

Microstructural and corrosion behavior of D3 tools steel and 440C SS for blade application

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Abstract. D3 tools steel and 440C stainless steel (SS) are normally being employed for application such as knife blade and cutting tools. These steels are iron alloys which have high carbon and high chromium content. In this study, lab work focused on the microstructural and corrosion behavior of D3 tools steel and 440C SS after went through heat treatment processes. Heat treatments for both steels were started with normalizing at 1020 °C, continue with hardening at 1000 °C followed by oil quenching. Cryogenic treatment was carried out in liquid nitrogen for 24 hours. The addition of cryogenic heat treatment is believed to increase the hardness and corrosion resistance for steels. Both samples were then tempered at two different tempering temperatures, 160 °C and 426 °C. For corrosion test, the samples were immersed in NaCl solution for 30 days to study the corrosion behavior of D3 tool steel and 440C SS after heat treatment. The mechanical properties of these steels have been investigated using Rockwell hardness machine before heat treatment, after heat treatment (before corrosion) and after corrosion test. Microstructure observation of samples was carried out by scanning electron microscopy. The corrosion rate of these steels was calculated after the corrosion test completed. From the results, the highest hardness is observed for D3 tool steel which tempered at 160 °C (54.1 HRC). In terms of microstructural analysis, primary carbide and pearlite in the as-received samples transform to tempered martensite and cementite after heat treatment process. From this research, for corrosion test, heat treated 440C SS sample tempered with 426 °C possessed the excellent corrosion resistance with corrosion rate 0.2808 mm/year.

Keywords: 440C stainless steel (SS); corrosion; cryogenic treatment; D3 tool steel

1. Introduction

Tool steel is a specific type of metal that is used for the purpose of cutting, sculpting, or otherwise forming a material into a component or part that can be adapted to a particular application (Bahadur *et al.* 2019). Typically, forming operations that take place at temperatures lower than 200 °C call for cold work tool steels. The AISI D series is a subset of this family; chromium (at a concentration of 12%) is the primary alloying element in this material. One of the most notable mechanical properties

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of D3 tools steel is its hardness. It has a Rockwell hardness of 56-62 HRC (Lajis *et al.* 2010), making it one of the hardest materials available for toolmaking. Among them, AISI D3 steel stands out due to its wear and toughness resistance, combined with a high compression resistance.

AISI D3 steel is a high-carbon, high-chromium steel designed to withstand wear, severe pressure, and abrasion in a variety of settings. Tools such as slitting cutters, cold trimmer and burnishing rolls, slitting dies, blanking dies, punching dies, and stamping dies are all examples application of tool steels (Ajay *et al.* 2018). Due to this tool steel's superior working performance and affordable price, D3 tools steel continues to expand in the knife industry, where it competes with other low-cost knives that are currently establishing their reputation.

440C SS (stainless steels) is classified as martensitic stainless steel as this steel is high in chromium content and carbon content. 440C SS is the martensitic stainless steels that possess the highest mechanical strength, hardness and wear resistance due to its composition (Salleh *et al.* 2009). 440C SS is usually being utilized in the manufacturing of propellers and valves which requires materials with high mechanical strength and moderate corrosion resistance. These 440C SS usually received in the annealed state, and the final products are hardened by quenching that induces martensite solid-state transformation, followed by tempering treatments. Martensitic stainless steel undergoes heat-treatment process starting with annealing to soften the steel in preparation for subsequent cold work or machining, continued with austenitizing in order to form an austenitic structure and fully or partially dissolve carbides, and then direct quenching were carried out to transform the austenite to martensite phase and lastly tempering to a specific temperature to enhance the toughness and ductility of the steel (Barlow and Du Toit 2012).

