

Effect of Cooling Medium and Tempering on Microstructures and Hardness of SK3 High Carbon Steel

Open
Access

Ridhwan Jumaidin^{1,2,*}, Iffa Marina Jopri², Rafidah Hasan^{2,3}, Muhammad Ilman Hakimi Chua Abdullah^{1,2}, Mohamed Saiful Firdaus Hussin², Alia Ruzana Aziz⁴, Abdul Rafeq Saleman^{2,3}, Febrian Idral^{2,3}

- ¹ Fakulti Teknologi Kejuruteraan Mekanikal dan Pembuatan, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia
² Centre for Advanced Research on Energy, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia
³ Fakulti Kejuruteraan Mekanikal, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia
⁴ Department of Aerospace Engineering, Khalifa University of Science and Technology (KUST), 127788 Abu Dhabi, United Arab Emirates

ARTICLE INFO

ABSTRACT

Article history:

Received 16 April 2019
Received in revised form 4 September 2019
Accepted 2 October 2019
Available online 30 November 2019

This paper presents a study on the effect of heat treatments i. e annealing, quenching, normalizing, and tempering on the characteristics of SK3 high carbon steel. The specimens were heated to austenitizing temperatures at 760°C, 840°C, 920°C, 1000°C and 1080°C, before cooled by using different cooling medium i.e. oil quenching, air cooling, and furnace cooling. Tempering process was conducted on the quenched specimen by reheating at 500°C and cooled to room temperature. Material characterization techniques such as hardness test and morphological investigation were carried out for all specimens. The heat treatment has significantly improved the hardness of the material. The quenched specimen (rapid cooling) shows the highest increment of hardness by 709%. This was followed by the normalized specimen (moderate cooling) and annealed specimen (slow cooling) which shows 425% and 170% of increment respectively. However, tempering treatment on the quenched specimen has shown a slight decrease in the hardness value. In terms of microstructure, the annealed specimen produced coarse grain size while the quenched specimen produced a fine grain size. The hardness of the specimen subjected to tempering process is lower than that of quenching process.

Keywords:

High carbon steel; heat treatment; annealing; quenching; tempering

Copyright © 2019 PENERBIT AKADEMIA BARU - All rights reserved

1. Introduction

High carbon steel is a common material that can be found for applications which require high strength, high hardness, and good wear resistance. In addition, this material is also known to be cost-effective, making them as a promising material for the high abrasive environment [1]. The industrial applications of high carbon steel include cutting tools, cable, music wire, springs, and cutlery. The carbon content of the steel is the vital criteria for the hardness and strength of this material. Higher

* Corresponding author.

E-mail address: ridhwan@utem.edu.my (Ridhwan Jumaidin)

carbon content was often associated with higher hardness and strength of the heat-treated material. Basically, high carbon steel is classified based on its carbon content which is more than 0.6 wt.% and it is categorized as heat treatable materials.

Heat treatment can be defined as a combination of heating and cooling processes which is applied to a metal or alloy in solid state condition [2, 3]. It is an important manufacturing process of carbon steel which will determine the performance of the final product [4, 5]. Apart from steel, heat treatment were also applied to other materials such as aluminium alloy, sodium niobate, and natural fiber composites [6, 7] By applying the heating process, the microstructure and mechanical properties of the material can be altered, and most of the energy is consume [8]. Mainly, heat treatment was used to increase the mechanical properties of the steel in order to meet the desired manufacturing specification of the material [9]. This properties i.e strength and hardness are highly dependent on the microstructure of the material [4, 10–12].

Although there are many studies conducted on carbon steel, however, the study conducted on heat treatment of high carbon steel are very rare. Hence, this study is intended to investigate the effect of cooling medium and tempering on the microstructure and hardness of SK3 high carbon steel.

2. Materials and Methodology

2.1 Specimen Preparation

SK3 high carbon steel with a carbon content of more than 0.6wt.% was used in this study. The specimens were cut to a diameter of 16 ± 0.5 mm and thickness of 5 ± 0.5 mm. The measured hardness of the as-received specimen is 6.96 HRC.

2.2 Material Composition

Composition analysis of the as-received specimen was performed by using Energy Dispersive X-Ray (EDX) spectroscopy.

2.3 Hardness Test

Hardness tests were conducted on the specimens by using Rockwell Hardness Tester according to ASTM E18-17e1. The load of 1471N was used for all specimens. The hardness scale and type of indenter used are HRC and diamond 120° respectively. This procedure was repeated for five different points on the specimen in order to get an average value of hardness number.

