

Effect of tempering temperature on microstructure and work hardening behavior of a triple-phase AISI 4140 steel

A. Talebi*¹, R. Bakhtiari², B. Abbasi Khazaei³, M. Ghobeiti-Hasab⁴

¹ Faculty of Materials & Manufacturing Technologies, Malek Ashtar University of Technology, Tehran, Iran

^{2,3} Department of Materials and Textile Engineering, Faculty of Engineering, Razi University, Kermanshah, Iran

⁴ Materials and Energy Research Center, Dezful Branch, Islamic Azad University, Dezful, Iran

Abstract

In this research, AISI 4140 steel samples were normalized at 850 °C for 60 min, then, they were kept at 720 °C for 3 min and were directly transferred to a salt bath with a temperature of 400 °C for 4 min and after that, the samples were quenched in water (25 °C). Finally, the triple-phased samples were tempered at 250, 450, and 650 °C for 90 min. The microstructure and tensile properties were examined by an FE-SEM equipped with an EDS detector and a universal tensile test machine, respectively. Results showed that the microstructure of sample tempered at 250 °C was upper bainite and martensite along with carbide particles resulting from the decomposition of martensite, while the microstructure of sample tempered at 450 °C was lower bainite and martensite along with carbide particles. Nevertheless, in the sample tempered at 650 °C, bainite and martensite phases were decomposed completely to carbide. In comparison with the not-tempered sample, the sample tempered at 250 °C had higher yield strength, greater elongation, and lower ultimate tensile strength. As the tempering temperature increased from 250 to 650 °C, the yield strength and ultimate tensile strength decreased while the elongation increased. Tensile test results of the tempered samples showed a two-stage work-hardening behavior. In both stages, the strength coefficient (k) decreased with increasing the tempering temperature. The work-hardening exponent (n) in the first stage decreased only for the samples tempered at higher than 250 °C, while in the second stage, it continuously decreased for all tempered samples.

Keywords: Triple-phase steel; Tempering temperature; Microstructure; Work-hardening.

1. Introduction

Triple-phase steels contain a soft matrix (ferrite) and higher strength phases (bainite and martensite)

exhibit properties such as good strength, high work-hardening capacity, and continuous yielding behavior ^{1, 2}). The mechanical properties of these steels depend on microstructure, which can be controlled by the tempering temperature ³). Improving the mechanical properties during tempering is due to modified microstructure including precipitation of carbides in ferrite, bainite, and martensite ⁴).

Anazadeh and Kheirandish ⁵) were reported with tempering of dual-phase steel at temperatures higher than 200 °C decrease the yield strength and ultimate tensile strength. Furthermore, they observed the continuous yielding behavior of the steel at tempering temperatures higher than 300 °C. Pouranvari ⁶) found that by increasing

*Corresponding author

Email: amir.talebi1368@gmail.com

Address: Faculty of Materials & Manufacturing Technologies, Malek Ashtar university of Technology, Tehran, Iran.

O. BOX: Tel.: +98 916 938 7901.

1-PHD student

2- Associate Professor

3- Associate Professor

4- Assistant Professor

the martensite volume fraction in dual-phase steel, the work-hardening exponent and strength coefficient increased. Moreover, at martensite volume fractions lower than 50%, work-hardening occurred at one stage, while at volume fractions higher than 50%, two-stage work-hardening was observed. Akbarpour and Ekrami ⁷⁾ stated that for ferrite-bainite dual-phase steels, work-hardening occurred at two stages and Hollomon equation parameters increased with increasing the tempering temperature at both work-hardening stages. Zare and Ekrami ⁸⁾ found that as the martensite volume fraction in dual and triple-phase steels increased, the Hollomon equation parameters increased. In addition, they found that the difference between ultimate tensile strength and yield strength increased with increasing the martensite volume fraction.

