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The effects of cryogenic treatment on the toughness and tribological behaviors of eutectoid steel †

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Abstract

Cryogenic treatment is a supplementary heat treatment usually applied after quenching. Its effects are permanent and this process affects the entire section. There have been many studies related to cryogenic treatment, most of which have been focused on tool steels. In the current study, a high-speed-train railway material was investigated, and different heat treatment processes were applied to the eutectoid steel. The effects of quenching and cryogenic treatment were investigated on the mechanical properties (toughness, hardness and wear). Four different structures were obtained with different heat treatment cycles: Pearlitic, tempered martensite, 12 hour cryo-treated tempered and 36 hour cryo-treated tempered. As a result of Charpy v-notch tests and hardness tests, cryogenic treatment was found to improve the toughness and hardness of quenched samples. The results of the ball-on-disc wear tests showed that the cryo-treated samples have better wear resistance than pearlitic and martensitic samples.

Keywords: Eutectoid steel; Cryogenic treatment; Hardness; Microstructure; Retained austenite; Sub-zero treatment toughness; Wear resistance

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1. Introduction

Cryogenic treatment is an additional heat treatment that has been applied to different materials, especially to metals, to improve the wear characteristics of the material. Significant improvement in the wear resistance for several tool steels has been reported in the Refs. [1-3]. Cryogenic treatment is usually performed just after quenching. Some researchers have focused on determining the optimum soaking time for cryogenic treatments. Most studies have been focused on tool and die steels [2-7], however, further studies have been performed on carbon steels, carburized steels and cast irons [8-11]. Carbon content severely affects the hardenability of steels. Applications that requires high wear resistance, such as tool and dies, requires high carbon steels. The main mechanism used to strengthen this material is quenching. High carbon ratio and the presence of other alloying elements lower the M_s (martensite start) and M_f (martensite finish) temperatures. For most of the commercial tool steels the M_f temperature is below room temperature. As a result, full transformation of austenite to martensite cannot be obtained through conventional heat treatment and quenching. Significant amounts of retained austenite remain in the structure. Retained austenite is undesirable since it lowers the hardness and wear resistance of the steel, on the other hand martensite is hard but brittle phase, which requires tempering to allow further usage. Untempered martensite and retained austenite are quite unstable phases that can cause distortion and dimensional instability. In conventional hardening of steels, the retained austenite ratio is decreased by tempering, which is performed repeatedly in some cases. The duration and temperature of tempering is tailored for different cases. This process has some disadvantages, however tempering, especially for long durations, can lead to softening of the material, coarsening of grain structure and may result in other undesirable effects. An alternative way to transform retained austenite is through cryogenic treatment which has no negative effects on hardness or grain structure. Subzero treatment can, in some cases, improve wear resistance and mechanical properties. Studies have shown that subzero treatment also has benefits related to dimensional stability and service life [12, 13]. The main mechanisms of cryogenic treatment were reported in previous studies [14, 15]. These studies illustrated that cryo-treatment induces significant modifications in the precipitation behavior of Secondary carbides (SCs) and a marked reduction in the retained austenite content, thereby improving the tribological properties of die steels. It can be said that formation and distribution of secondary carbides are the main mechanisms that control the tribological and mechanical behaviors of tool steels. At certain

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durations, cryo-treatment gives optimum performance. Previous work related to D2 steel suggests that the soaking duration has a significant effect on the precipitation behavior of SCs. The amount, population density and distribution of SCs exhibit excellent correlation with the variation of the estimated average wear resistance of the cryo-treated specimens [16]. The afore mentioned study recommends 36 hour, -196 °C treatment for D2 tool steels.

The subzero treatment temperature varies with the application mode. For example, temperatures down to -50 °C can be obtained with dry ice or electric powered cooling systems. Nitrogen is used to obtain temperatures below -50 °C, a process generally termed as cryo-treatment. Cryo-treatment is classified with respect to the operational temperature as deep (approximately -196 °C) or shallow (approximately -86 °C). Operation durations are quite long, generally lasting between 12-72 hours. Cryogenic treatment removes a remarkable amount of retained austenite in both deep and shallow treatment. However, wear resistance and hardness values change with the duration and operating temperature of the treatment. Although cryogenic treatments have great potential in some cases, it should be noted that cryogenic processing results can also be controversial [17].

