

Preparation and Mechanical Properties of Nano/Ultrafine Bainitic Structure in AISI 52100 Steel

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Abstract

The development of nano/ultrafine bainitic structure in AISI 52100 steel was the goal of this study. For this purpose, the AISI 52100 the specimens were austenitized at 1050 °C for 60 min followed by low-temperature austempering treatment at different temperatures and times. The austempered samples were characterized using field emission scanning electron microscopy (FESEM), X-ray diffraction (XRD) and tension test. According to achieved results, the microstructure of AISI 52100 steel after the austempering treatment consisted of ultrafine bainitic ferrite plates and retained austenite with two morphologies of micrometer-block and fine film. At the austempering temperature up to 250 °C, the micrometer-blocky morphology of austenite completely vanished from the microstructure, and the strength and ductility increased to about 2000 MPa and 7 %, respectively. By increasing the austempering temperature to 300 °C, the strength and ductility reduced (to about 1808 MPa and 3 %) simultaneously as a result of changing in the shape and size of bainite phase.

Keywords: Nanostructures; 52100 steel; X-ray diffraction; Mechanical properties.

1. Introduction

AISI 52100 is a high carbon- low alloy steel which serves suitable ultimate strength, hardness and abrasion resistance¹⁾. Due to its properties, this steel is used in ball and roll bearings, punches, spinning tools and dies in usual hardened and tempered condition²⁾. Low fracture toughness and destructive residual tensile stress are the main problems of this steel³⁾. A number of attempts such as structural and compositional modification have been made to overcome these drawbacks. Among the different methods, formation of bainitic microstructure is the best way for increasing the ductility of this steel³⁻⁵⁾.

However, this steel is not quite amenable to austempering process and it may typically take several days to reach the desired degree of transformation. Furthermore, the mechanical properties of fully bainitic AISI 52100 are inferior to those in the same steel in usual hardened and tempered condition. Although the conventional bainitic structure may not be an at-

tractive proposition for ball-bearing applications, formation of ultrafine-bainitic (super-bainitic) structures in this steel may prove superior in terms of mechanical properties including hardness, tensile strength and toughness as compared to those obtained in the usual hardened and tempered conditions⁵⁾.

The super-bainitic steel represents a wide class of steels in which interesting mechanical properties (ultimate tensile strength and toughness of about 2300 MPa and 30 MPa.m^{1/2}, respectively) are obtained from relatively simple and cheap austempering transformation at low temperatures. This treatment intended to achieve a particular mixture of two phases in which very fine ferrite platelets alternate with thin films of retained austenite. This complex structure provides a combination of strength and ductility in steel⁶⁻⁹⁾.

Although, a lot of studies have been conducted about the formation and characterization of bainitic structure^{10,11)} in AISI 52100 steel, the exact effects of low temperature austempering treatment on the structure and mechanical properties of this steel has not been properly investigated. Therefore, this report attempts to determine the effect of low temperature austempering process on the structure and mechanical properties of AISI52100 steel.

2. Experimental Procedure

Cylindrical samples of 22 mm diameter and 5 mm thickness were cut from ingot of AISI 52100 steel with

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a nominal composition of 0.98%C, 1.41Cr, 0.23%Si, 0.33%Mn, 0.01%Mo, 0.01%S, 0.012%P and balance Fe (in wt.%). The samples were austenitized at 1050 °C for 60 min and then were rapidly transferred to a salt bath furnace in the temperature range of 150-300 °C. The austempered samples were mechanically polished with up to 0.1 μm diamond paste and etched with 2 mol.% Nital etching solution. The polished and etched samples were subjected to a detailed microstructural investigation using an optical microscope with an image-analyzer and a field-emission scanning electron microscope (VEGA-TESCAN-XMU) at an accelerating voltage of 20 kV. An X-ray diffractometer with Cu K α radiation ($\lambda=0.15406$ nm; 40 kV; Philips PW3710) was used to follow the structural changes of the specimens (2θ range: 20-120°, step size: 0.05°; time per step: 1 s). The volume fraction and carbon content of retained austenite were determined based on XRD results and using direct comparison¹²⁾ and lattice parameter measurement¹³⁾ methods, respectively. Room temperature mechanical properties of produced specimens were also conducted according to ASTM E8M-04 standard using an FM2750 testing machine.

3. Results and Discussion

There are several factors, such as austenitizing condition, quenching rate, the properties of salt bath furnace and austempering temperature and times which influence the structure and mechanical properties of the austempered specimens^{14,15)}. A slight variation in these processing parameters can often cause large variations in the microstructure and properties of produced samples. In the present study, all of these parameters were kept nominally constant and the effects of austempering condition on the structure and mechanical properties of AISI 52100 steel were investigated.

