Development of Ultrafine Bainitic Structure in AISI 431 Stainless Steel

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Abstract

The development of ultrafine bainitic structure in AISI 431 stainless steel was the goal of this study. For this purpose, the AISI 431 specimens were austenitized at 1100 °C for 60 min followed by low-temperature austempering treatment at different temperatures and times. According to achieved results, the microstructure of AISI 431 steel after austempering treatment consisted of ultrafine bainitic ferrite plates and retained austenite with two morphologies of micrometer-block and fine film. At austempering temperature up to 300 °C, the micrometer-blocky morphology of austenite was vanished completely from the microstructure. The maximum values of strength and ductility of about 1664 MPa and 9.5 % were achieved respectively in the austempered sample at 350 °C for 24 h.

Keywords:Ultrafine bainite; Austempering; AISI 431 steel.

1. Introduction

Bainitic transformation is known as the diffusion less growth of ferrite from austenite just above the martensite start temperature (Ms). Since the early studies about this transformation, it is found that the presence of coarse carbides and blocky retained austenite limits the strength of the microstructure [1)]. A number of attempts have been made to overcome this drawback such as development of ultra-high strength super-bainitic structure [2-4)]. The super-bainitic steels have attracted much attention for their outstanding mechanical properties (ultimate tensile strength and toughness of about 2300 MPa and 30 $MPa.m^{1/2}$, respectively). Their heat treatment includes austenitization followed by a long isothermal transformation stage at a temperature slightly above Ms. The microstructure of these steels composed of nanoscale laths of bainitic ferrite and films of carbon-enriched retained austenite. The size of the sandwiched laths of ferrite/austenite aggregates can go down to 30 nm and provides a combination of strength and ductility in steel [1-5)].

The medium and high carbon steels are most suitable for producing such ultrafine bainitic structures, since higher-carbon content tends to decrease Ms, favoring low-temperature bainitic transformation [6-8)]. In contrast, it is not possible to obtain low-temperature bainitic structure in low-carbon steels via the conventional isothermal transformation above Ms because of their high Ms. According to a literature review it was concluded that the addition of suitable alloying elements such as Cr, Ni, Mo, Nb and Si instead of carbon is one of the most important route to decrease the Ms value of low carbon steels [1)]. In other words, the possibility of producing low-carbon low-temperature bainitic structure can increase by adding the amount of these alloying elements to the composition.

Since Cr, Ni, Mn and Si are benefitial alloying

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elements to decrease the Ms value of steels, AISI 431 martensitic stainless steels with composition of Fe-(1.2-2 wt. %)C- (15-18 wt. %)Cr- (1.3-3 wt. %)Ni- (0-1 wt. %)Mn- (0-1 wt. %)Si can be a good candidate for formation of low-carbon low-temperature bainitic structure. Thus, in the present work, the possibility of producing super-bainitic structure in AISI 431 martensitic stainless steel has been investigated.

2. Experimental Procedure

The AISI 431 steel with a nominal composition of Fe-0.18C-16.1Cr-2.2Ni-0.07Si-0.33Mn-0.014S-0.012P (wt.%) was used as the raw material. For the formation of a bainitic structure, all the alloying elements must be dissolved in Fe matrix. The optimum austenitizing temperature and time for the dissolution of all alloying elements in Fe matrix were chosen to be 1100 °C and 60 min, respectively. The average grain size of austenitized samples at this condition was estimated about 18±4 μm. After austenitizing process, the samples were rapidly transferred to a salt bath furnace. The austempering treatment has been done in temperature range of 250-350 °C for different holding times up to 96 h. The austempered samples were mechanically polished with up to 0.1 μm diamond paste and were etched with Nital etching solution. The polished and etched samples were subjected to a detailed microstructural investigation using a field-emission scanning electron microscope (VEGA-TESCAN-XMU) at an accelerating voltage of 20 kV. An X-ray diffractometer with Cu Ka radiation (λ= 0.15406 nm; 40 kV; Philips PW3710) was used to follow the structural changes of the specimens (2θ range: 20-110°, step size: 0.05°; time per step: 1 s). Room temperature mechanical properties of produced specimens were also evaluated according to ASTM E8M-04 standard using an FM2750 testing machine.

3. Results and Discussion

Austenitizing condition, quenching rate, the properties of salt bath furnace and austempering temperature and times are the main factors which influence the structural and mechanical properties of the austempered specimens [1, 2]. A slight variation in these processing parameters can often cause large variations in the microstructure and properties of produced samples. In the present study, in order to investigate the effects of austempering condition on the structural and mechanical properties of AISI 431 steel, all the aforementioned parameters were kept nominally constant It is worthwhile to note that, the Ms temperature of this steel was calculated at about 230 °C according to the following formula [1)]:

$$M_s=539-423C-30{,}4Mn-12{,}1Cr-17{,}7Ni \qquad \text{Eq. (1)}$$

The typical comparison of the engineering strain-stress curves between austempered samples at 250 °C for different periods of time up to 96 h is shown in Fig. 1. According to this figure, by enhancement the annealing time from 12 to 24 h, the fracture mode has been changed from brittle to ductile and the values of tensile strength and ductility reach about 1534 MPa and 5.9 %, respectively. More increasing in austempering time beyond 24 h has not had significant effects on the mechanical properties. Scanning electron microscopy observations of the tensile fracture surface of austempered sample for 96 h are shown in Fig. 2. As seen, the fracture future of this sample mainly consists of dimples, whereas a large amount of quasi cleavage morphologies and fine microscopic cracks are observed in the region immediately