2.4 Metallurgical Investigation

This process was conducted to investigate the changes in the microstructure of the specimens following the heat treatment. The direct microscopic observation was conducted by using the optical microscope in order to analyze the phase present and their proportions, as well as the grain size. The procedure of the metallurgical investigation consists of mounting, grinding, polishing, etching, cleaning, and finally observing using a microscope.

2.5 Heat Treatment

There are four types of heat treatments used in this study i.e quenching, normalizing, annealing, and tempering. The specimens were heated to specific austenitizing temperatures of 760°C , 840°C ,

920°C, 1000°C and 1080°C in an electric furnace. Then, the specimen was held at austenitizing temperature for 1 hour of soaking time. Consequently, the specimens were taken out from the electric furnace and cooled in three different cooling medium which i.e oil quenching (quenching), air cooling (normalizing) and furnace cooling (annealing). In tempering process, the quenched specimen was reheated at 500°C for 10 min and air cooled. Table 1 shows the heat treatment parameter for SK3 high carbon steel.

Table 1
Soaking temperature and cooling medium

Soaking Temperature	Cooling medium			Tempering (500°C)
	Oil quenching	Air cooling	Furnace cooling	
760°C	OQ1	AC1	FC1	T1
840°C	OQ2	AC2	FC2	T2
920°C	OQ3	AC3	FC3	T3
1000°C	OQ4	AC4	FC4	T4
1080°C	OQ5	AC5	FC5	T5

3. Results and Discussion

3.1 Composition Analysis

Chemical composition analysis was carried out for the as-received specimens in order to identify the percentage of carbon and other elements in the material. The result of the chemical composition analysis showed the percentage of carbon is in the range of 1 – 1.10 wt.%. Since the amount of carbon is more than 0.6wt.%, hence it can be confirmed that the specimen is a high carbon steel. Table 2 lists the chemical composition of the material used in this study.

Table 2
Chemical composition of SK3 carbon steel

Element	wt.%
C	1.00-1.10
Si	0.15-0.35
Mn	0.15-0.50
P	0.03
S	0.03

3.2 Effect of Cooling Medium on Hardness

Figure 1 shows the effect of different cooling medium and temperature on the hardness value of SK3 high carbon steel. In general, all heat treatment process has effectively increased the hardness value of the as-received specimen. Highest hardness value which is 56.34 HRC was shown by specimen OQ2 while lowest hardness which is 4.28 HRC was shown by specimen FC1 in Table 3.

Table 3
Hardness value of various heat treatment processes (HRC)

Process/ Heating temperature	Hardness value (HRC)				
	760°C	840°C	920°C	1000°C	1080°C
Annealing	4.28	12.68	18.8	18.2	8.98
Oil quenching	42.04	56.34	54.88	48.9	30.2
Normalizing	22.62	30.62	36.54	26.8	26.38
Tempering	38.3	52.92	50.72	41.84	30.32

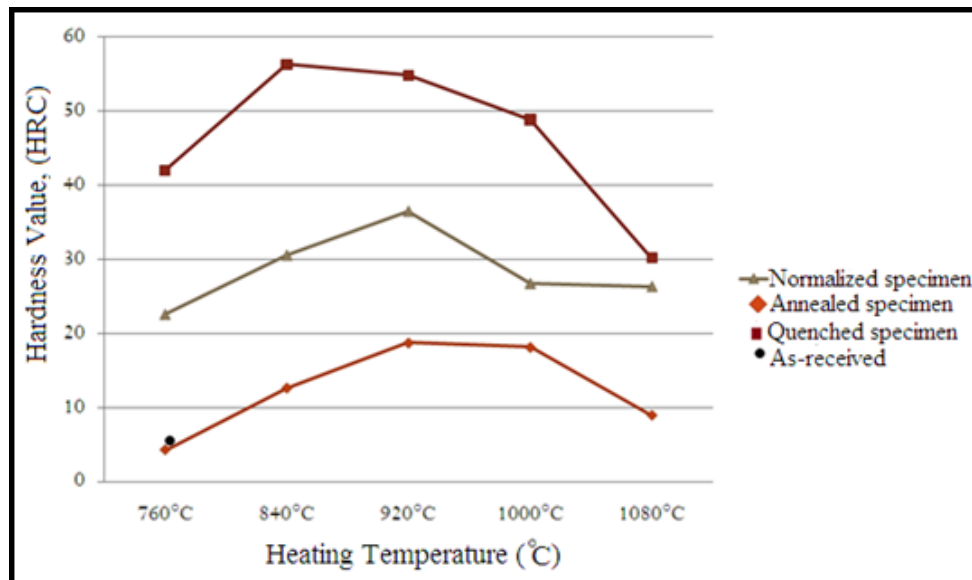


Fig. 1. Hardness values of annealing, normalizing and quenching of SK3 high carbon steel