Okafor (2012) in his study stated that the main purpose of steels undergoes heat treatment are to improve their ability to be machined and improved their mechanical properties such as yield strength, resistance to corrosion, tensile strength, and creep performances, increase strength and toughness, release residual stress and control their microstructures. In the process of conventional heat treatment, steps like as annealing, case hardening, tempering, normalizing, and quenching, are among the many steps that can be performed (Barlow and Du Toit 2012). Quench heat treatment is a commonly used process of hardening steel (Samuel and Narayan 2022). While cryogenic treatment which is proposed in this study is regarded as one of the supplementary treatments for conventional heat treatment (Uygur 2015, Zhang *et al.* 2022). The purpose of applying cryogenic treatment is to improve some mechanical properties of materials by changing microstructure to enhance service performance or extend the service life of tools or parts (Zhang *et al.* 2022).

In this research, the lab work focuses on the effect of heat treatment to microstructure, hardness, and corrosion behaviour of D3 tool steel and SS 440C. Heat treatment consisting of normalizing, quenching, cryogenic and tempering at two different temperatures. The difference in this tempering temperature is applied onto those steels to modify the microstructures of the steels, to increase the hardness of the steels and to enhance the corrosion resistance in means for blade applications. Other than that, the addition of cryogenic heat treatment to this research is believed to increase the hardness of cold work steel (Mahmoud *et al.* 2023). Nandakumar (2017) explained that cryogenic treatment on the AISI D series steels results in improvements of hardness and wear resistance because of more

Table 1 Composition analysis of D3 tools steel

Element	C	Mn	Si	Cr	V	W	Fe
Composition (wt.%)	0.918	0.244	0.259	12.28	0.345	0.027	84.79

Table 2 Composition analysis of 440C SS

Element	C	Mn	Si	Mo	Cr	Fe
Composition (wt.%)	0.841	0.244	0.387	0.394	16.48	81.06

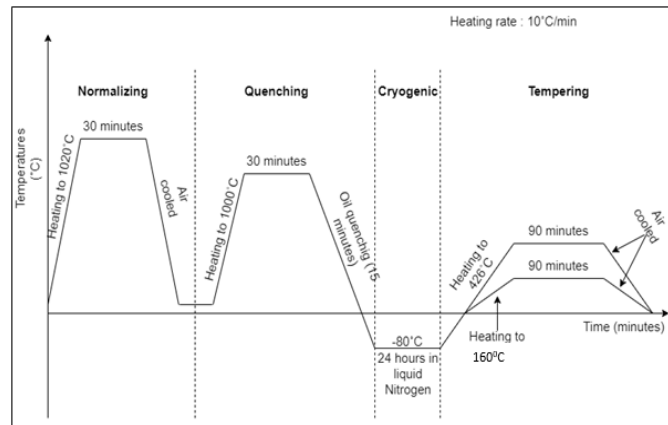


Fig. 1 Overall heating profile for D3 tool steel and 440C SS

fine carbide formation. Yuhua *et al.* (2020) reported that tensile strength and reduction of the cross-sectional area of steel were increased by 170 MPa and 9% and yield ratio and elongation were decreased by 0.13 and 9% due to the existence of the large-angle grain boundary. Their study confirms that the mechanical properties of steel can be improved effectively by the deep cryogenic treatment and tempering (Yuhua *et al.* 2020).

2. Materials and methods

2.1 Material

The steels studied in this research are D3 tools steel and 440C SS. The as-received materials were in annealed condition. The chemical composition for the material acquired using arc-spark spectroscopy (OES). Table 1 and Table 2 show the composition of D3 steel and 440C SS.

2.2 Sample preparation and heat treatment

The received annealed samples of D3 tools steel and 440C SS were already cut to the exact size (2 cm x 2 cm). For each steel, there will be one reference sample which remains untreated (already annealed). The samples were grounded, polished, and etched with ferric chloride (FeCl₃) etchant for 7 minutes before conducting microstructural analysis. Samples then were normalized to 1020 °C in muffle furnace, soaked for 30 minutes and air-cooled. After that, both steels were gone through hardening process at 1000 °C and immediately quenched in oil for 15 minutes. The samples were immersed in liquid nitrogen for cryogenic treatment for 24 hours. Then, the samples for each type of steel were divided equally for two different tempering temperatures 160°C and 426 °C. Fig. 1 shows heating profile for normalizing, quenching, cryogenic treatment and tempering at 160 °C and

426 °C.