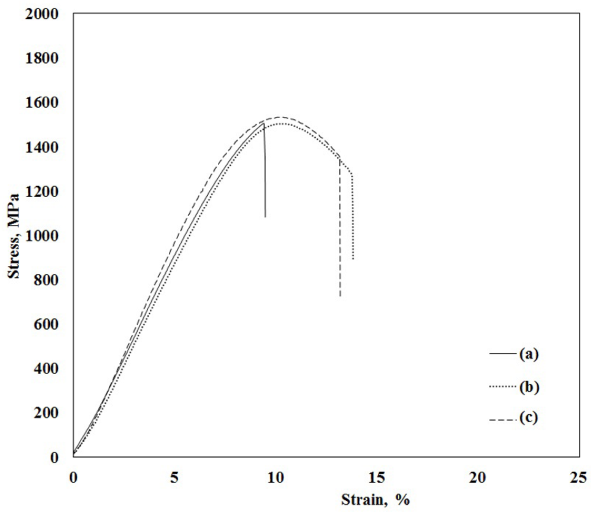


Fig. 1. The engineering stress-strain curves of austempered samples at 250 °C for a) 12, b) 24 and c) 96 h.

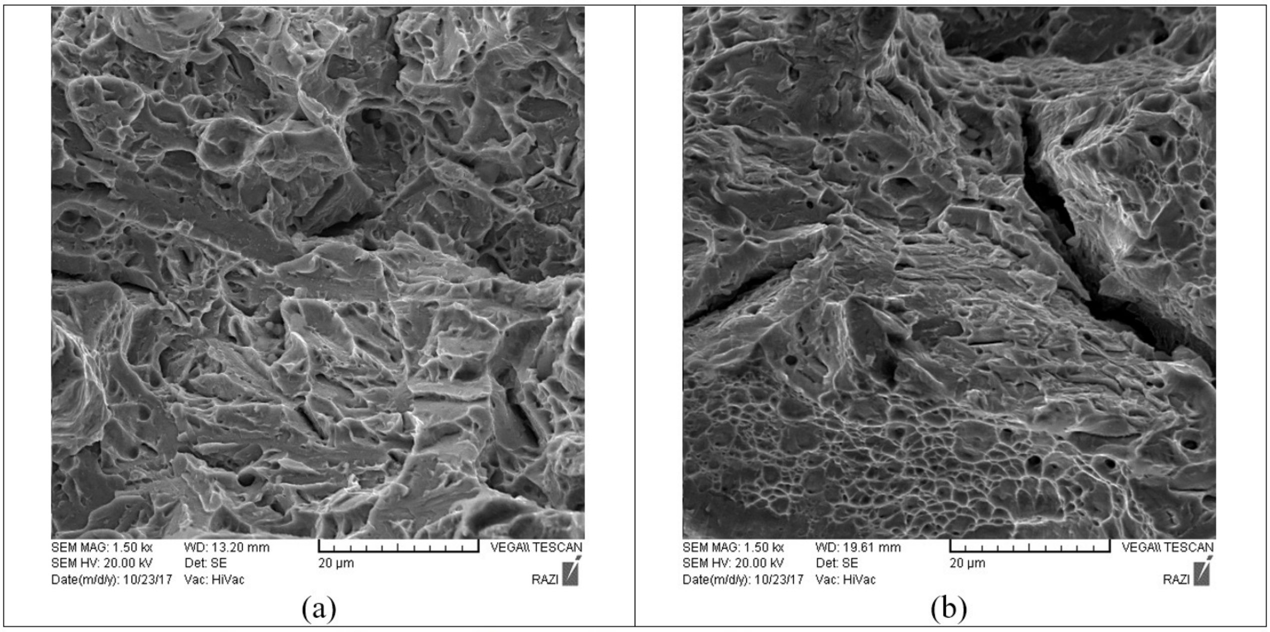
prior to the overload. The nonlinear nature of microscopic cracks intermingled with pockets of dimple-like structure is reminiscent of "locally" brittle fracture mechanism occurring in the region of overload.