Generally, quenched specimen shows the highest hardness followed by the normalized specimen and annealed specimen respectively. The quenched specimen (rapid cooling) shows the highest increment of hardness by 709%. This was followed by the normalized specimen (moderate cooling) and annealed specimen (slow cooling) which shows 425% and 170% of increment respectively. Both annealed and normalized specimen showed an increase in the hardness value following the increase in soaking temperature up to 920°C. The drop in hardness value was observed at 1000°C. For quenched specimen, the hardness increased as the temperature increased up to 840°C and started to decreased at 920°C. In general, all treatments conducted shows increment in the hardness value of the high carbon steel. This improvement might be attributed to the microstructure transformation upon subjected to heat treatment which will be further discussed in the next section.

The higher hardness of quenched specimen might be attributed to the formation of fine martensite resulting from fast cooling rate [4]. Meanwhile, the lower hardness of annealed specimen can be attributed to the formation of soft pearlite microstructure resulting from the slow cooling rate. In general, as the cooling rate increases, the hardness of material also increases. This finding is in agreement with the previous study [2]. The decrease in hardness value for the annealed specimen might be attributed to the formation of fine pearlite and proeutectoid ferrite. The annealed specimen at a temperature of 1080°C shows the transformation of bainite microstructure containing cementite and α ferrite. For the normalized specimen, the decreased of hardness value at 1000°C was due to the coarsening of grain. Generally, increase in soaking temperature had coarsened the grain of the material. This finding is in agreement with the previous study [5].

The decreased in the hardness value at soaking temperature of 920°C and 1000°C might be associated to the effect of coarse grain size. The decreased in hardness for the quenched specimen at a temperature of 1080°C, might be attributed to the transformation to bainite microconstituent containing ferrite, cementite and martensite microstructure. In terms of mechanical properties, bainite microstructures have a lower hardness than martensite which decreases the hardness value. This phenomenon can be attributed to the increasing of grain size due to a higher rate of diffusion at a higher temperature which further decreases the strength of the material by permitting more dislocation motion to occur as compared to finer grain. In addition, the formation of micro-crack is one of the reasons for hardness drop that being discovered in this paper which will be further elaborated in the microstructure analysis section.

3.3 Effect of Cooling Medium on Microstructure

Figure 2 shows the microstructure of as-received specimen contains ferrite crystals filled with sphere-like cementite particle. This microstructure was referred as spheroidite. The existence of cementite particle in high carbon steel microstructure is consistent with the previous study [13].

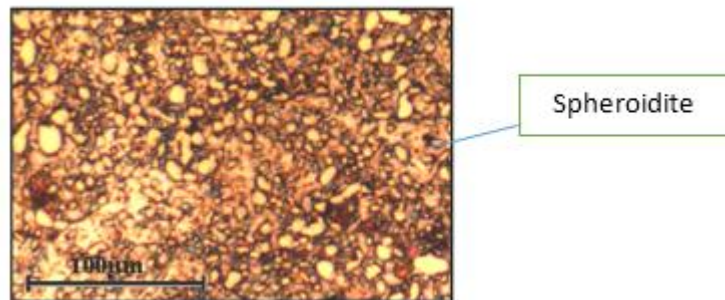


Fig. 2. As- received specimen

3.3.1 Annealed specimen

Figure 3 shows the microstructure of annealed specimen for various soaking temperature. As indicated in Figure 3(a), specimen FC1 showed similar spheroidite phase as the as-received material. Meanwhile, Figure 3(b) shows the microstructure of specimen FC2 contains smaller cementite particles.

In annealing, the microstructure transformations involved the decomposition of austenite and slow cooling process form coarse pearlite microstructure. During slow furnace cooling, the carbon precipitates out in the form of pro-eutectoid cementite network at the prior austenite grain boundary. Thereafter prior austenite grains eutectoidally transform to pearlite [14]. Figure 3(c) shows larger grain size of specimen FC3 following higher soaking temperature of 920°C. In addition, according to Al-Qawabah *et al.*, [15], a carbon steel subjected to annealing will experience grain coarsening due to slower cooling rate. Furthermore, Figure 3(c) and (d) showed that specimen FC4 and FC5 formed fine pearlite and proeutectoid ferrite. The layers of pearlite were formed due to the diffusion of carbon atoms with minimal distances for the formation of the structure.