In the present research, the microstructure, mechanical properties and work-hardening behavior of a triple-phase (Ferrite-Bainite-Martensite) 4140 steel in different tempering temperatures have been investigated.

2. Experimental

A low-alloyed medium-carbon steel bar with diameters of 20 mm was used as the initial material. Table 1 shows the chemical composition of the initial steel, which was measured by a spectrometric analyzer. As can be seen, the initial steel is a low alloy steel containing elements of chromium, molybdenum, and manganese, with a composition consistent with AISI 4140 steel. According to Fig. 1, the microstructure of the initial steel is composed of ferrite and pearlite phases.

According to the T-T-T diagram of AISI 4140 steel (Fig. 2) ⁹⁾, first, steel bars were normalized at 850 °C for 60 min with a heating rate of 100 °C/min. Then, the samples continuously were kept at 720 °C for 3 min and were directly transferred to a salt bath with a temperature of 400 °C. The holding time at this temperature was 4 min and after that, the samples were quenched in water (25 °C). In all steps, the heat treatment was done continuously.

Table 1. Chemical composition of initial steel bar measured by a spectrometric analyzer.

Element	Fe	C	Cr	Mn	Mo	Si	P	S
Alloy analysis wt.%	Balance	0.416	1.1	0.75	0.221	0.342	0.015	0.021

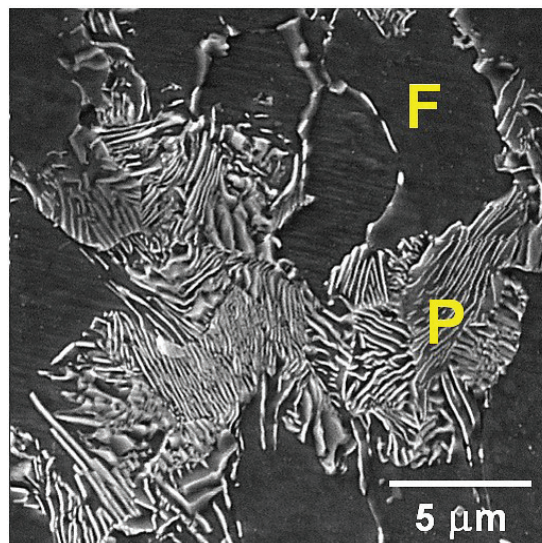


Fig. 1. Microstructure of the initial steel (F: ferrite, P: pearlite).

Finally, the triple-phased samples were tempered at different temperatures of 250, 450, and 650 °C for 90 min. After tempering at various temperatures, the samples were held in a furnace for 24 hours to cool the equilibrium condition.

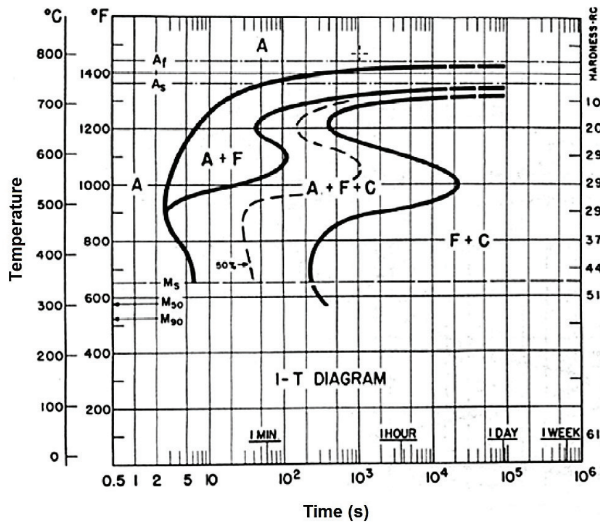


Fig. 2. T-T-T diagram of AISI 4140 steel ⁹⁾.