In the current study, eutectoid steel was investigated. One of the most basic uses of eutectoid steel is railway infrastructure. Currently, the majority of rails are made from plain carbon-manganese steel. This material is used in different operating conditions with different heat treatments. Heat-treated rails are cooled at higher rates to obtain smaller spacing and higher hardness values of 340-400 HB. The hardness of modern HT pearlitic rail can be increased to 350-400 HB without alloying in pearlitic structure [18, 19]. Generally, studies related to cryogenic treatment are focused on tool steels that contain serious amounts of alloying elements (Cr, Mo, Mn, Ni, Si, V…). However, plain carbon steel was chosen for the current study. The reason for this selection is that for plain carbon steels, understanding the microstructure and the effects of cryogenic treatment is much simpler. The current study also shows the potential of cryogenic treatment on case carburized steels. There are several studies in the literature related to the effect of cryogenic treatment on wear properties; however, most of these are focused on short duration wear properties. Generally machine elements are replaced or repaired after determined wear limits but some of the elements must perform long duration conditions such as dies, train rails and biomedical devices. Short duration tests represent the parts that were replaced or failed after initial degradation. Long duration wear tests represent the machine elements which were used with deformed condition for longer durations. In the current study, wear tests were carried out at two different durations. The effect of cryogenic treatment on long duration properties is also important.

This work was divided into five steps: Determining the properties of the reference material, designing the heat treatment processes, evaluating the microstructures, performing the Charpy v-notch test, conducting the hardness test and the wear test on the samples. In the scope of this study, different microstructures were handled by applying different heat treatment procedures. The effects of different heat treatments on the toughness, hardness and wear behaviors were investigated.

Table 1. Spectral analysis results of the material investigated.

Element	Weight %
C	0.86075
Si	0.3275
Mn	0.99025
Mo	0.002175
Fe	Balance

Fig. 1. Cryogenic processing unit.

2. Experimental work

At the beginning of the experimental work, the supplied material was investigated and verified. The chemical composition of the material was obtained using spectral analysis as listed in Table 1. The experimental procedure adopted in the present study is schematically shown in Fig. 2.

2.1 Heat treatment

Heat treatment data were obtained by using Calphad techniques. A similar approach was found in Ref. [20]. The initial grain size and chemical composition of the material were measured with an optical microscope. The austenitizing temperature was determined as 860 °C. This method was previously used to alloy designs with a similar material [19]. TTT diagrams were compared to the AISI 1080 diagram and used to design the quenching process.

Two sample groups were austenitized and cryo-treated at -190 °C using two different durations. Each sample was tempered at 200 °C for two hours after cryogenic treatment.

(1) Austenitizing at 860 °C: The samples were put into the furnace at room temperature and maintained in the furnace for one hour.

(2) Preparation of the oil bath at 20 °C: Samples (Group 2, 3 and 4) were quenched immediately after austenitizing.