Based on presented XRD patterns in Fig. 3, the microstructure of austempered sample at 250 °C for 12 h is mainly martensitic and the percentage of the retained austenite in this sample is close to zero. It can be related to insufficient stability of austenite phase at this condition. By increasing the austempering time, the initial austenite phase (γ) was decomposed into bainitic ferrite (α_{BF}) structure, according to reaction (1) [4].

$$\gamma \rightarrow \alpha_{BF} + \gamma_R \tag{1}$$

As Bhadeshia proposed, the bainitic transformation has two steps [2-4, 8]. Initially, ferrite sheaves form displacively and get constrained by the surrounding parent austenite lattice. Immediately after that, in the second step, carbon atoms are forced back to the parent austenite lattice through a time-consuming diffusive process. By enrichment of retained austenite with carbon in this process, martensitic transformation was suppressed during cooling down to room temperature. Thus the microstructure consists essentially of a mixture of two phases, bainitic-ferrite and carbon enriched regions of retained austenite as shown in Fig. 3.

According to another literature review, the retained austenite in this reaction consists of two morphologies: micrometer-block (γ_{MB}, >1000 nm) between the sheaves of bainitic ferrite plates and nano films (γ_{NF}, <100 nm) between the subunits of bainite [4].



Strain, %

Fig. 2. The SEM fracture surfaces of austempered samples at 250 °C for 96 h in two magnifications.

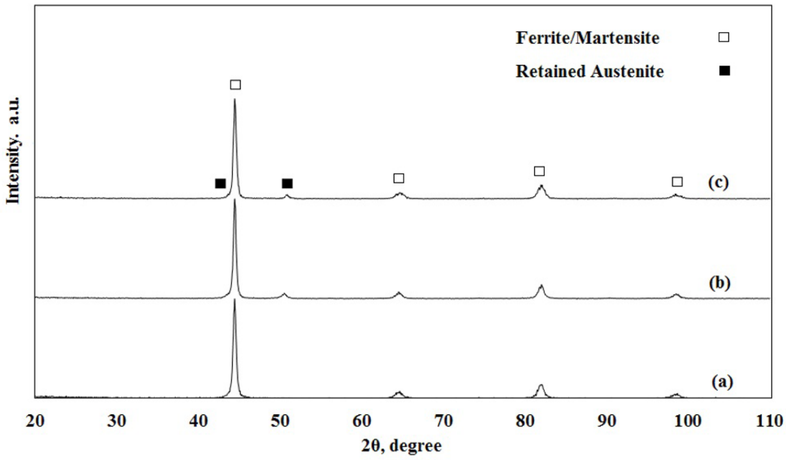


Fig. 3. The XRD patterns of austempered samples at 250 °C for a) 12, b) 48 and c) 96 h.

Fig. 4 illustrates the microstructure of specimens, treated for both 48 and 96 h. At lower magnification, it is possible to observe micro-block retained austenite trapped between sheaves of bainite. Sheaf is a term which refers to groups of bainitic-ferrite plates sharing a common crystallographic orientation and interwoven with thin films of retained austenite, also visible at higher magnifications. In terms of mechanical stabilization, the manner of micrometer-block and nano films retained austenite is so different. The thin film-like retained austenite possesses high mechanical stability because of the size effect and high carbon content within it [9-11]. In contrast, micrometer-block retained austenite with an unstable performance can be transformed into high-carbon un-tempered martensite at the initial stage of deformation [12, 13]. The fresh martensite can act as crack initiation sites, which are detrimental to the deformation behavior. By taking this issue into consideration, the presence of micrometer-block retained austenite in austempered samples at 250 °C and the formation of brittle martensite phase during deformation were the main reason of locally brittle fracture mechanism (Fig. 2) as well as low ductility of prepared