A field emission scanning electron microscope (FE-SEM, model: Mira 3-XMU) was used to investigate the microstructure (2% Nital etchant - secondary electrons detector). The ferrite volume fraction was measured using an Olympus image analyzer. Tensile test samples were prepared according to ASTM E8/E8M standard and analyzed using a universal tensile test machine at a crosshead speed of 2 mm/min. The tensile testing machine with a capacity of 20 tons had been made by Gotech Taiwan Company.

The Microhardness test was ASTM E384 standard made by the American company of Buehler.

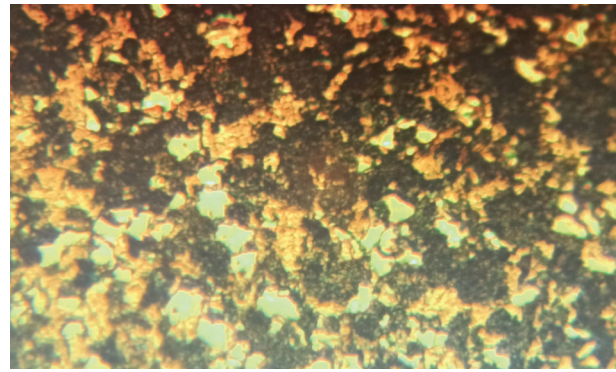
3. Results and discussion

3.1. Microstructure before tempering

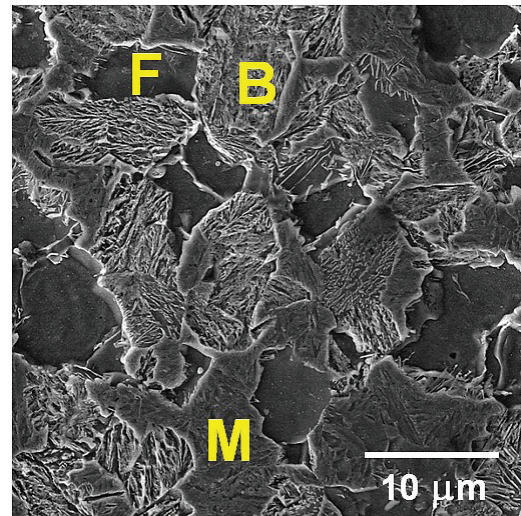
Fig. 3 shows an FE-SEM photograph of the triple-phase microstructure before any tempering treatment. As can be seen the phases of ferrite, bainite and martensite exist distinctly. The ferrite volume fraction was estimated at 33.4%.

A Micro hardness test was performed on the sample with a triple-phase structure. The purpose of the Microhardness test was to ensure the formation of bainite and martensite phases. Micro hardness Vickers results showed that hardness of black areas into layer structure in Figure 3a was 513HV and yellow areas were 341 HV.

Vickers Micro hardness results show that black areas have martensite hardness and yellow areas have Bainite hardness ¹⁾.



(a)



(b)

Fig. 3. a) Optical microscope image with 400x magnification b) FE-SEM photograph of the sample before tempering (F: ferrite, B: bainite, M: martensite).

3.2. Microstructure after tempering

Previous research reported 90 min as the optimum tempering time for the AISI 4140 steel ¹⁰⁻¹⁴⁾. Furthermore, tempering temperatures lower than 200 °C did not affect the mechanical properties ¹⁵⁾. Therefore, in the present research, the triple-phase samples were tempered at 250, 450, and 650 °C for 90 min.

Fig. 4a-c shows FE-SEM photographs of triple-phase microstructure tempered at 250, 450, and 650 °C, respectively. In Fig. 4a, lath-like morphology of upper bainite is seen, while, martensite has been decomposed as fine particles that EDS analysis indicated this particle are carbide (due to detection of C and Cr elements). In Fig. 4b, needle-like morphology of lower bainite and decomposed martensite are observed. As can be seen, the bainite phase is stable even at 450 °C. According to Fig. 4c, bainite and martensite phases are decomposed completely, thus, higher contents of carbides are observed compared to other tempering temperatures. With increasing the tempering temperature, the precipitation of carbides increases ¹⁶⁾.