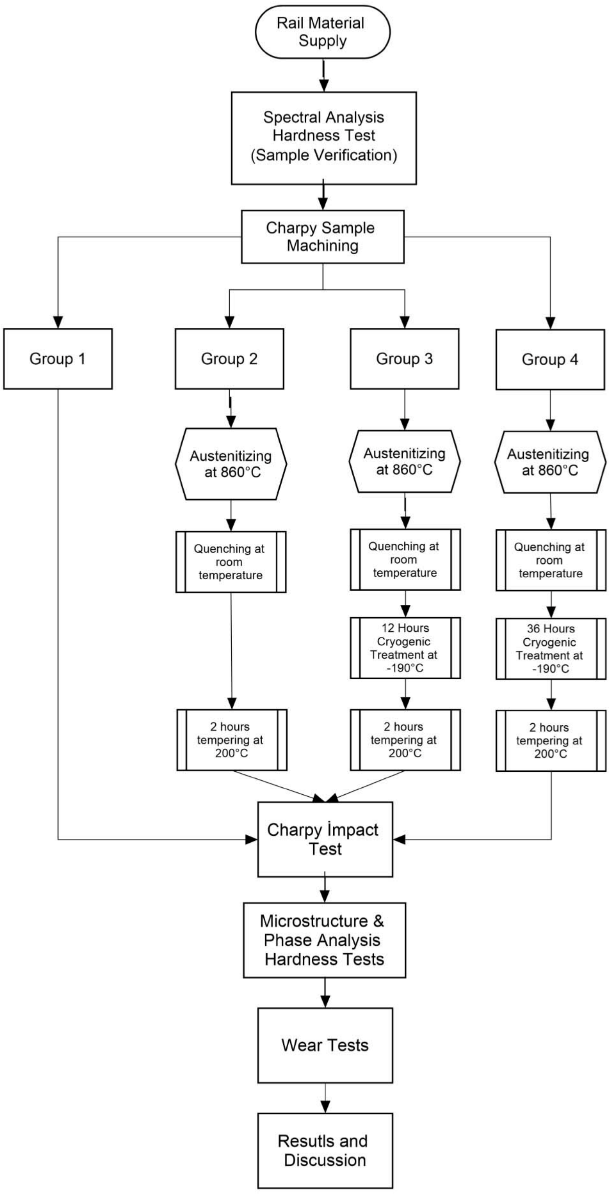


Fig. 2. Experimental procedure.

(3) The samples were put into the cryogenic treatment unit just after the quenching. Group 3 samples were held for 12 hours and group 4 samples were held for 36 hours at -190 °C. All samples were cooled slowly to prevent thermal damage. The cryogenic processing unit is shown in Fig. 1 and the cycle used is shown in Fig. 3.

(4) After cryogenic treatment, samples were heated slowly to room temperature. Heating was conducted slowly to prevent damage.

(5) Group 2, 3 and 4 samples were tempered at 200 °C for two hours.

Fig. 3 illustrates the cryogenic process steps. Following the quenching process, cryogenic treatment was carried out to achieve the retained austenite to martensite transformation.

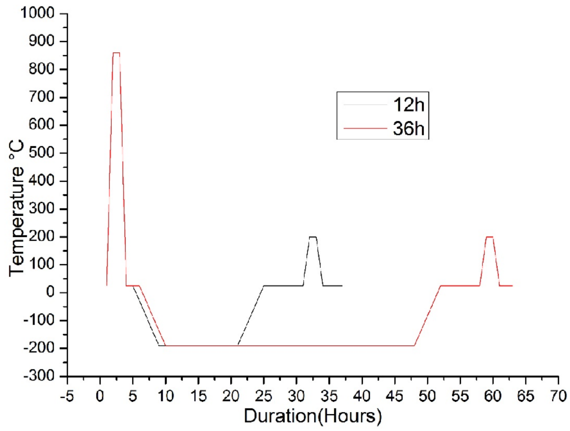


Fig. 3. Cryogenic treatment cycle.

The subsequent tempering process was also carried out to reduce internal stresses in the structure. It should be noted that the size of the sample treated in the cryogenic process is also influential on the process time. Decreasing the temperature reduces the diffusion rate, which is one of the reasons why the processing times are relatively long. The sample sizes (55 mm × 10 mm × 10 mm) used in this study also have an effect on the processing time. At larger sizes, it will take more time for the parts to cool to -190 °C, and thus, the process times are likely to be longer.

3. Results and discussion

3.1 Charpy impact test

According to American Society for Metals (ASM), the Charpy impact test standards are ASTM D 256 and ISO 179. The Charpy impact test consists of a simply supported beam with a centrally applied load on the reverse side of the beam from the notch. The test creates a stress concentration at the notch and produces a constrained multi-axial state of tension slightly below the bottom of the notch. The load is applied dynamically using a free-falling pendulum with the initial potential energy of 300 J. The tests were performed under these conditions. The prepared Charpy v-notch samples were tested at room temperature conditions. The results of the impact test show consistency among the test samples. According to test result, impact energy was decreased for quenched samples relative to pearlitic structure. Cryogenic treatment improves fracture energy for cryo-treated tempered samples by 27 % and 26 % for 12 and 36 hour treatments, respectively, relative to quenched and tempered samples.