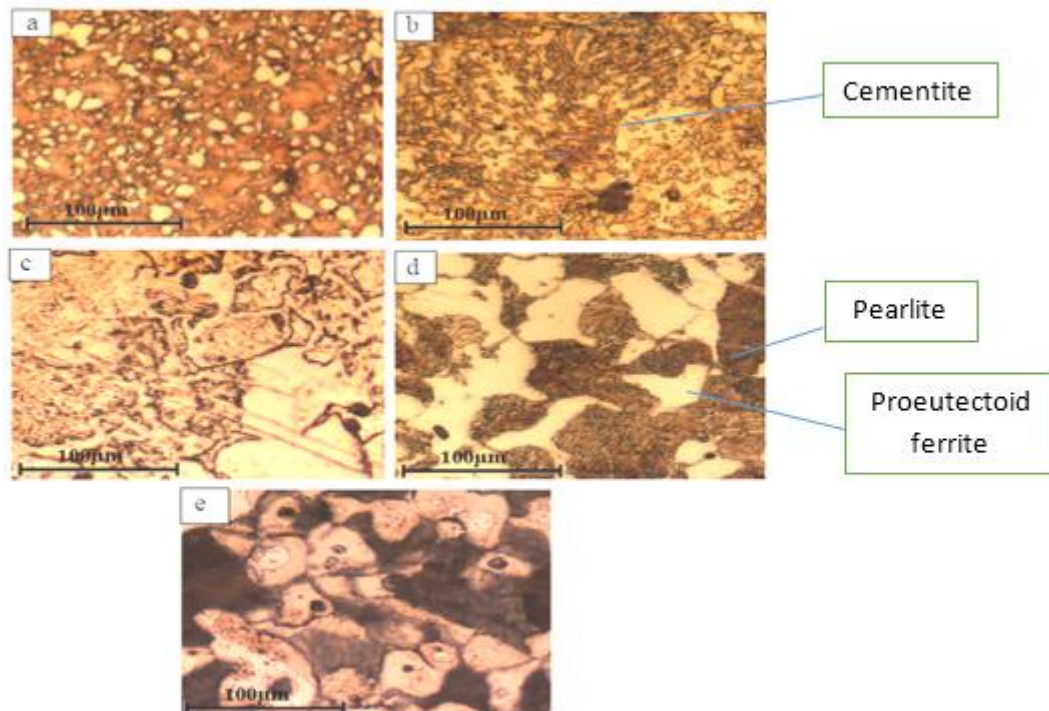


Fig. 3. Annealed specimen microstructure at various heating temperature (a) 760°C (b) 840°C (c) 920°C (d) 1000°C and (e) 1080°C

3.3.2 Normalized specimen

The microstructure of specimen AC1 was shown in Figure 4(a). The specimen shows bainite microstructure containing very fine and elongated particles of cementite Fe C in α ferrite matrix. By increasing the austenitizing temperature to 840°C in the normalizing process, the particles of cementite Fe C was dissolved in specimen AC2 as shown in Figure 4(b). It was found that martensite microstructure exists in the normalized specimen of high carbon steel SK3 with a heating temperature of 920°C, and this has contributed to the highest hardness value shown in Table 3 which is 36.54HRC. Figure 4(c) shows that the martensite plate size increased with the increasing of austenitizing temperature.

Figure 4(d) and 4(e) also show martensite micro-constituent for the normalized specimen. It is observed that the grain sizes for specimen AC3 and AC4 were coarser than AC1 and AC2. This coarser grain has contributed to lower hardness value as shown in Table 3. According to Saha *et al.*, [14], during the subsequent non-equilibrium forced air cooling, the austenite regions transform to ferrite-cementite aggregate. It is important to note that in this case cementite being partially dissolved in austenite which also contributes to lower hardness value than quenched specimens.