3.1. Austenitizing condition

For the formation of a bainitic structure, the initial carbide phases from as-received ingot, must be completely decomposed and the chromium and carbon elements be dissolved in Fe matrix. The SEM micrographs of AISI52100, after austenitizing at different conditions and quenching in water (25 °C), are presented in Fig. 1. According to these micrographs, the optimum austenitizing condition for the dissolution of all carbides in Fe matrix was recognized 1050 °C and 60 min. The average austenite grain size of AISI52100, after austenitizing at this condition, was about 20±4 μm. It is important to note that, the bainite (B_s) and martensite (M_s) temperature start of this steel was estimated about 310 and 90 °C, respectively.

3.2. Austempering treatment

3.2.1. Austempering at 150 °C

The engineering stress-strain curves (from tension tests at room temperature) of the austempered samples at 150 °C for different periods of time up to 288 h are presented in Fig. 2. According to these curves, all austempered specimens at this austempering temperature have been broken in elastic region and the ductility of produced samples is close to zero. In this regards, the SEM fracture surface micrograph of the austempered sample at 150 °C for 288 h is shown in Fig. 3 (a). As seen, this fracture surface consists of cleavage facets and confirms that the fracture behavior of this specimen is brittle.

The SEM micrographs of the austempered samples at this temperature for different periods of time are presented in Fig. 4. According to the figures, the microstructure of the austempered sample at 150 °C for 10 min (Fig.4 (a)), is mainly martensitic. By increasing the annealing time, the acicular bainite phase was formed in the microstructure and the volume percentage of this phase increased progressively to a constant value of about 60 %. Considering this point, during austempering treatment, the initial austenite phase (γ) was decomposed into bainitic ferrite (α_{BF}) structure, according to reaction (1).



This reaction is so similar to that occurring in austempered ductile cast iron¹⁴⁾. According to Fig. 3 (b), the retained austenite in this reaction consists of two morphologies: as micrometer-blocks ($\gamma_{MB} > 1000$ nm) between the sheaves of bainitic ferrite plates and as nano films ($\gamma_{NF} < 100$ nm) between the subunits of bainite.

The XRD patterns of the austempered samples at this temperature for different periods of time are also presented in Fig. 5. Moreover, the carbon contents and percentage of retained austenite of the austempered samples at 150 °C for different periods of time are also presented in Table. 1.

Table. 1. The carbon contents and percentage of retained austenite (based on XRD results and using lattice parameter measurement and direct comparison methods) after austempering at 150 °C for different periods of time.

	Austempering time			
	10 min	1h	24h	288h
Carbon content of γ_R (wt. %)	0.98	1.6	1.83	1.87
Volume percentage of γ_R	0	42.2	37.8	36.4