The impact energy per unit area for all the samples is shown in Fig. 4. The experimental results show that quenched samples possess lower toughness. This is expected for martensitic transition. However, slightly higher toughness values were obtained for cryo-treated samples, despite the fact that cryo-treated samples have less retained austenite. Most of the retained austenite was transformed during the cryogenic treatment and tempering processes. The increase in toughness can

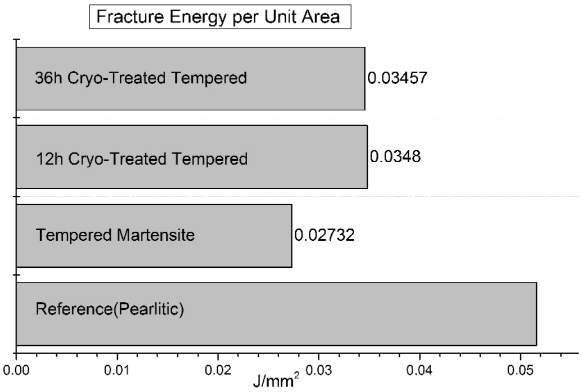
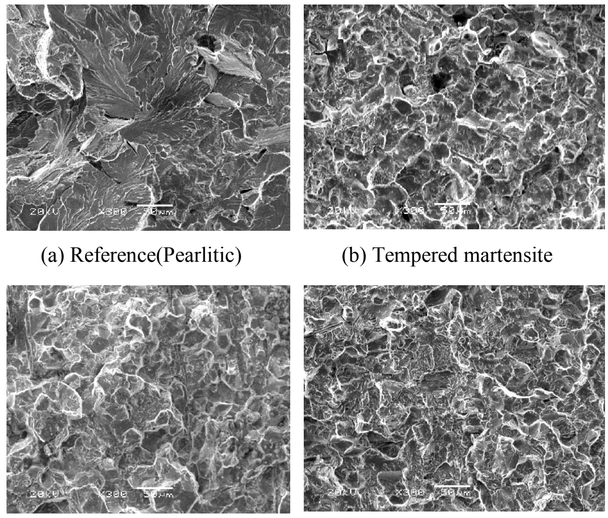


Fig. 4. Average fracture toughness of samples.

be explained by the finer precipitation of carbides that was formed during cryogenic treatment. These results show that higher toughness can be obtained by using subzero treatment without sacrificing hardness.

Similar results were previously obtained with AISI H13 steel [21]. In their work, H13 steel was subjected to different cryo-treatments as well as conventional heat treatments. The researchers showed that Deep cryogenic treatment (DCT) can substantially (24 %) improve the fracture toughness of tool steel without affecting other mechanical properties, such as hardness and strength. One of the main reasons for this is the reduction of retained austenite content. The authors reported that the improvement in fracture toughness of steels by DCT has been attributed to the refinement of SCs and their improved homogeneous distribution, in addition to the development of a tougher tempered martensite matrix. This mechanism is based on the carbon reduction from the martensite phase. The carbon atoms were extracted from the martensite resulting in the precipitation of SCs at cryogenic temperatures. Secondary carbides were clearly shown in the study by Das et al. with high magnification [14].

The fracture process has two steps: Crack formation and crack propagation. The fracture mode is determined based on the crack propagation mechanism. Ductile fracture is identified with major plastic deformation around the initiated crack. The fracture proceeds when the crack length increases. In ductile fracture, major plastic deformation is observed at the fracture surface, such as twisting and tearing. For brittle fracture, the initiated cracks rapidly spread, so that large plastic deformations cannot occur. After the crack initiation, fracture soon occurs. Fig. 5 shows SEM images of fracture areas for all samples. Pearlitic structure shows ductile-brittle fracture, while for quenched samples, the fracture model were more likely brittle fracture. Cryo-treated samples have more microcracks which was a sign of improved toughness. CT treatment leads to fine carbide precipitation, precipitated carbides acts as a plastic deformation preventive barrier point. This is the basic mechanism that improves the mechanical properties. It can be said that quenching decreases fracture toughness but cryogenic treatment helps to improve fracture toughness at some level.



(a) Reference(Pearlitic) (b) Tempered martensite

(c) 12h Cryo-treated tempered (d) 36h Cryo-treated tempered

Fig. 5. SEM image of fracture areas.

