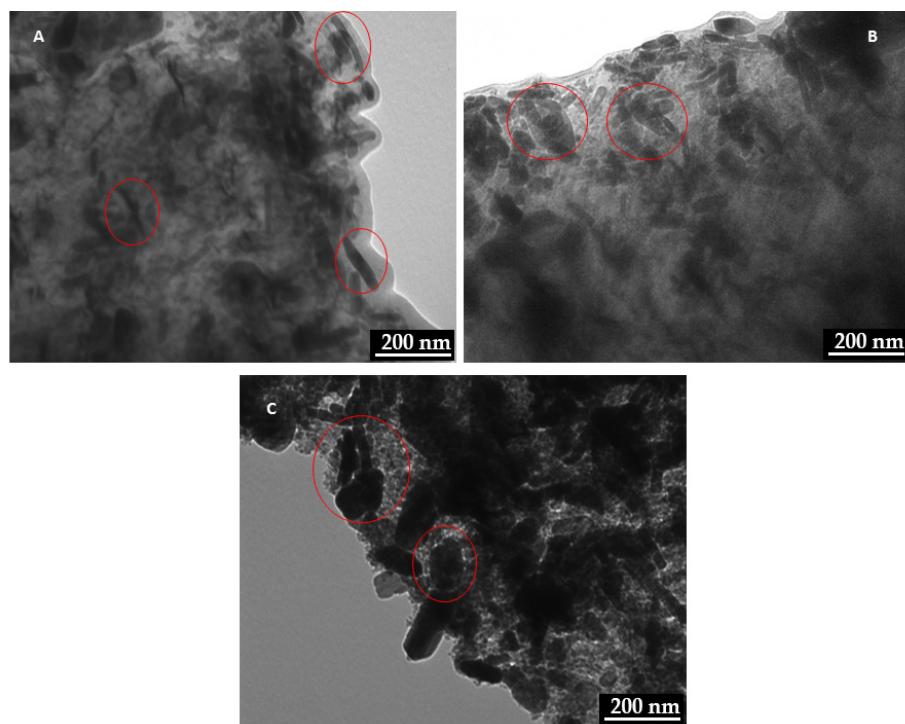


Figure 9. TEM micrographs of the samples after different heat treatments: quenched and tempered at 590 °C for 6 h (**A**), quenched and tempered at 590 °C for 10 h (**B**), quenched and tempered at 590 °C 6 h + 590 °C 4 h (**C**).

4. Discussion

The results previously reported highlighted that for S690 steel a double tempering of 6 h and 4 h after quenching permits to achieve increased impact toughness if compared to a single tempering of 6 h or 10 h. This behavior is not related to the presence of retained austenite (absent according to XRD analysis evaluation). All the samples present a mainly bainitic microstructure with carbides at prior austenitic grain boundaries and in-between bainitic plates. Also, the presence of tempered martensite was observed. The shape of such carbides is the key of the mechanical behavior of the samples: indeed, passing from single to double tempering it was recorded a modification from lenticular to globular shape. This combination of microstructural features causes the increase in impact toughness. This evidence is in accordance with literature results, obtained on different steels. In detail, Takebayashi et al. [20] found that carbide size and distribution significantly influence impact toughness of martensitic steels.

In this work, the modification in the morphology of carbides is linked with the type of heat treatment: a short soaking time (tempering at 590 °C for 6 h) does not permit the formation of a large number of lenticular carbides. Increasing treatment time (tempering at 590 °C for 10 h) produces an increase in the dimension of carbides but does not substantially modify their shape. Instead, after a double tempering (590 °C 6 h + 4 h) carbides morphology changes, which besides becoming coarser, acquires a spherical shape. However, the hardness of the steel was not affected by such combination of microstructural modifications.

In literature, it is reported that the following carbides, characterized by different thermal stability, can precipitate depending on tempering time and temperature: M_3C , M_2C , M_7C_3 , $M_{23}C_6$, and M_6C [11]. Increasing tempering time leads to evolution of the unstable carbides towards the equilibrium ones, whose nature depends on the steel composition. This event is usually accompanied by coarsening and spheroidization of carbides [21].

Carbides sequence precipitation was studied for S690 steel with software simulation in the case of quenching at 890 °C and single tempering at 590 °C, and the results are reported in Figure 10.

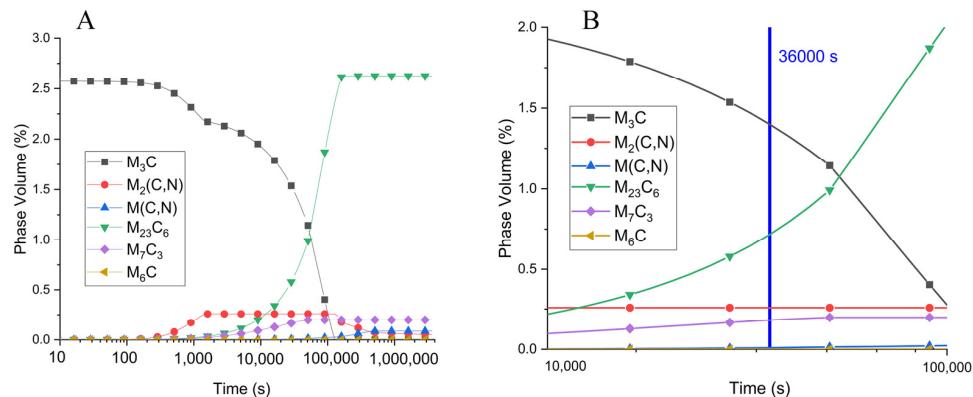


Figure 10. Results of the simulation of carbides precipitation during Quenching at 890 °C and Tempering at 590 °C for S690 steel performed with JMatPro® software; overview (A) and close up of the region near 10 h treatment time (B).

The diagram reported in Figure 10 confirmed, also for S690 steel, the results found in literature: the M₃C (characterized by lenticular morphology) is the first carbide to form and M₂₃C₆ (characterized by rod-like morphology) is the equilibrium carbide at this temperature.

In this work, it is likely that double tempering accelerates the transition from metastable to equilibrium carbides, by modifying the mechanism of carbon diffusion as well as diffusion of other elements.

Moreover, the evolution of carbide shape from lenticular to rod-like could explain the observed increase in impact toughness, as also observed by Dudova et al. [22] and Hao et al. [23]. This shape evolution is probably predominant in comparison to coarsening and this explains the almost constant hardness after the different treatments.

5. Conclusions

In this work, the effect of different heat treatments on a forged S690 steel was studied. Three different tempering treatments, performed after quenching, were studied: 590 °C for 6 h, 590 °C for 10 h and a double tempering at 590 °C for 6 h + 4 h. The different tempering treatments produce significant variations in impact toughness: the double tempering treatment results in a remarkable improvement in impact toughness of approximately 80 J, in comparison with the sample tempered for 6 h, and of about 60 J in comparison with the single 10 h tempering treatment. None of the samples contain retained austenite, as evidenced by XRD analysis, and are characterized by a mainly bainitic microstructure with the presence of carbides, as observed with OM, SEM, and TEM. The main difference among the various samples is due to the shape of carbides, as evidenced by TEM observation. In detail, with a two-stage tempering treatment, a morphological change was observed from lenticular to globular. These variations can be explained with the modification in the mechanism of transition from metastable to equilibrium carbides during the double tempering. In fact, the formation of the equilibrium carbides induces their spheroidization and can justify the recorded increase in the impact toughness.

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