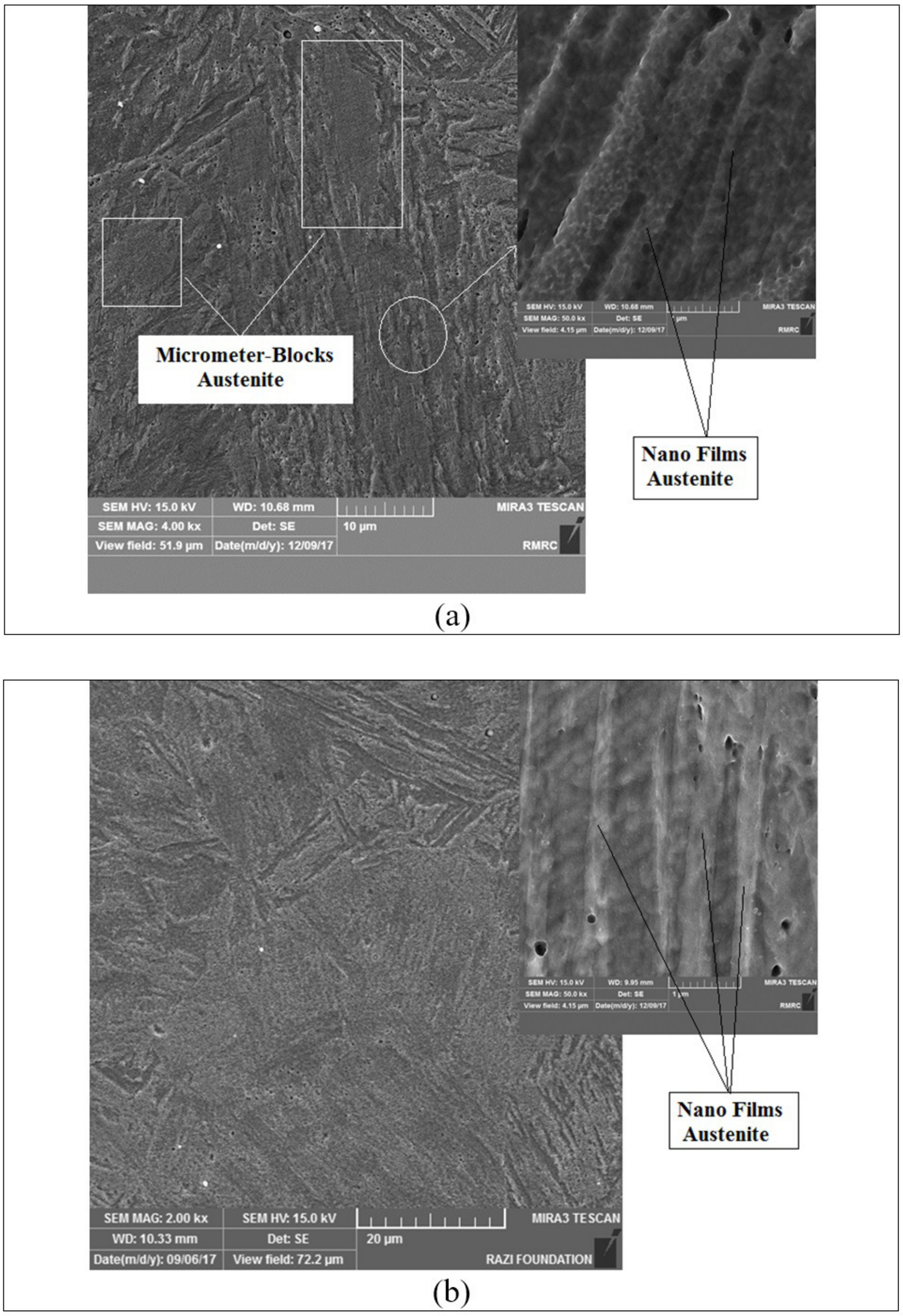


Fig. 4. The cross sectional SEM micrographs of austempered samples at 250 °C for a) 48 and, b) 96 h.

samples at this temperature.

The stress-strain curves of austempered samples at 300 °C for different periods of time up to 48 h are shown in Fig. 5. As seen, overall mechanical characteristics of austempered samples at this temperature are so similar to austempered ones at 250 °C and by enhancement the annealing time from 6 to 12 h, the fracture mode has been changed from brittle to ductile. The maximum values of strength and ductility of austempered samples at this temperature reaches about 1577 MPa and 7.7 % respectively, which are slightly higher than austempered ones at 250 °C. Moreover, more increasing in annealing time has negligible effects on mechanical properties of prepared specimens.

The fracture surface topography of the austempered sample at 300 °C for 48 h is shown in Fig. 6. As seen, same as fracture surface topography of the austempered sample at 250 °C, there is some evidence to suggest the existence of micro cracks in intermingled with pockets of dimple-like structure in the fracture surface future of this sample. The presence of these micro cracks can be attributed to un-reacted retained austenite in the

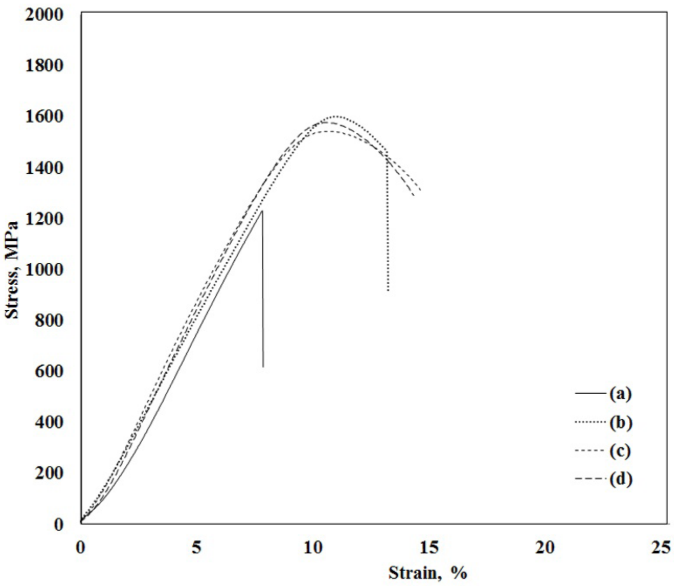


Fig. 5. The engineering stress-strain curves of austempered samples at 300 °C for a) 6, b) 12, c) 24 and d) 48 h.