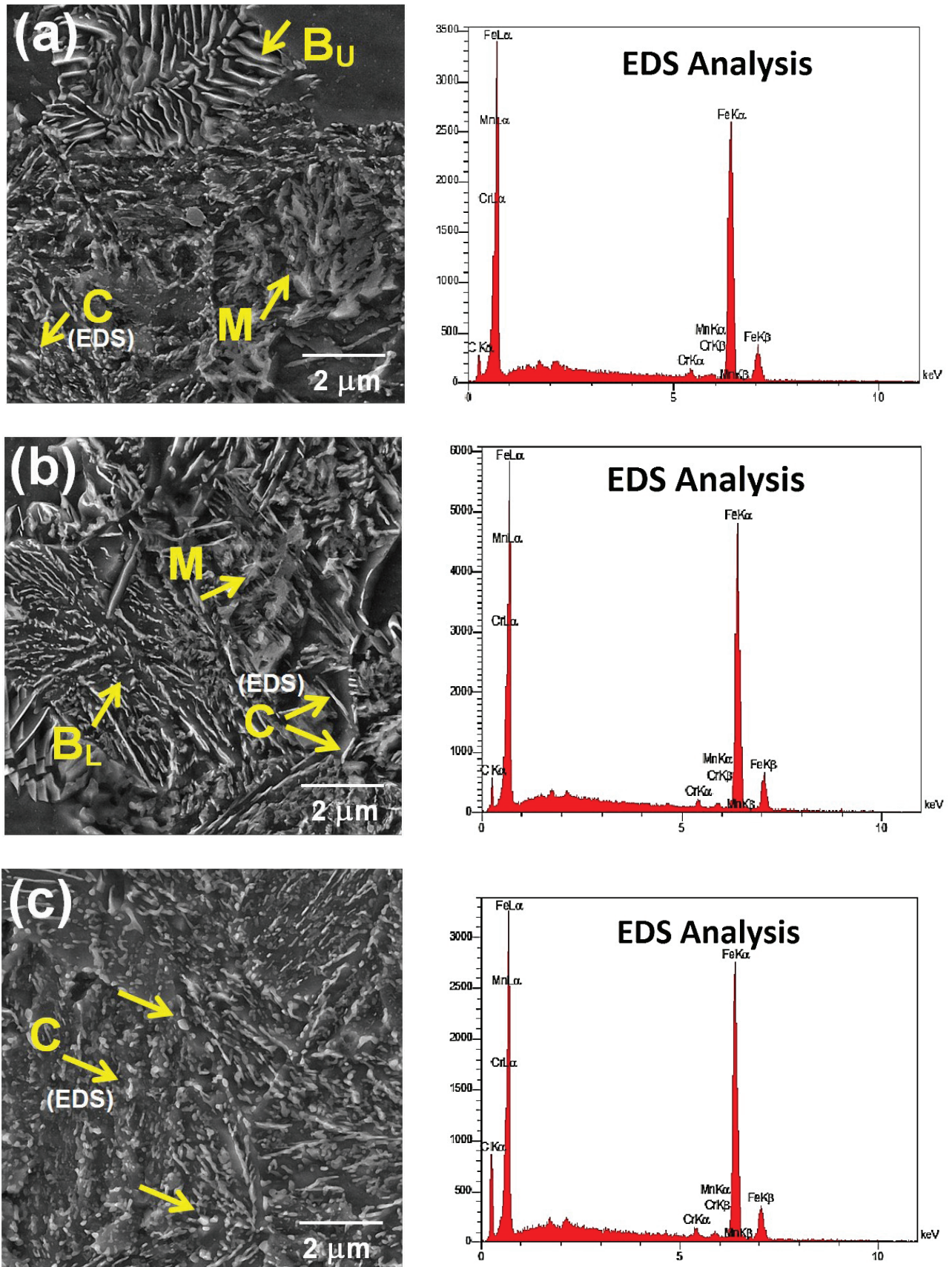


Fig. 4. FE-SEM photograph of the samples tempered at (a) 250 °C, (b) 450 °C, (c) 650 °C (BU: upper bainite, BL: lower bainite, M: martensite, C: carbide).