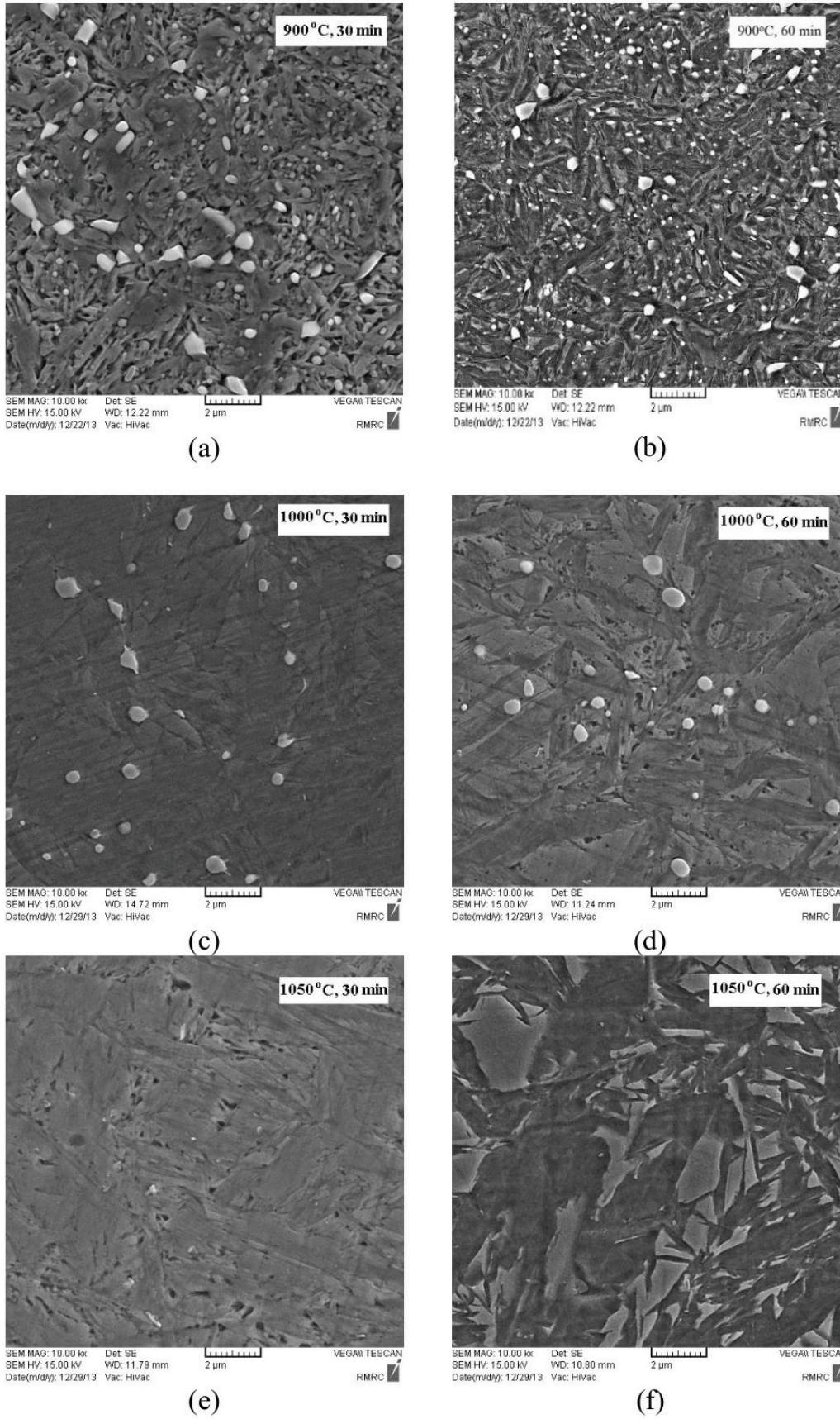


Fig. 1. SEM micrographs of as-received AISI52100 steel after austenitizing at different temperatures and time and quenching in cold water.

Regarding to these results, several points can be concluded as:

- The XRD patterns of these specimens are so similar and consist of ferrite/martensite and obvious retained austenite peaks. This result is in agreement with SEM findings.
- By enhancing the annealing time, the carbon content of retained austenite was increased progressively, and reached a constant value of about 1.8 wt.%.
- According to Table. 1, the percentage of retained austenite in the austempered sample at 150 °C for 10 min is close to zero due to insufficient stability of austenite phase at this condition. By increasing the austempering time, the percentage of bainite phase increases. The carbon content of bainite is so low and by formation of this phase from initial austenite, the surplus carbon was deposited in nearby austenite. By increasing the carbon content of un-decomposed austenite, the Ms value of retained austenite decreases. This point is the main reason of increasing the percentage of retained austenite in the structure of the austempered samples.

By more increasing the annealing time, the fraction of retained austenite slightly decreases and reaches a constant value of about 37 vol. %. This result confirms that the carbon-rich retained austenite is stable at 150 °C and its transformation to bainitic ferrite structure is a time-consuming process. According to the literature^{16, 17}, the carbon-rich retained austenite can be turned into high-carbon un-tempered martensite, during the deformation process. In fact, the percentage of retained austenite in these samples is so high and the formation of brittle un-tempered martensite during the deformation is the main reason of low ductility of the austempered samples at this temperature (Fig. 2).

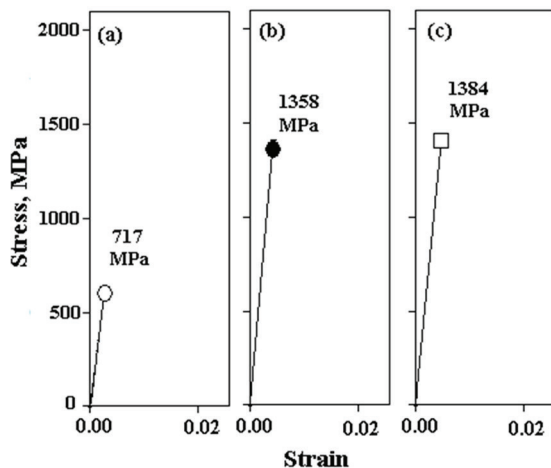


Fig. 2. Engineering stress-strain curves of the austempered samples at 150 °C for a) 96, b) 192, and c) 288 h.

3. 2. 2. Austempering at 200 °C

The engineering stress-strain curves of the austempered samples at 200 °C for different time are pre-

sented in Fig. 6. According to this figure, the fracture mechanism in these specimens is also brittle, and the amount of ductility is about zero. The SEM fracture surface morphology of these specimens also confirms this result (Fig. 7 (a)). In this regards, the SEM micrographs and the XRD pattern of the austempered specimen at 200 °C for 192 h are shown in Figs. 7 (b) and 8, respectively. As seen, the microstructure of this sample is similar to the micrograph of the austempered samples at 150 °C and consists of bainitic ferrite (α BF) and retained austenite. The volume percentage of retained austenite in this specimen (12.5 vol. %) is lower and the bainitic ferrite plate size (about 70 nm in thickness) is higher than the austempered sample at 150 °C. The same as above, the bainitic transformation has not been completed at this austempering condition (until 192 h) and the low ductility of produced samples can be related to the presence of retained austenite in the microstructure.