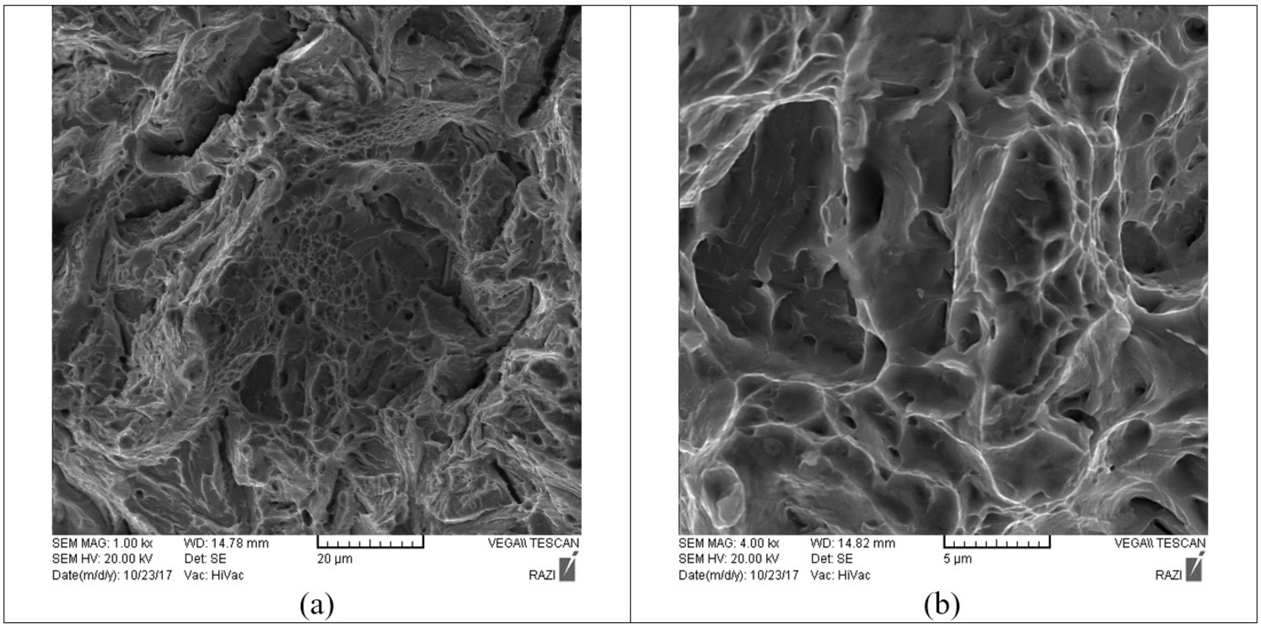


Fig. 6. The SEM fracture surfaces of austempered samples at 300 °C for 48 h in two magnifications.

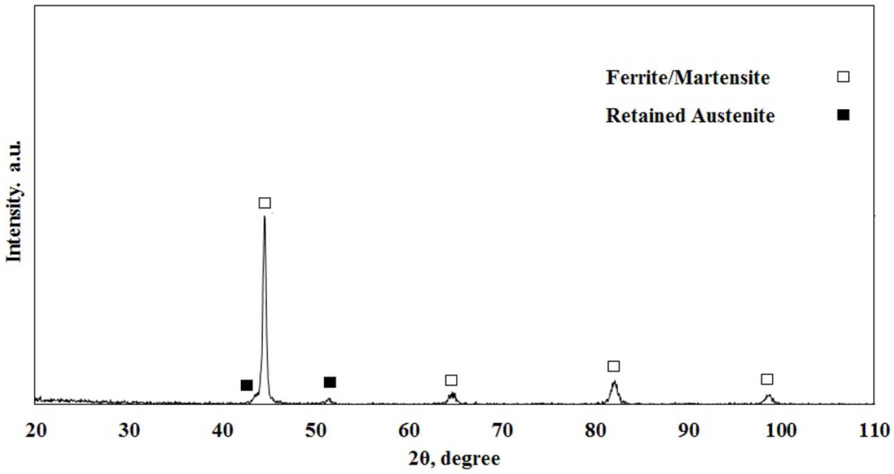


Fig. 7. The XRD pattern of austempered samples at 300 °C for 48 h.