3.3. Tensile properties

Fig. 5 shows the stress-strain curve obtained from tensile tests of the samples tempered at various temperatures and the not-tempered sample. Continuous yielding behavior is seen in all curves. Values of yield strength, ultimate tensile strength, and elongation are given in Table 2. Accordingly, the yield strength of the sample tempered at 250 °C in comparison with the not-tempered sample increases from 972 to 1036 MPa. This is due to the precipitation of the fine carbides¹⁷⁾. With increasing the tempering temperature up to 650 °C, the yield strength decreases to 447 MPa, the ultimate tensile strength decreases from 1377 to 825 MPa, and the elongation increases from 7.2 to 19.3%.

According to Figure 5 (b), the reason for higher

amounts of mechanical properties of the sample tempered at 250 °C can be due to the conversion of retained austenite to martensite during temper treatment¹⁾.

3.4. Work-hardening

Work-hardening behavior of dual and triple-phase steels can be explained based on the general equation of Hollomon as the following:

$$\sigma = k \varepsilon^n \quad \text{Eq. (1)}$$

where k and n are constants named as strength coefficient and work-hardening exponent, respectively. This relation can be written as follows:

$$\log \sigma = n \log \varepsilon + \log k \quad \text{Eq. (2)}$$

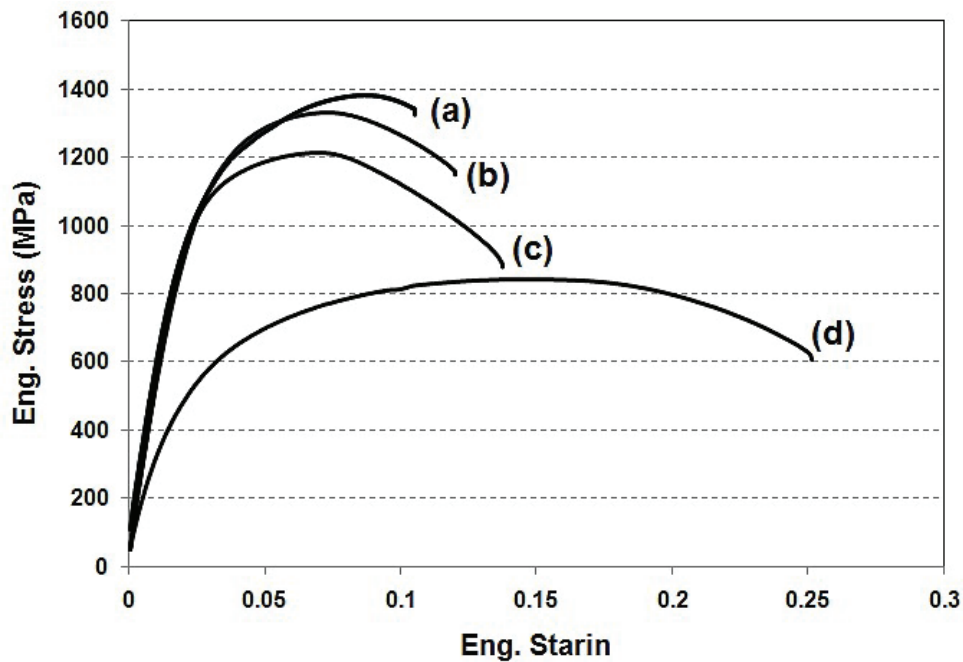


Fig. 5. Engineering stress-strain curve of the samples (a) not-tempered, and tempered at (b) 250 °C, (c) 450 °C, (d) 650 °C.