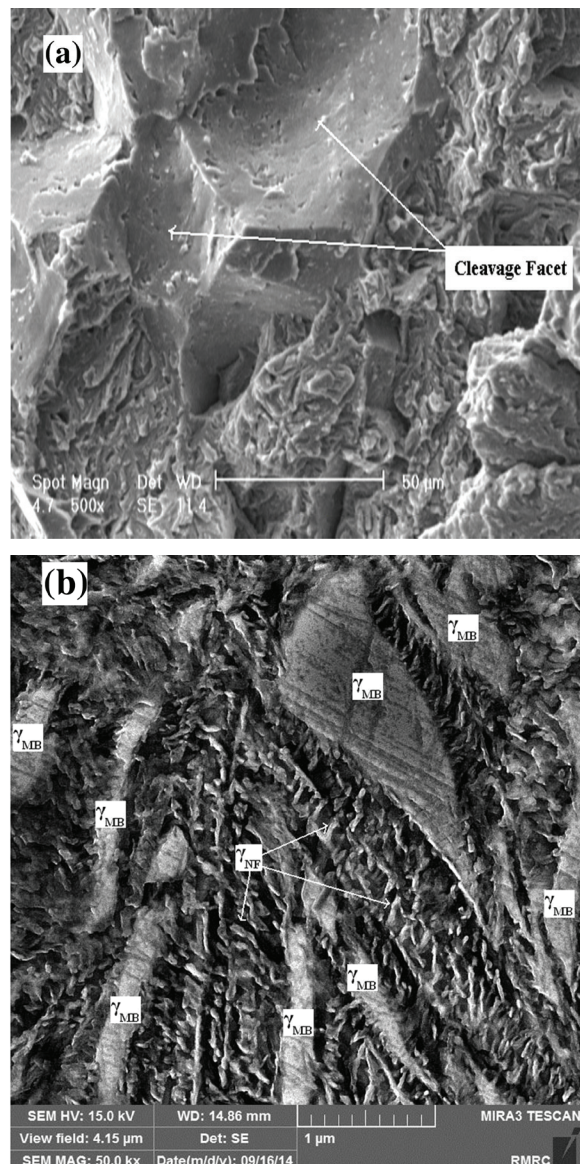


Fig. 3. SEM a) fracture surface and b) cross sectional micrographs of the austempered sample at 150 °C for 288 h.