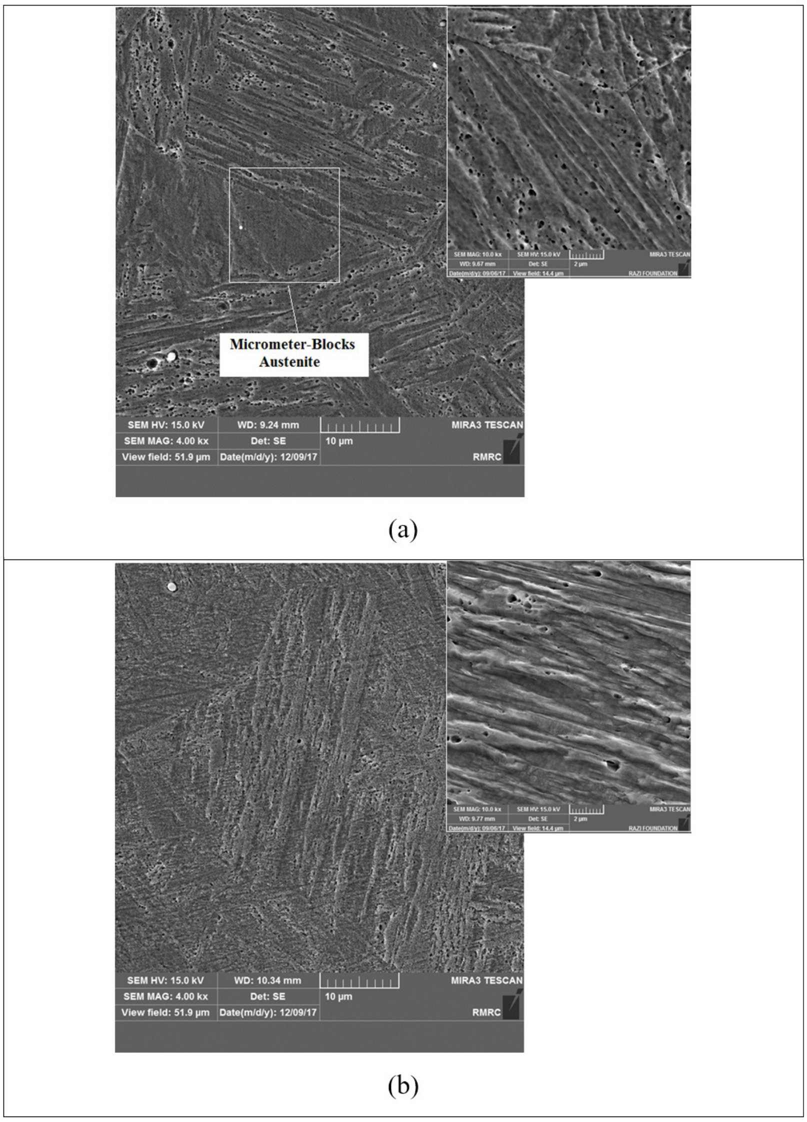


Fig. 8. The cross sectional SEM micrographs of austempered samples at 300 °C for a) 12 and b) 48 h.

microstructure. This result is in agreement with the XRD pattern and SEM micrographs of this specimen which are presented in Figs. 7 and 8, respectively. As mentioned above, during the deformation process, un-reacted retained austenite has turned into high-carbon un-tempered martensite [12, 13]. This phase is so brittle and its formation is the main reason of locally brittle fracture mechanism of this sample.

Same as above, the stress-strain curves of austempered samples at 350 °C for different periods of time are presented in Fig. 9. According to this figure, in this austempering temperature, the transition from brittle to ductile fracture mode occurs in shorter holding times than lower temperatures. As seen, the maximum values of strength and ductility in this annealing temperature achieved after 24 h of holding time and reached 1664 MPa and 9.5%, respectively. In this regard, the XRD pattern and SEM cross-sectional micrographs of the austempered sample at 350 °C for 24 h are shown in Figs. 10 and 11, respectively. According to these figures, the structure of this sample only consists of bainitic-ferrite phase and there is no evidence of micrometer-block

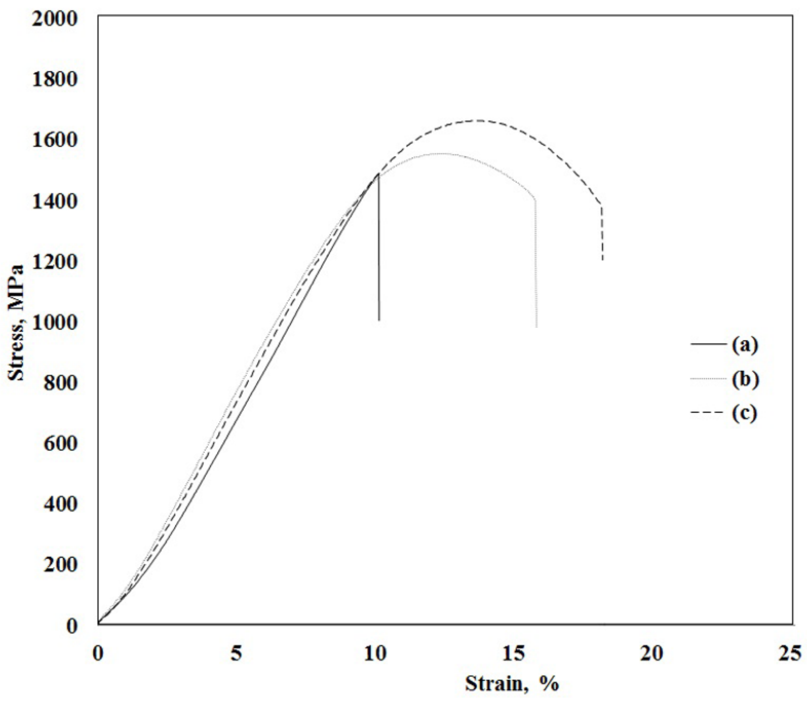


Fig. 9. The engineering stress-strain curves of austempered samples at 350 °C for a) 3, b) 12 and, c) 24 h.