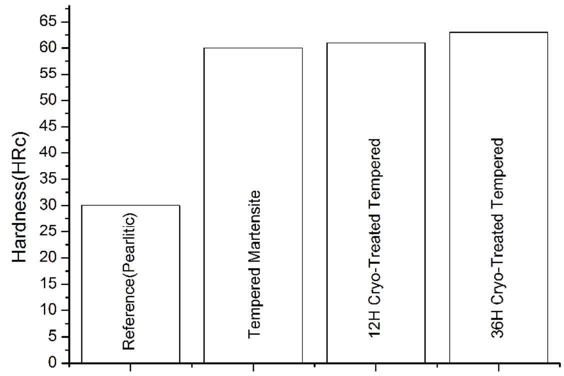


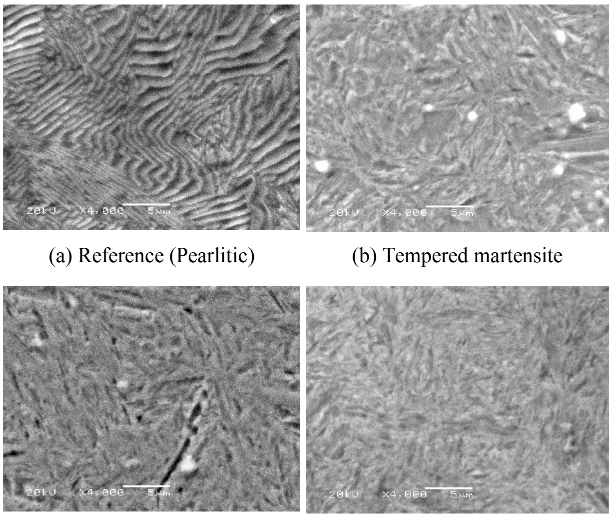
Fig. 6. Average hardness of samples (HRC).

3.2 Hardness test

Hardness is a measure of the resistance of the material to localized plastic deformation, e.g., the averaged hardness of the reference samples is 33 HRc, which is approximately 350 Hb. Hardness can be increased by quenching. Additional cryogenic treatment increases hardness of samples slightly. As the holding time increases, hardness increases slightly. Fig. 6 shows the average hardness of samples.

3.3 Microstructure and phase analysis

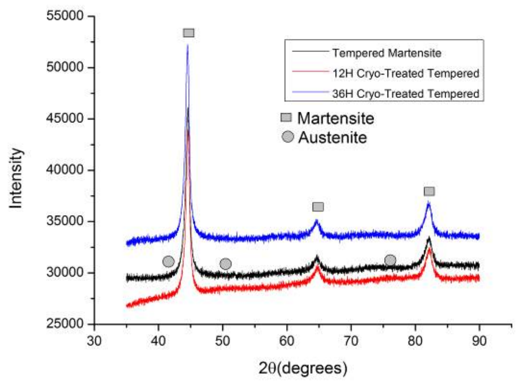
For the microstructure investigations, the samples were cut from the Charpy v-notch samples into 10 mm sized cubes using a cutting machine. The samples were mounted on polymer at approximately 180 °C for three minutes. The surfaces of the samples were ground using a Struers Tegraforce automatic grinding machine at three stages of 220, 500 and 700. The grinding load for each sample was 30 N for 10 minutes. After grinding, the samples were polished using the same



(a) Reference (Pearlitic) (b) Tempered martensite

(c) 12h Cryo-treated tempered (d) 36h Cryo-treated tempered

Fig. 7. Microstructure of samples SEM 4000X.



Volume (%)	Tempered martensite	12h Cryo-treated tempered	36h Cryo-treated tempered
Martensite	92.2	93.3	96.7
Retained austenite	7.8	6.7	3.3

Fig. 8. XRD patterns and volume fractions of different phases.

machine for three minutes with 3 μm diamond suspension. The samples were etched with 2 % nital. Fig. 7 shows the microstructure of the samples. For the reference sample, pearlitic structure was clearly detected. Only slight differences were noticed at 4000X magnification between quenched and cryo-treated samples.