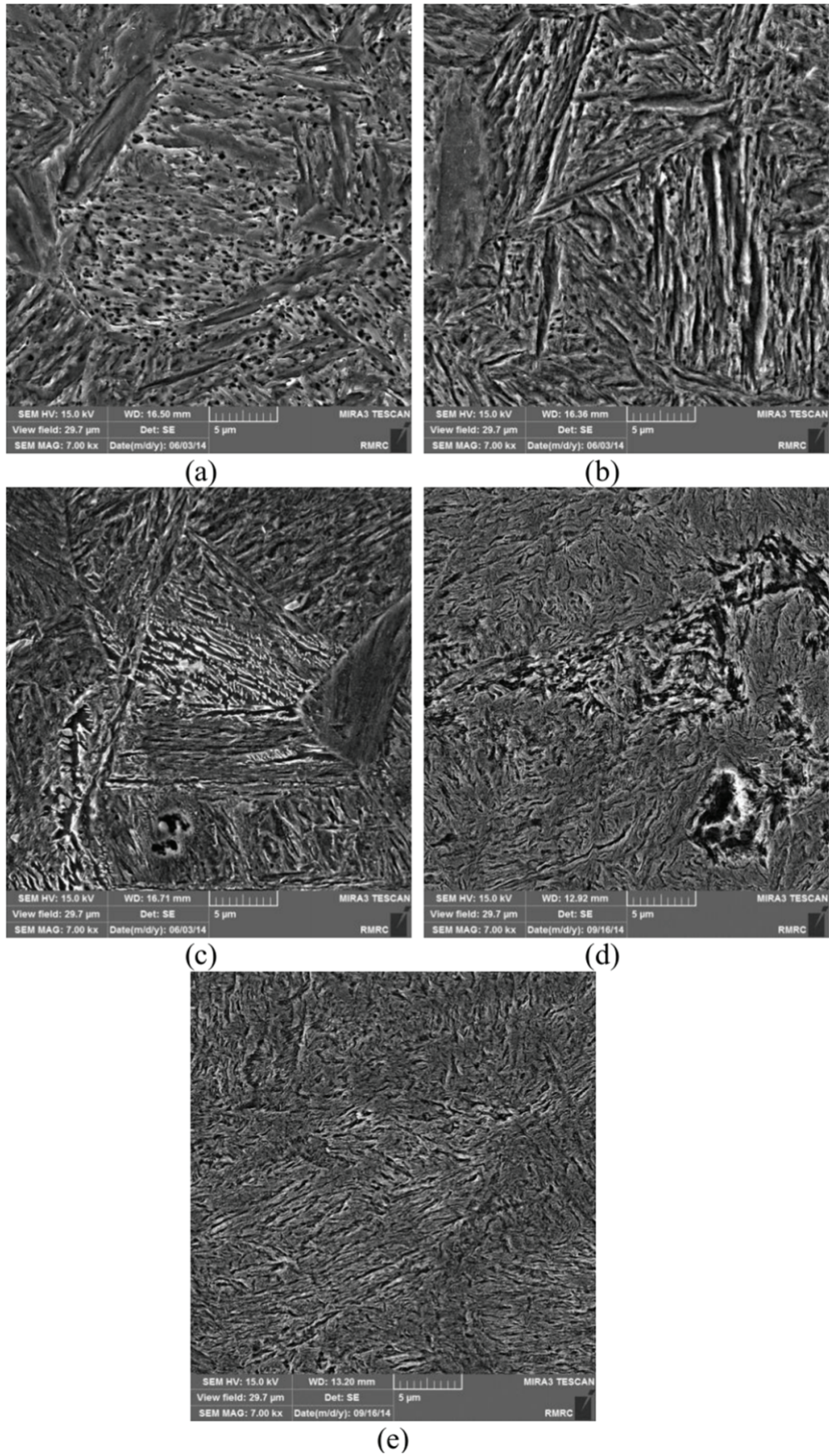


Fig. 4. SEM micrographs of the austempered samples at 150 °C for a) 10 min, b) 1, c) 24, d) 192 e) 288 h.

3. 2. 3. Austempering at 250 °C

The stress-strain curves of the austempered samples at 250 °C for different time are shown in Fig. 9. As seen, by increasing the annealing time in this austempering temperature, the mode of fracture has been changed from brittle to ductile and the ductility of produced specimens was increased to about 7 %. This result is in agreement with the fracture surface morphology investigations of these samples. For instance, the fracture surface morphologies of the austempered samples at 250 °C for 24 and 96 h are shown in Fig. 10. As seen, the fracture mode of the austempered sample at this temperature has been changed from brittle to ductile by increasing the austempering time from 48 to 96 h.

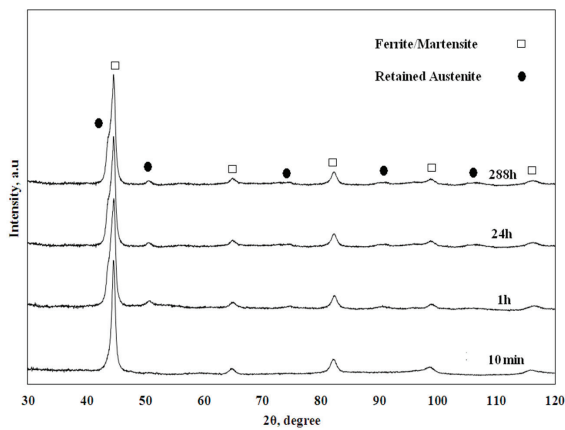


Fig. 5. XRD patterns of the austempered samples at 150 °C for different time.

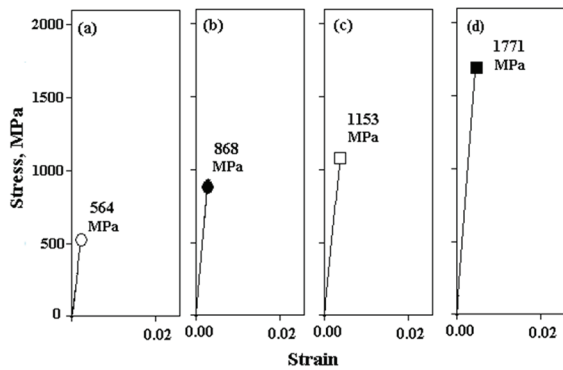


Fig. 6. Engineering stress-strain curves of the austempered samples at 200 °C for a) 48, b) 96, c) 144 and d) 192 h.

The FESEM cross-sectional micrograph and the XRD pattern of the austempered sample at 250 °C for 96 h are shown in Figs. 11 and 12, respectively. According to these figures, the bainitic transformation is completed at this austempering condition and the microstructure of this sample only consists of bainitic-ferrite (α_{BF}) and nano films (γ_{NF}) retained austenite. In fact, there is not any evidence of micrometer-block austenite phase in these microstructures. It is important to note that, decreasing the amount of retained

austenite from the microstructures is the main reason of increasing the ductility of these samples. Moreover, according to the literature¹⁸⁻²⁰, the nano-film retained-austenite in these samples acts as an obstacle against the crack growth and plays an important role in increasing the toughness of specimens. Of course, the amount of nano-film retained-austenite in these samples is so low (lower 5 vol.%) and this phase is virtually undetectable by XRD.