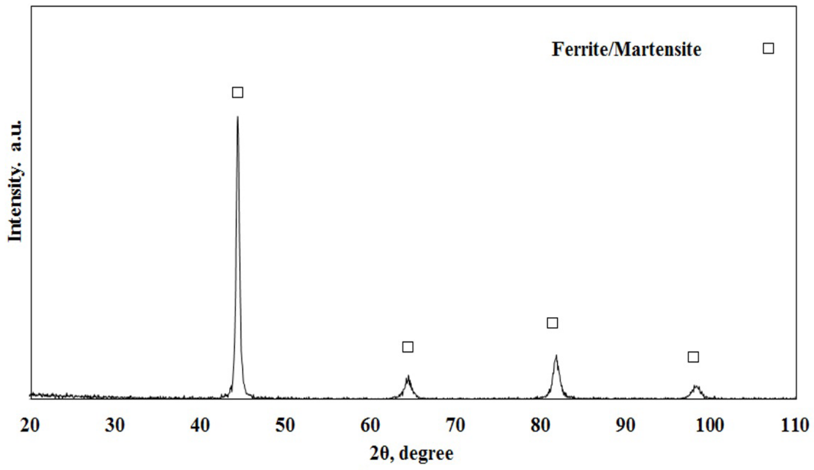


Fig. 10. The XRD pattern of austempered samples at 350 °C for 24 h.

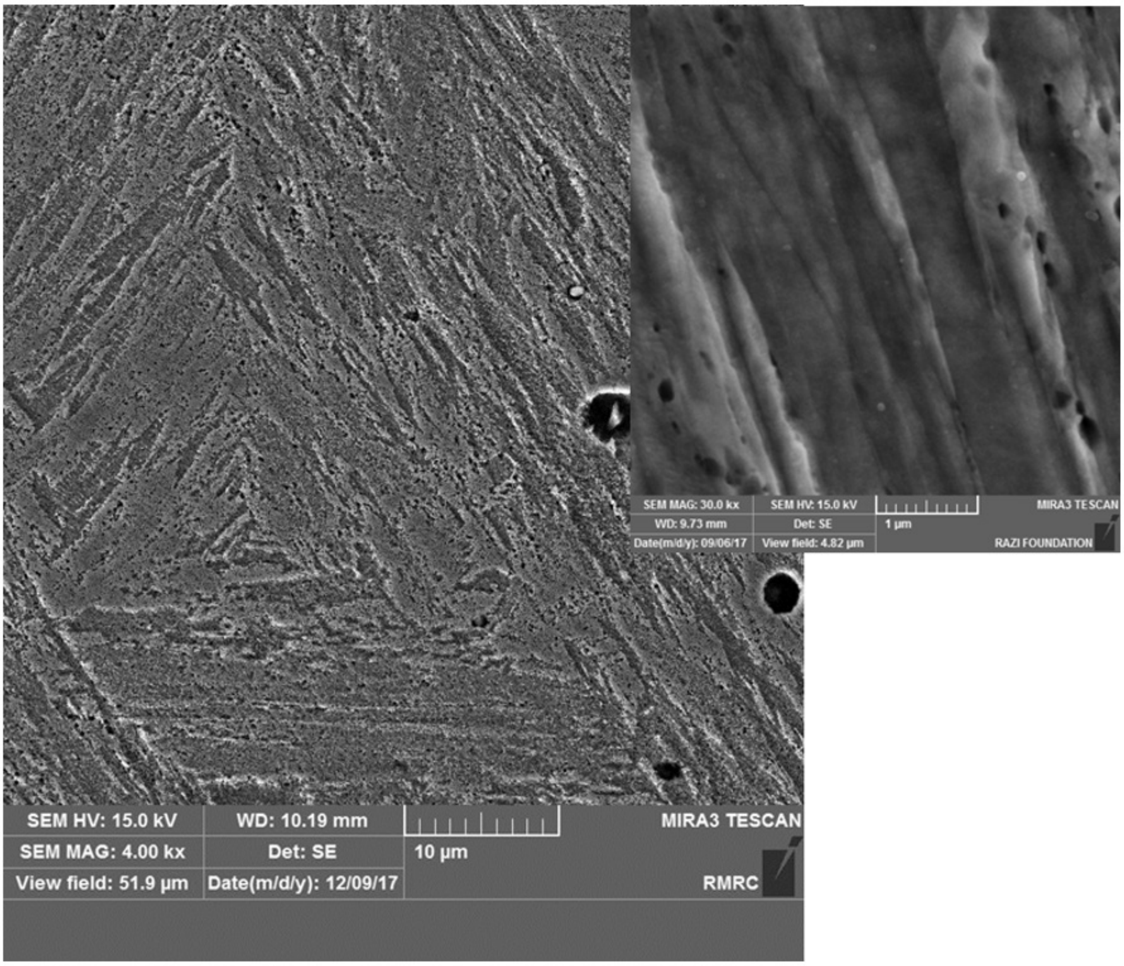
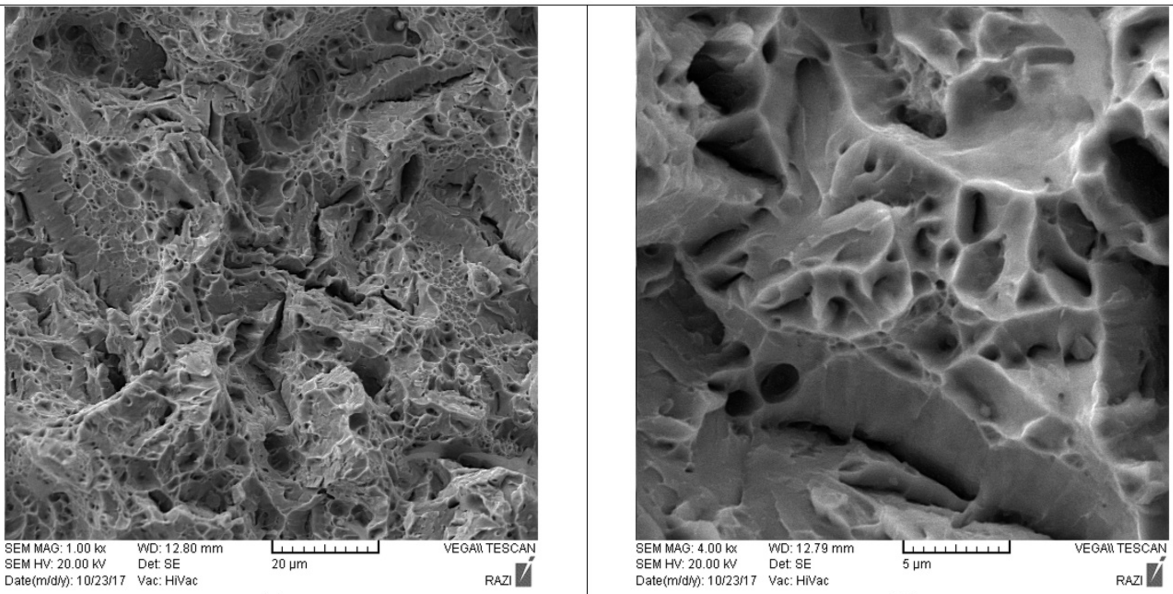