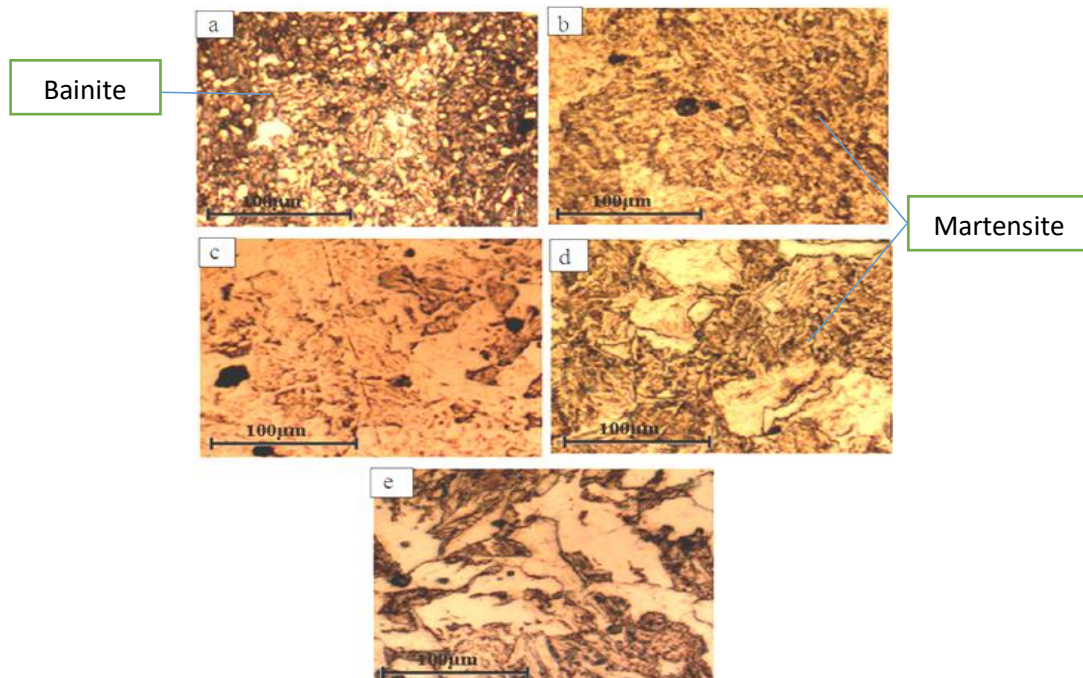


Fig. 4. Normalized specimen microstructure at various heating temperature (a) 760°C (b) 840°C (c) 920°C (d)1000°C and (e) 1080°C

3.3.3 Quenched specimen

Specimen OQ1 shows microconstituent martensite phase as shown in Figure 5(a). Martensite phase formed when austenitized iron-carbon alloys rapidly cooled by oil quench in the vicinity of the ambient low temperature. Here, quench rate was rapid enough to prevent carbon diffusion. As shown in Figure 5(a), the white region is austenite that failed to transform during quenching process.

Specimens OQ2, OQ3, and OQ4 show similar martensite phase as shown in Figure 5(b), (c) and (d). Austenite is slightly denser than martensite and therefore, during the phase transformation upon quenching, there is a net volume increase. Consequently, relatively large pieces of austenite undergone rapid quenched were cracked and appeared as black pores due to internal stresses as shown in Figure 5(b) and 5(c). It is observed that specimen OQ4 has coarser grain which explains the drop in hardness value to 48.9 HRC. Increase in hardness after quenching can be attributed to delay in the formation of ferrite which then promotes the formation of pearlite and martensite at the higher cooling rate. Thus, the increased of hardness with quenched steels can be explained by the increasing relative value of pearlite and martensite after quenching [16].

Figure 5(e) shows the microstructure of specimen OQ5. Increase in the soaking temperature up to 1080°C had coarsened the cementite particles, which explains the tremendous drop in the hardness.

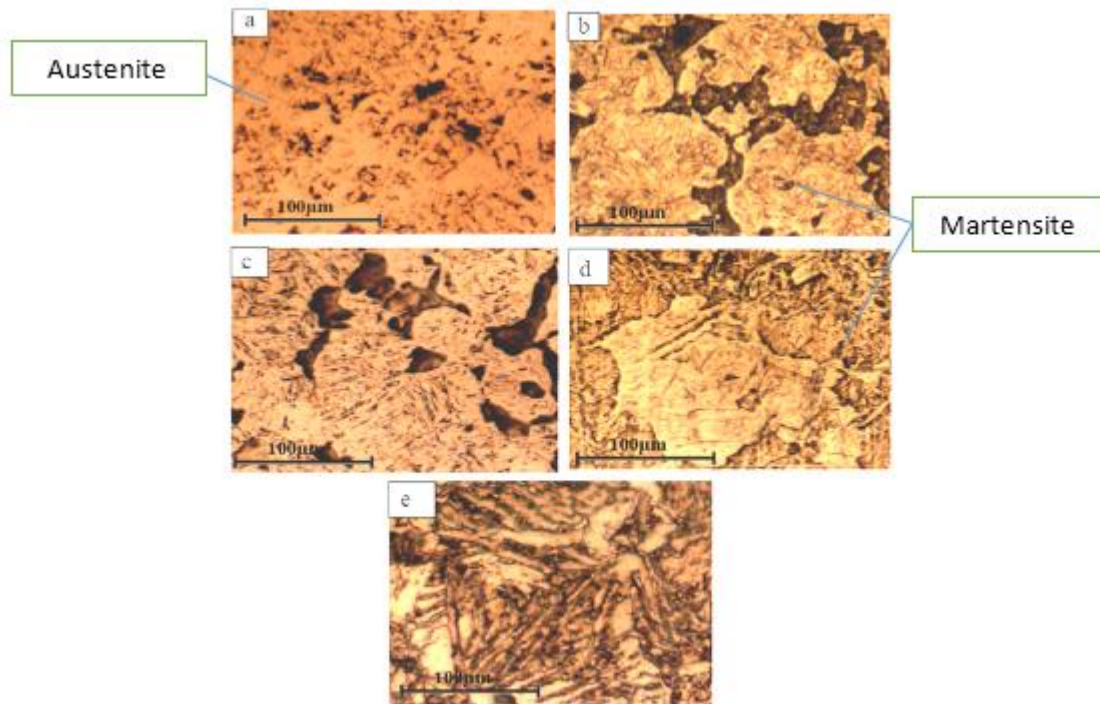


Fig. 5. Quenched specimen microstructure at various heating temperature (a) 760°C (b) 840°C (c) 920°C (d)1000°C and (e) 1080°C