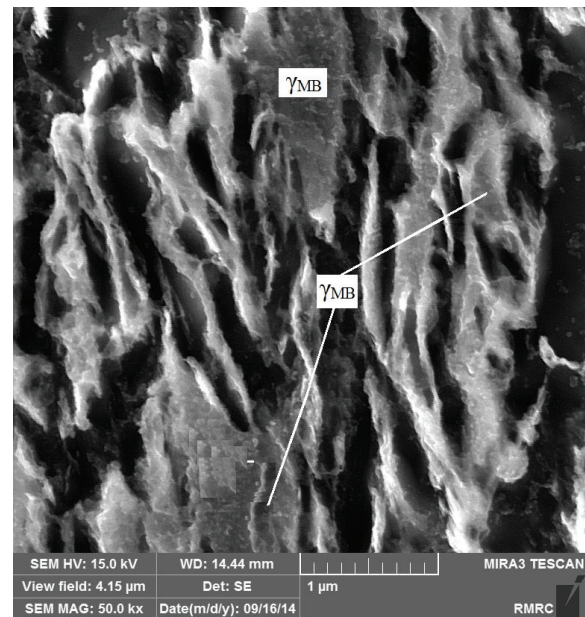
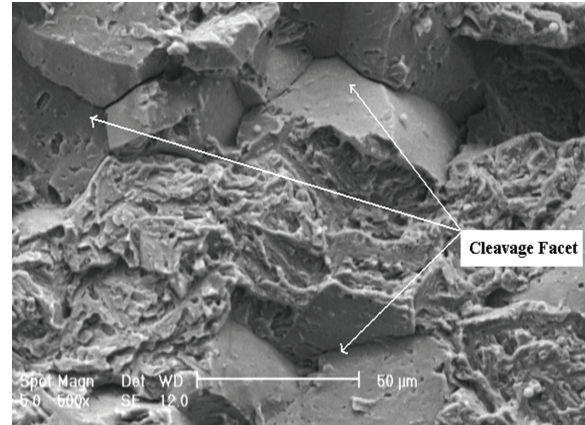


Fig. 7. SEM a) fracture surface and b) cross sectional micrographs of the austempered sample at 200 °C for 192 h.

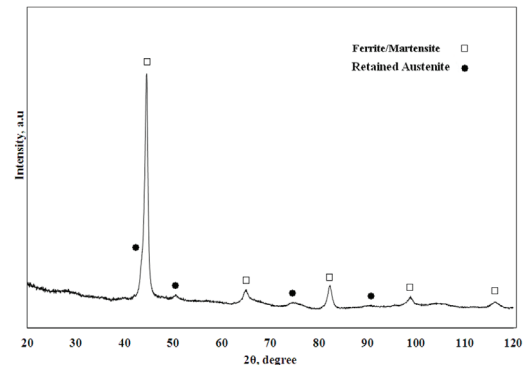


Fig. 8. XRD pattern of the austempered sample at 200 °C for 192 h.

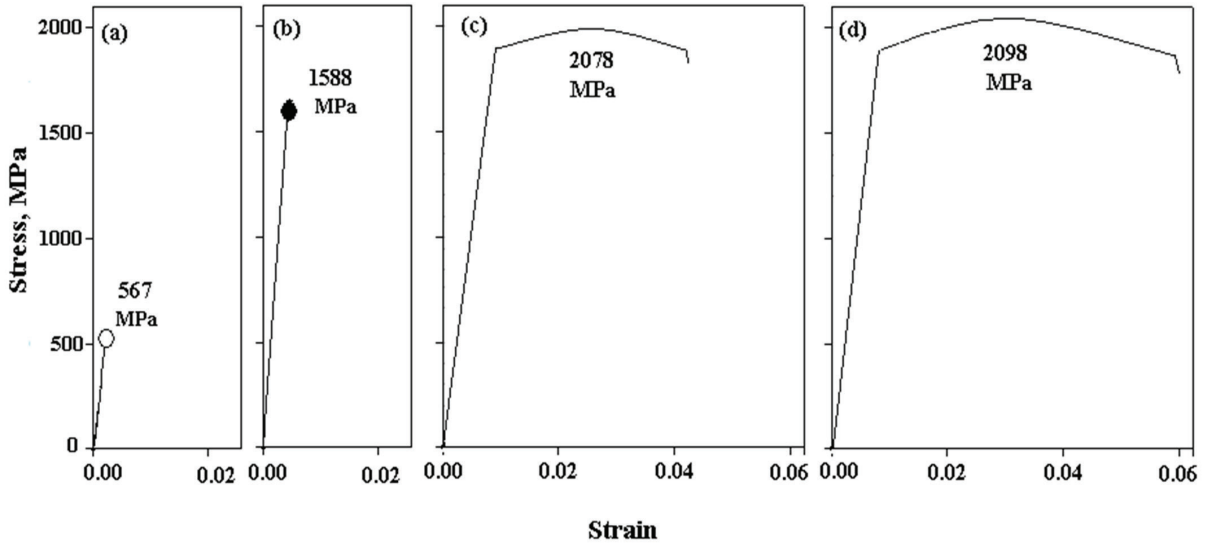


Fig. 9. Engineering stress-strain curves of the austempered samples at 250 °C for a) 1, b) 24, c) 48 and d) 96 h.