Table 2. Tensile test results for the samples not-tempered and tempered at various tempering temperatures.

Tempering temperature (°C)	YS (offset - 0.2%) (MPa)	UTS (MPa)	Elongation (%)
Not- Tempered	972	1377	7.2
250	1036	1349	9.6
450	880	1151	12.2
650	447	825	19.3

The $\text{Ln}\sigma\text{-Ln}\epsilon$ curve of all samples has been given in Fig. 6. The plots are non-linear and have been best fitted as two linear zones, which indicate two-stage work-hardening behavior. Two-stage work-hardening behavior has been reported for dual-phase steels with a volume fraction of hard phases higher than 50%¹⁸⁾. The presence of different stages of work-hardening can be related to various mechanisms of work-hardening. The first stage of work-hardening is due to plastic deformation of ferrite, while the second stage is attributed to simultaneous plastic deformation of ferrite, bainite, and martensite. The plastic deformation of martensite initiates in the second stage of work-hardening, due to the higher strength of martensite compared to ferrite and bainite¹⁹⁾.

The values of the work-hardening exponent and the strength coefficient in both stages are given in Table 3. In the first stage, the work-hardening exponent of the sample tempered at 250 °C increased slightly and then decreased with increasing the tempering temperature. While in the second stage the work-hardening exponent decreased continuously with increasing the tempering temperature. A similar trend was also observed for the values of the strength coefficient. During the tempering treatment, carbon exits from the supersaturated structure of martensite and precipitates as carbides. Decreasing the volume fraction of hard phases leads to a decrease in the work-hardening rate. The results showed that the work-hardening exponent has a direct relation with yield and tensile strengths.

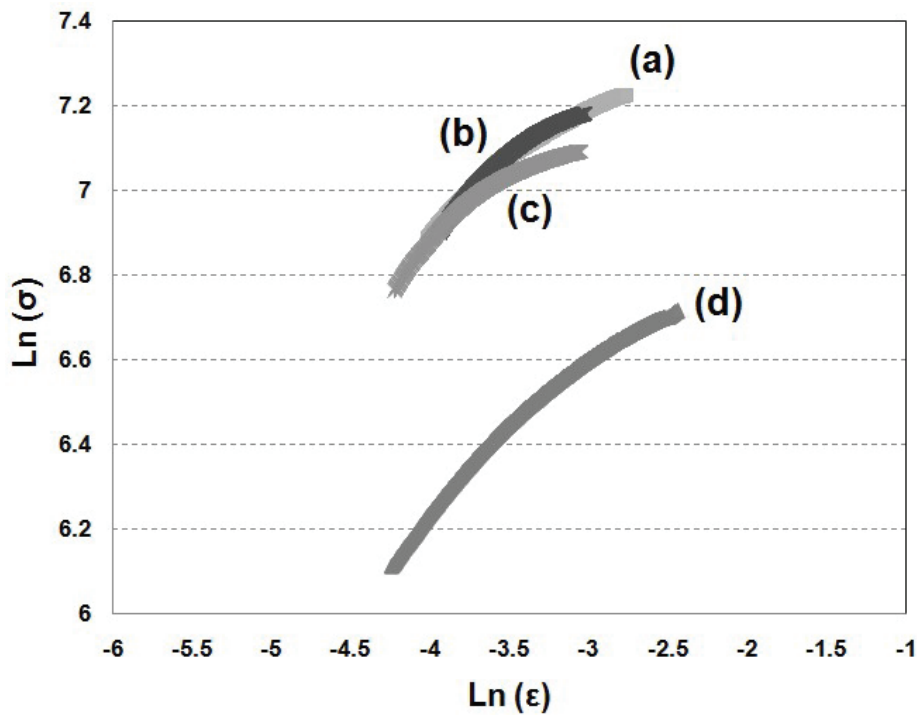


Fig. 6. $\text{Ln}\sigma\text{-Ln}\epsilon$ curve of the samples (a) not-tempered, (b) tempered at 250 °C, (c) 450 °C, (d) 650 °C.