Fig. 11. The cross sectional SEM micrographs of austempered samples at 350 °C for 24 h in two magnifications.



(a) (b)

Fig. 12. The SEM fracture surfaces of austempered samples at 350 °C for 24 h in two magnifications.

retained-austenite and cementite phases in the microstructure. This means that any possible amount of retained-austenite and cementite phases lie in the critical characterization limit of the XRD machine and bainitic transformation has been completed at this annealing condition. The presented fracture surface morphology of this sample in Fig. 12, which has dimple-like structure without any cracks, also confirms this phenomenon.

However, based on presented results, the maximum value of strength which can be achieved in AISI 431 steel during austempering treatment is about 1664 MPa. This value of strength is so higher than the values that can be achieved in AISI 431 during common quench and tempering treatment (about 1100 MPa) [14]. In fact, the higher value of strength in austempered samples can be explained by:

1. Hall-Patch effect: According to the Hall-Patch equation ($\sigma = k/d^{-0.5}$, 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 austempered samples can be related to the formation of thin plates of bainitic-ferrite (about 110 nm) in the microstructure [5].
2. Increasing in dislocation density: According to the Bailey-Hirsch equation, the yield strength of steel increases with increasing dislocation density. The bainitic transformation is mainly dominated by displacive mechanism [2]. The plastic relaxation accompanied with the shape deformation during bainite transformation lead to local residual stress and increasing in dislocation density [15]. Thus, the bainitic-ferrite steels present a high density of dislocations that consequently leads to high yield strength.
3. Concentration of carbon atoms around dislocations: Based on previous works [8-10], 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 they reduce the mobility of them [15].

Moreover, the value of ductility of austempered samples in optimum condition is so high (about 9.5 %) and comparable with the ductility value which can be achieved during common quench and tempering treatment (11.5 %) [14]. It is important to note that, the presence of nano-film retained-austenite between bainitic-ferrite plates is the main reason of high ductility of these samples. Lattice resistance in the face-centered cubic (fcc) structure is lower than that of the body-centered cubic structure; in addition, dislocation motion is notably facilitated in the former structure, thus increases the plasticity. Moreover, 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 [8].

4. Conclusions

In the present work, the development of ultrafine bainitic structure in AISI 431 stainless steel was investigated. According to achieved results, the microstructure of AISI 431 steel after austempering treatment consists of ultrafine bainitic ferrite plates and retained austenite with two morphologies of a micrometer-block and fine film. At austempering temperature up to 300 °C, the bainitic transformation was completed and the micrometer-block morphology of austenite vanished completely from the microstructure. The maximum values of strength and ductility of about 1664 MPa and 9.5 % were achieved in austempered sample at 350 °C for 24 h, respectively.

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