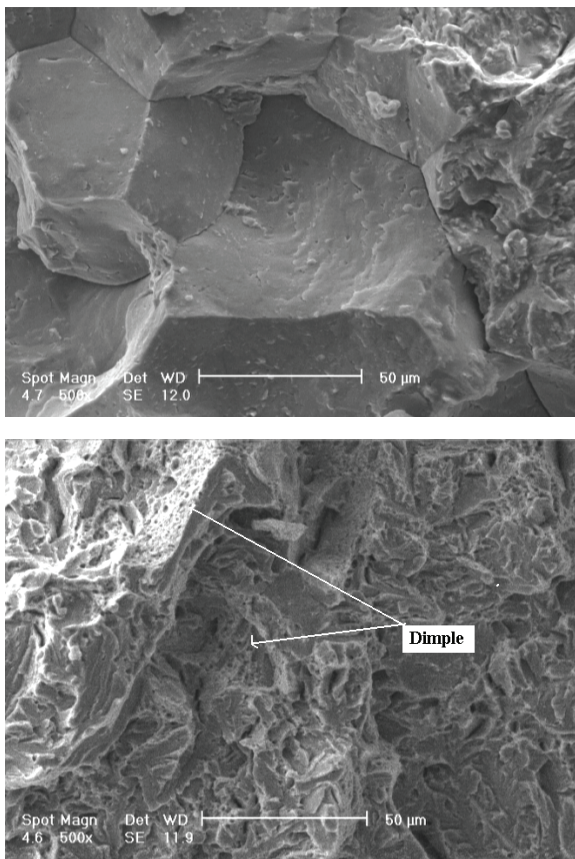


Fig. 10. SEM fracture surface micrographs of the austempered sample at 250 °C for a) 24 and b) 96 h.

To compare Fig. 6 and Fig. 9, it is clear that with an increase in the austempering temperature from 200 to 250 °C, the strength increases sharply and reaches about 2000 MPa. In fact, the high strength of produced samples in this condition can be explained by two important strengthening mechanisms:

a) Hall-Patch effect; according to the Hall-Patch equa-

tion ($\sigma=k/d^{0.5}$, where d is crystallite size and k is a constant value), there is an inverse correlation between the strength and the crystallite size of the specimen. In this equation, the high strength of the austempered samples can be related to the formation of thin plates of bainitic-ferrite (about 70 nm) in the microstructure²¹⁾.

b) The concentration of carbon atoms around dislocations: According to the previous investigations^{7, 22-24)}, the carbon content of bainitic-ferrite phase is higher than conventional equilibrium heat-treatments (as a result of the displacive mode of bainitic transformation). In this condition, the carbon atoms are trapped around the dislocation lines and reduced the mobility of them²²⁻²⁴⁾.

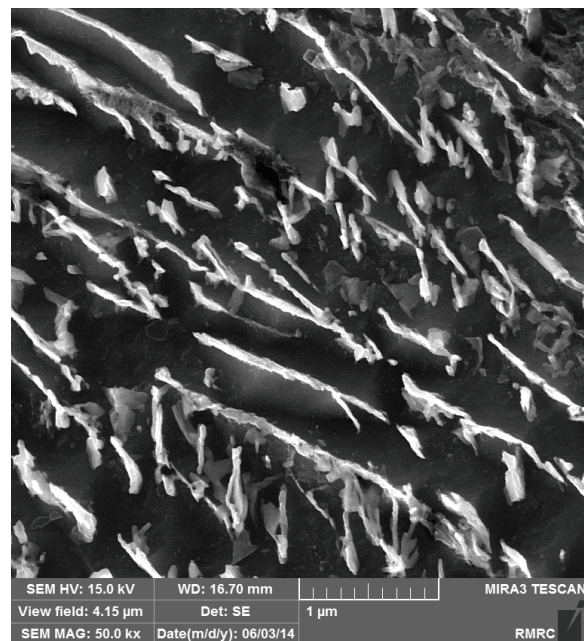


Fig. 11. FESEM micrograph of the austempered sample at 250 °C for 96 h.

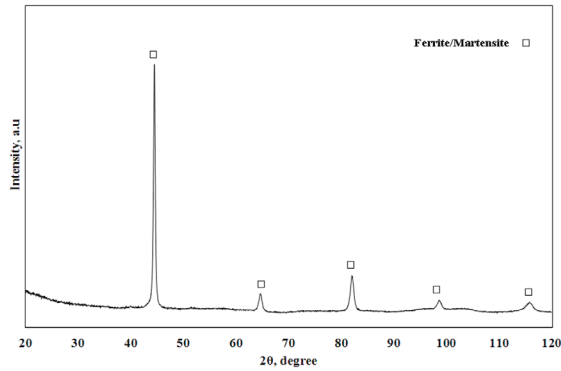


Fig. 12. XRD pattern of the austempered sample at 250 °C for 96 h.