Table 3. The values of the work-hardening exponent (n) and the strength coefficient (k).

Tempering temperature (°C)	The first stage of work-hardening		The second stage of work-hardening	
	n_1	n_2	k_1	k_2
Not- Tempered	0.3826	0.2099	8424	7814
250	0.3841	0.1685	8427	7692
450	0.3625	0.1451	8281	7474
650	0.3362	0.1089	7784	7230

Conclusion

In this research, AISI 4140 steel with triple-phase microstructure (ferrite-bainite-martensite) was tempered at 250, 450, and 650 °C. The results of microstructural studies and tensile tests indicated that:

- During tempering due to the precipitation of carbides, the strength (YS, UTS) decreased and the elongation increased.
- Continuous yielding behavior was observed at engineering stress-strain curves.
- Two-stage work-hardening behavior was observed for tempered samples.
- The work-hardening exponent and strength coefficient decreased with increasing the tempering temperature.

Reference

- [1]. R. Bakhtiari, A. Ekrami: *Mater. Sci. Eng. A*, 525 (2009) 159.
- [2]. A. Zare, A. Ekrami: *Mater. Sci. Eng. A*, 530 (2011) 440.
- [3]. B. London, D.V. Nelson, J.C. Shyned: *Metall. Trans. A*, 20 (1989) 1257.
- [4]. A. Kampa, S. Celottob, D.N. Hanlonb: *Mater. Sci. Eng. A*, 538 (2012) 35.
- [5]. A. Anazadeh Sayed, Sh. Kheirandish: *Mater. Sci. Eng. A*, 532 (2012) 21.
- [6]. M. Pouranvari: *BHM Berg*, 157 (2012) 44.
- [7]. M. Akbarpour, A. Ekrami: *Mater. Sci. Eng. A*, 477 (2008) 306.
- [8]. A. Zare, A. Ekrami: *Mater. Sci. Eng. A*, 528 (2011), 4422.
- [9]. M. Samler: *Jominy End Quenching of 4140 Steel: The effect of time and temperature on austenitic grain growth*, Worcester Polytechnic Institute, Bachelor thesis, (2010), 8.
- [10]. A. Mandal, T.K. Bandyopadhyay: *Mater. Sci. Eng. A*, 620 (2015) 463.
- [11]. X. Tao, Ch. Li, L. Han, J. Gu: *J. Mater. Res. Technol.*, 5 (2016) 45.
- [12]. G. Chakraborty, C.R. Das, S.K. Albert, A.K. Bhaduri, V. Thomas Paul, G. Panneerselvam, A. Dasgupta: *Mater. Charact.*, 100 (2015) 81.
- [13]. H.S. Hasan, M.J. Peet, M-N. Avettand-Fènoël, H.K.D.H. Bhadeshia: *Mater. Sci. Eng. A*, 615 (2014) 340.
- [14]. N. Saeidi, A. Ekrami: *Mater. Sci. Eng. A*, 527 (2010) 5575.
- [15]. K. Davut, C. Hakan: *9th European Conf. on NDT*, Germany, Berlin, (2006), 180.
- [16]. J. Akre, F. Danoix, H. Leitner, P. Auger: *Ultramicroscopy*, 109 (2009) 518.
- [17]. X. Fang, Z. Fan, B. Ralph, P. Evans, R. Underhill: *J. Mater. Process. Technol.*, 132 (2003) 215.
- [18]. P. Movahed, S. Kolahgar, S.P.H. Marashi, M. Pouranvari, N. Parvin: *Mater. Sci. Eng. A*, 518 (2009) 1.
- [19]. M.R. Akbarpour, A. Ekrami: *Mater. Sci. Eng. A*, 477 (2008) 306.