3.4 Effect of Tempering

Figure 6 shows the hardness of tempered specimen is lower than the quenched specimen. This is consistent with finding by Tkalec *et al.*, [17]. The decreased in hardness value was due to the microstructural change accompanying tempering which is a loss of acicular martensite pattern and the precipitation of tiny carbide particles. This microstructure is referred known as tempered martensite. According to Edmonds *et al.*, [18] tempering causes the retained austenite content to rapidly decreases due to transformations of retained austenite to martensite constituent. The martensite hardness also continuously decreases as carbon precipitates, showing that it is mainly influenced by carbon in solid solution [17].

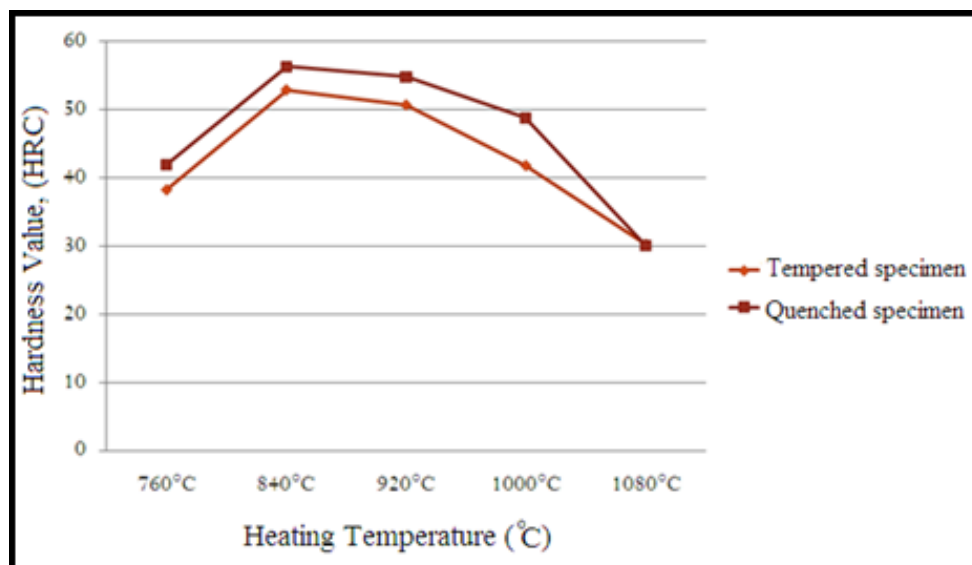


Fig. 6. Hardness values of tempered and quenched specimen

Furthermore, other factors that cause a drop in hardness of tempered specimen was discussed by Suchanek *et al.*, [19] that tempering relived the internal stress and cause carbon to diffused from martensite carbide. On the other hand, the prevalent martensite is an unstable structure and the carbon atoms diffuse from martensite to form tempered martensite and results in concurrent formation of ferrite and cementite. This process allows microstructure transformation which reduces the hardness to the desired level while increasing the ductility [20].

Figure 7 shows similar tempered martensite microstructure which contains small and uniformly dispersed cementite particles embedded within a continuous ferrite matrix. However, the increase in cementite particle size can be observed as the soaking temperature increases from 840°C until 1080°C.