3. 2. 4. Austempering at 300 °C

The same as above, the stress-strain curves of the austempered samples at 300 °C for 24 and 48 h are presented in Fig. 13. According to this figure, the strength and ductility of the austempered samples at 300 °C (1808 MPa, 3 %) is lower than prepared samples at 250 °C (2000 MPa, 7 %). In this regard, the cross-sectional micrograph and the XRD pattern of the austempered sample at 300 °C for 48 h are shown in Figs. 14 and 15, respectively. According to these figures, the structure of the austempered samples at 300 °C only consists of bainitic-ferrite phase, and there is no evidence of micrometer-block retained-austenite in the microstructure. The shape of bainitic-ferrite phase in these specimens is different from other austempering conditions and the transformation products do not have a layered structure. Moreover, the size of formed bainitic-ferrite after austempering at 300 °C is bigger than other austempering conditions which can be related to the lower nucleation rate and higher growth rate of ferrite, at higher temperatures⁶⁻⁹). Considering these results, reducing the strength and ductility of produced samples in this condition can be related to changes in the shape and size of formed phases.

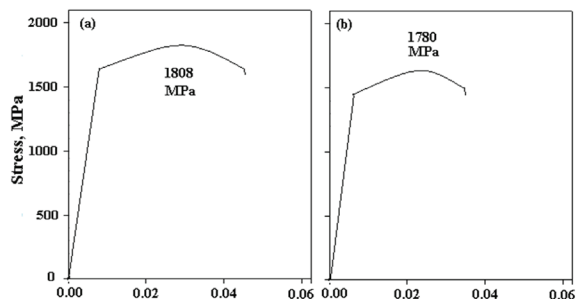


Fig. 13. Engineering stress-strain curves of the austempered sample at 300 °C for 24 and 48 h.

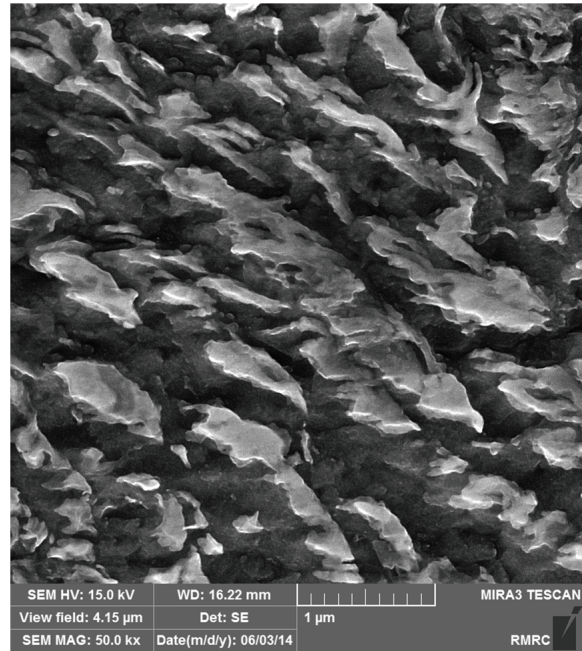


Fig. 14. FESEM micrograph of the austempered sample at 300 °C for 48 h.

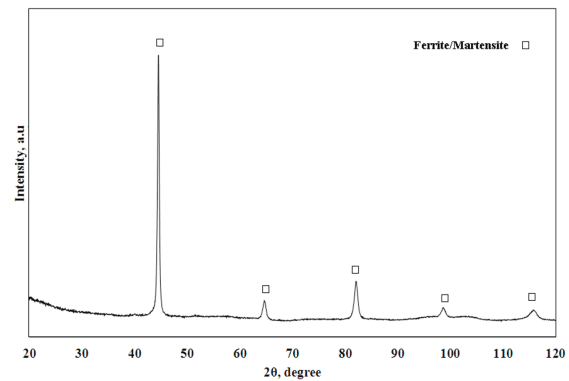


Fig. 15. XRD pattern of the austempered sample at 300 °C for 48 h.

4. Conclusions

The development of nano/ultrafine bainitic structure in AISI 52100 steel was the goal of this study. According to achieved results, the optimum temperature and time for dissolution of carbide phase on microstructure of AISI 52100 steel is about 1050 °C and 60 min, respectively. The microstructure of this steel after the austempering process consists of ultrafine bainitic-ferrite plates and retained austenite with two morphologies of micrometer-block and fine film. At the austempering temperature up to 250 °C, the micrometer-blocky morphology of austenite vanished completely and the strength and ductility increased to about 2000 MPa and 7 %, respectively. By increasing

the austempering temperature to 300 °C, the strength and ductility reduced simultaneously to about 1808 MPa and 3 %, as a result of change in the shape and size of bainite phase.

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