XRD tests were performed to determine the austenite and martensite ratio to determine the phase transformation. XRD results were evaluated with Rietveld refinement method. Fig. 8 shows the XRD peaks of the samples. The amount of retained austenite for tempered martensite was approximately 7.8 %, while for the 36h cryo-treatment, the amount of retained austenite decreased to 3.3 %. The 12h cryo-treatment also lowered the amount of retained austenite.

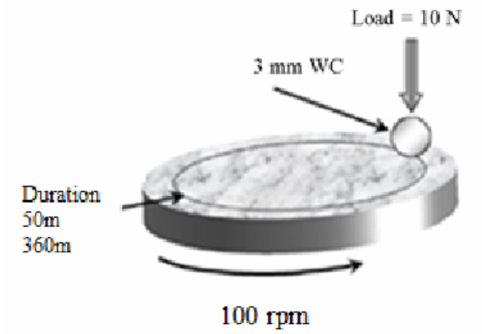


Fig. 9. Wear test conditions.

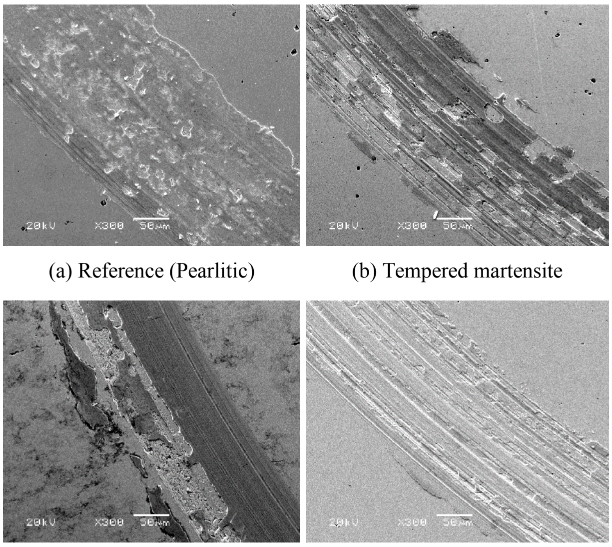
Fig. 10. Wear test module, CSM tribometer.

3.4 Wear tests

The wear test samples were ground, and the polished-surface roughness of the samples was between 0.010-020 μm. A new, certified WC ball was used in the wear tests. The wear tests were performed using a ball-on-disk geometry according to the DIN 50324 standard. In the experiments, the counterpart was Ø 3 mm with a WC-6 % ball, whose sphericity and compositions were certified. The hardness of the ball was 91.6 HRC, its modulus of elasticity was 690 GPa, and its Poisson ratio was 0.22. Only the test material was worn at the end of the experiment because the material that was used was quite harder than the samples. The wear test parameters were 10 N load, 2.5 mm wear radius and 50 and 360 m distance. The calculated Hertz pressure during the tests was 2.903 GPa. Test setup and parameters are shown in Fig. 9.

The wear test was performed on a ball-on-disk tribometer. The load was maintained constant at 10 N during all tests. The test conditions were the same for all tests, and designed to represent extreme wear cases. Fig. 10 shows the tribometer system. All wear test samples were cleaned with alcohol prior to testing. After each wear test, several profile measurements were performed to determine the worn area of the wear section. The worn sections were evaluated with using a special software. Wear volumes were calculated by taking account of many tests and average values were obtained. This approach is in line with a similar method published in the Ref. [22].

The wear tests were performed in two stages under identical conditions. The first test was performed for 50 m while the



(a) Reference (Pearlitic) (b) Tempered martensite

(c) 12h Cryo-treated tempered (d) 36h Cryo-treated tempered

Fig. 11. SEM images of the worn sections.

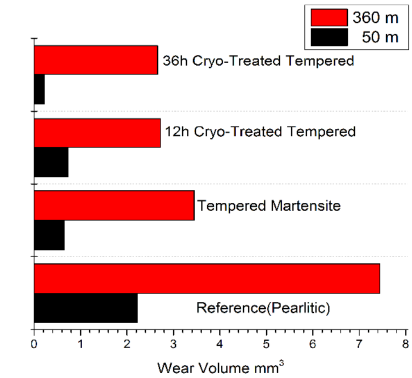


Fig. 12. Average wear volume of samples.