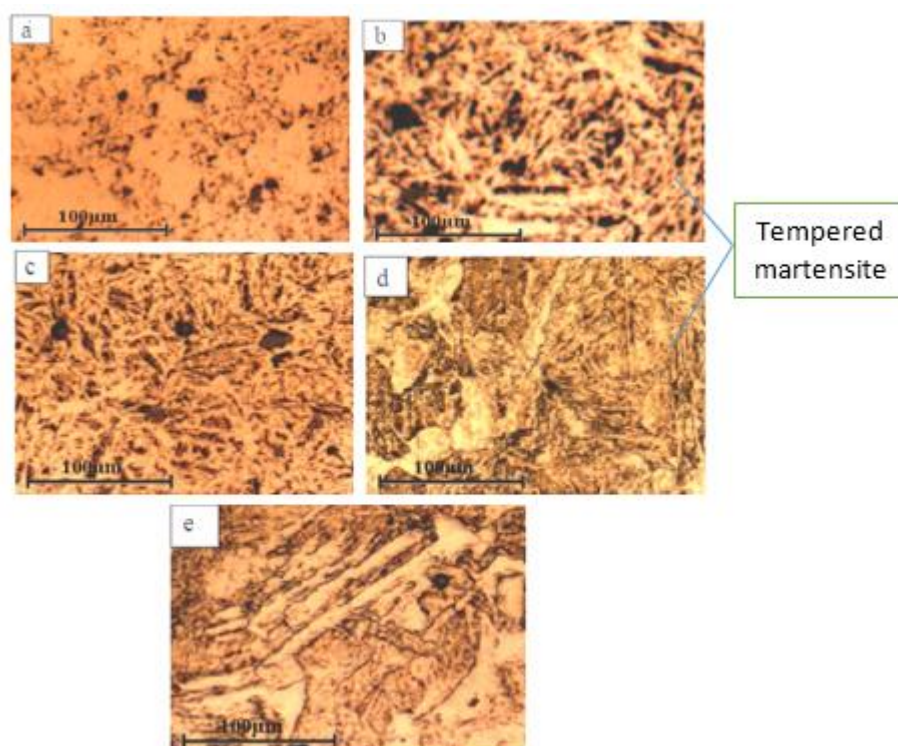


Fig. 7. Tempered specimen microstructure at various heating temperature (a) 760°C (b) 840°C (c) 920°C (d) 1000°C and (e) 1080°C

Microstructures of the quenched specimen on Figure 4 shows martensite phase. After tempering at 500°C, supersaturated carbon in martensite microconstituent transform to the tempered martensite which decrease the hardness value as shown in Table 3. The size of cementite particles does influence the hardness of tempered specimen where increase in its size will decrease the ferrite-cementite phase boundary area as shown in Figure 7(d) and (e) and thus, decrease the hardness as shown in Figure 6. Overall, the modification of high carbon steel in this study has improved the potential application of this material especially for the heavy industries which requires high strength material [21].

4. Conclusions

Analysis of the microstructure and hardness of heat treated SK3 high carbon steel has shown that the hardness value tends to increase following the increase in the cooling rate. Rapid cooling of the quenched specimen recorded the highest hardness of 56.34HRC at 840°C soaking temperature with

the formation of fine martensite. This finding has increased 709% of the as-received hardness. The moderate cooling rate on normalized specimen shows intermediate hardness value whereas slow cooling rate on annealed specimen shows the lowest hardness of 4.28HRC at 760°C soaking temperature with the formation of pearlite and proeutectoid ferrite. The slow cooling rate on annealed specimen produced a coarse grain of pearlite while rapid cooling of the quenched specimen produced a fine grain of martensite. The hardness of tempered specimen was slightly dropped as compared to the quenched specimen due to phase transformation from martensite to tempered martensite.

Acknowledgments

The authors would like to thank Universiti Teknikal Malaysia Melaka for the financial support provided through Journal Publication Incentive Grant (project number JURNAL/2018/FTK/Q00004).