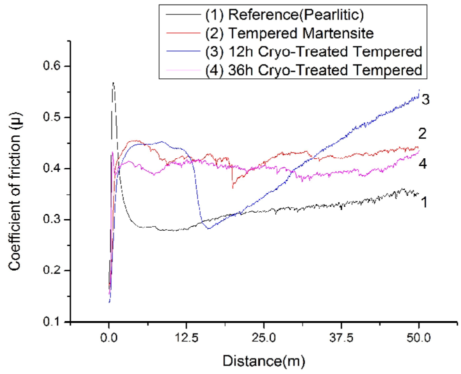


Fig. 13. 50 m wear test friction coefficients.

second wear test was performed for 360 m with identical parameters. Long duration wear tests show the effects of plastic deformation and hardening behavior of a material. The 36h cryo-treated and tempered structure has better wear performance than the pearlitic in both short and long duration tests. In particular, 36h cryo-treated samples have higher wear resistance than the 12h cryo-treated and tempered martensite structure. Similar results were obtained by previous researchers [23, 24]. Long duration wear test results were in good agreement with previous work [24]. The results shows that 36h cryo-treated samples offer 280 % better wear resistance compared to the rolled condition based on the 360 m tests. In addition, the 36h cryo-treated steel offers 30 % improvement compared to tempered martensite samples. The wear results of 50 m wear tests using 12h cryo-treated samples were close to tempered martensite. Soaking time resulted in a dramatic effect on the short duration wear results, in contrast to the long duration wear results which showed minimal dependence on soaking time. In order to obtain maximum performance of cryogenic treatment, the soaking time must be chosen according to the working environment. Fig. 11 shows the SEM images of the worn sections and Fig. 12 shows the average wear volume of samples. It can be seen that the 36h cryo-treated samples wear characteristic is much more abrasive than the other samples. Wear debris and delamination is severely reduced by the effect of cryogenic treatment.

In this study, wear tests show that the cryogenic process improves the wear properties of eutectoid steel. The sample subjected to the 36h cryogenic treatment showed an improved wear resistance of 70 % compared to tempered martensite in the 50 m test, and 23 % better resistance compared to the tempered martensite sample at 360 m test. In the 50 m test, 12h cryo-treated samples wear volumes were close to tempered martensite samples.

The tribometer can record in situ friction. Friction data were stored digitally during the tests. Friction coefficients (μ) of the samples are given in Fig. 13. Regarding the friction coefficient of the investigated ball-on-disk system, the reference sample has the lowest friction coefficient (approximately 0.34). The 36h cryo-treated samples offer a higher coefficient of friction (approximately 0.4).

The lowest coefficient of friction was observed in pearlitic sample. The oxide film was formed and broken continuously in pearlitic sample. The wear debris acted as solid lubricant which lowered the Coefficient of friction (COF). On the other hand quenched samples showed different characteristic. Oxide film was not worn out easily so abrasive marks were formed. For 12h cryo-treated sample, the fracture of the oxide film took place and wear debris lowered the friction at 15 m after this point the contact was dry and COF was raised. It was thought that if test duration is longer it will be stabilized like other samples. Ploughing effect was dominant in cryo-treated samples. The oxide film was not broken and this was result in abrasive marks. 36h cryo-treated samples did not show delamination or smearing and thus, this resulted in higher friction coefficient. In 12h cryo-treated samples, there are still abrasive marks but not intense as those of in 36h cryo-treated samples. It can be said that the surface was deformed but not worn out so the friction coefficient raised but worn surface were not broken so wear volumes were limited.

4. Conclusions

The Charpy v-notch tests show that the cryo-treated tempered samples exhibit better dynamic toughness than tempered martensitic steel. The wear results of the ball-on-disk tests show that the cryogenic treatment improves wear resistance and this effect becomes more dominant in long duration tests. Cryogenic treated samples performed better than the martensitic samples in wear tests. Cryogenic treatment leads to the transformation of retained austenite to martensite. This paper clearly shows the potential of cryogenic treatment on plain carbon steels and carburized steels. It is thought that the use of cryogenic processed materials will increase in the future. Considering the current effect of the process, it will be more effective to reduce the cost of cryogenic processing or to shorten the processing time with different methods.

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