References

- [1] Hossain, Rumana, Farshid Pahlevani, and Veena Sahajwalla. "Effect of small addition of Cr on stability of retained austenite in high carbon steel." *Materials Characterization* 125 (2017): 114-122.
- [2] Ridhwan, J., E. Hamzah, M. Z. Selamat, Z. Zulfattah, and M. H. M. Hafidzal. "Effect of aging treatment on the microstructures and hardness of Fe-Ni-Cr superalloy." *International Journal of Automotive & Mechanical Engineering* 8, (2013): 1430-1441.
- [3] Ridhwan, J., M. H. M. Hafidzal, M. S. Zakaria, and M. A. M. Daud. "Effect of cooling rate on microstructures and mechanical properties of C102 copper alloy." *Journal of Mechanical Engineering and Technology (JMETS)* 6, no. 1 (2014): 87-94.
- [4] Rahman, S. M., Kazi Ehsanul Karim, and MD Hasan Shahriar Simanto. "Effect of heat treatment on low carbon steel: an experimental investigation." In *Applied Mechanics and Materials*, vol. 860, pp. 7-12. Trans Tech Publications, 2017.
- [5] Ridhwan, J., J. A. Noor, M. S. Zakaria, Z. Zulfattah, and M. H. M. Hafidzal. "Effect of heat treatment on microstructure and mechanical properties of 6061 aluminum alloy." *Journal of Engineering and Technology (JET)* 5, no. 1 (2014): 89-98.
- [6] Akmal, MH Maziati, M. W. A. Rashid, U. A. A. Azlan, and N. A. Azmi. "The effects of different annealing temperature and number of deposition layers on the crystallographic properties of potassium sodium niobate (KNN) thin films synthesized by sol-gel spin coating technique." *Journal of Advanced Manufacturing Technology (JAMT)* 11, no. 1 (2017): 91-102.
- [7] Edynoor, O., A. R. M. Warikh, T. Moriga, K. Murai, E. Mohammad, and M. R. Salleh. "Effect of annealing time on resistivity of kenaf fiber modified indium zinc oxide prepared via dip coating process." *Journal of Advanced Manufacturing Technology (JAMT)* 11, no. 1 (1) (2017): 139-150.
- [8] Ridhwan, J., E. Hamzah, H. Effendy, M. Z. Selamat, and Z. Zulfattah. "Effect of cooling rate on the microstructures and hardness of Fe-Ni-Cr superalloy." *Journal of Mechanical Engineering and Technology (JMETS)* 5, no. 1 (2013): 45-57.
- [9] Ridhwan, J., M. Syafiq, R. Hasan, and Z. M. Zulfattah. "Effect of aging on the microstructures and mechanical properties of C102 copper alloy." *Journal of Engineering and Technology (JET)* 4, no. 2 (2013): 115-124.
- [10] Salleh, Mohd Shukor, Nurul Naili Mohamad Ishak, Mohd Azizul Hikmi Safian, and Saifudin Hafiz Yahaya. "Microstructural analysis and mechanical properties of LM6 alloy processed by cooling slope casting." *Journal of Advanced Manufacturing Technology (JAMT)* 10, no. 2 (2016): 109-124.
- [11] Ishak, N. N. M., M. S. Salleh, S. H. Yahaya, E. Mohamad, and M. A. Sulaiman. "The effect of equal channel angular pressing (ECAP) on the microstructure and hardness of A356 aluminium alloy." *Journal of Advanced Manufacturing Technology (JAMT)* 11, no. 2 (2017): 47-58.
- [12] Peng, Hanlin, Ling Hu, Liejun Li, Liyun Zhang, and Xianglin Zhang. "Evolution of the microstructure and mechanical properties of powder metallurgical high-speed steel S390 after heat treatment." *Journal of Alloys and Compounds* 740 (2018): 766-773.
- [13] Lv, Z. Q., Bo Wang, Z. H. Wang, S. H. Sun, and W. T. Fu. "Effect of cyclic heat treatments on spheroidizing behavior of cementite in high carbon steel." *Materials Science and Engineering: A* 574 (2013): 143-148.
- [14] Saha, Atanu, Dipak Kumar Mondal, Koushik Biswas, and Joydeep Maity. "Microstructural modifications and changes in mechanical properties during cyclic heat treatment of 0.16% carbon steel." *Materials Science and Engineering: A* 534 (2012): 465-475.

-
- [15] Al-Qawabah, S. M. A., Nabeel Alshabatat, and U. F. Al-Qawabeha. "Effect of annealing temperature on the microstructure, microhardness, mechanical behavior and impact toughness of low carbon steel, grade 45." *International Journal of Engineering Research and Applications* 2, no. 3 (2012): 1550-1553.
- [16] Güral, A., B. Bostan, and A. T. Özdemir. "Heat treatment in two phase region and its effect on microstructure and mechanical strength after welding of a low carbon steel." *Materials & Design* 28, no. 3 (2007): 897-903.
- [17] Tkalcec, I., C. Azcoitia, S. Crevoiserat, and D. Mari. "Tempering effects on a martensitic high carbon steel." *Materials Science and Engineering: A* 387-389, (2004): 352-356.
- [18] Edmonds, D. V., K. He, F. C. Rizzo, B. C. De Cooman, D. K. Matlock, and J. G. Speer. "Quenching and partitioning martensite—a novel steel heat treatment." *Materials Science and Engineering: A* 438-440, (2006): 25-34.
- [19] Suchanek, J., and V. Kuklik. "Influence of heat and thermochemical treatment on abrasion resistance of structural and tool steels." *Wear* 267, no. 11 (2009): 2100-2108.
- [20] Senthilkumar, T., and Tajudeen K. Ajiboye. "Effect of heat treatment processes on the mechanical properties of medium carbon steel." *Journal of Minerals & Materials Characterization & Engineering* 11, no. 2 (2012): 143-152.
- [21] Efzan, M. N. Ervina, S. Kesahvanveraragu, and J. Emerson. "Microstructural Characterization and Hardness Properties of A333 Grade 6 Low Carbon Steel in Offshore Platform Pipelines." *Journal of Advanced Research in Materials Science* 2, no. 1 (2014): 1–9.