2.3 Hardness

Hardness test before corrosion and after corrosion test of D3 tools steel and 440C SS have been conducted to study the changes in hardness of the treated and untreated sample. Hardness test was conducted according to ASTM E18 (Standard Test Methods for Rockwell Hardness of Metallic Materials) with 120° diamond indenter and 150 kgf of total test force.

2.4 Microstructural analysis

Characterization of the surface morphology of the untreated and heat treated D3 steels and 440C SS samples, were analyzed by scanning electron microscopy (SEM). The samples were etched with a ferric chloride etchant to get a high degree of smoothness to reveal its microstructure and a clearer image of the surface morphology. Tescan Vega 4th generation scanning electron microscope (SEM) is used to investigate the changes in microstructure and surface morphology in the sample before and after conducting heat treatment and after corrosion test. The sample size use for this microstructural analysis is 10 mm×10 mm.

2.5 Corrosion test

The as-received (annealed) and heat-treated sample for D3 tool steel and 440C SS were then immersed in the 3.5% of NaCl solution for corrosion resistance test. Corrosion tests were carried out according to ASTM G31-72 (Standard Practice for Laboratory Immersion Corrosion Testing of Metals). The weight loss for each sample was recorded once in three days. After 30 days of corrosion test, corrosion rate was calculated. The corrosion rate of the steel was calculated by using Eq. (1).

$$\text{Corrosion rates, } C_R = \frac{K \times W}{A \times T \times \rho} \quad (1)$$

Where

K =constant (8.76×10^4)

T =time of exposures in hours to nearest 0.01 h

A =area in cm^2 to the nearest 0.01 cm^2

W =mass loss in g, to the nearest 1 mg

ρ =density in g/cm^3

3. Results and discussion

The results obtained from the characterizations and corrosion test were carried out with the purpose of evaluating the effect of heat treatment on D3 tools steel and 440C SS, and to identify changes on the steel hardness, microstructure, and its performance against the corrosion.

3.1 Hardness before corrosion test

Table 3 shows the results for hardness of as received (annealed) and heat-treated (HT) of D3 tool steel and 440C SS before corrosion test. From Table 3, hardness for the D3 tool steel for annealed,

Table 3 Hardness of D3 tool steel and 440C SS before corrosion test

Samples	Hardness (HRC)	
	D3 tool steel	440C SS
As-received (annealed)	18.5	15.5
HT-Tempered at 160 °C	54.1	51.5
HT-Tempered at 426 °C	51.4	50.9

HT -tempered at 160 °C and HT-tempered at 426 °C are 18.5 HRC, 54.1 HRC and 51.4 HRC respectively. While for the 440C SS samples, the hardness analyzed for untreated sample is 15.5 HRC, HT-tempered at 160 °C sample exhibit hardness of 51.5 HRC and HT-tempered at 426 °C have hardness of 50.9 HRC.

As-received (annealed) D3 tool steel and 440C SS has the lowest hardness due to the presence of ferrite and carbide in the as-annealed microstructure which make the steel become soft (Ghasemi and Jahazi 2014). After undergoing a series of heat treatment that consists of normalizing at 1020 °C, oil quenching at 1000 °C to room temperature, cryogenic treatment at temperature -80 °C and tempering at 160 °C for 1.5 hours, the hardness of D3 steel and 440C SS significantly increases. Kumar *et al.* (2017) has reviewed the effect of cryogenic treatment on cutting tool life and its performance. In their study, they have made a conclusion which is cryogenic treatment has proven to be an effective method for improving the mechanical properties of various grades of steel, including their resistance to wear and their level of hardness. This is because the hardness of steel can be increased with cold treatments because the soft retained austenite is transformed to hard martensite.

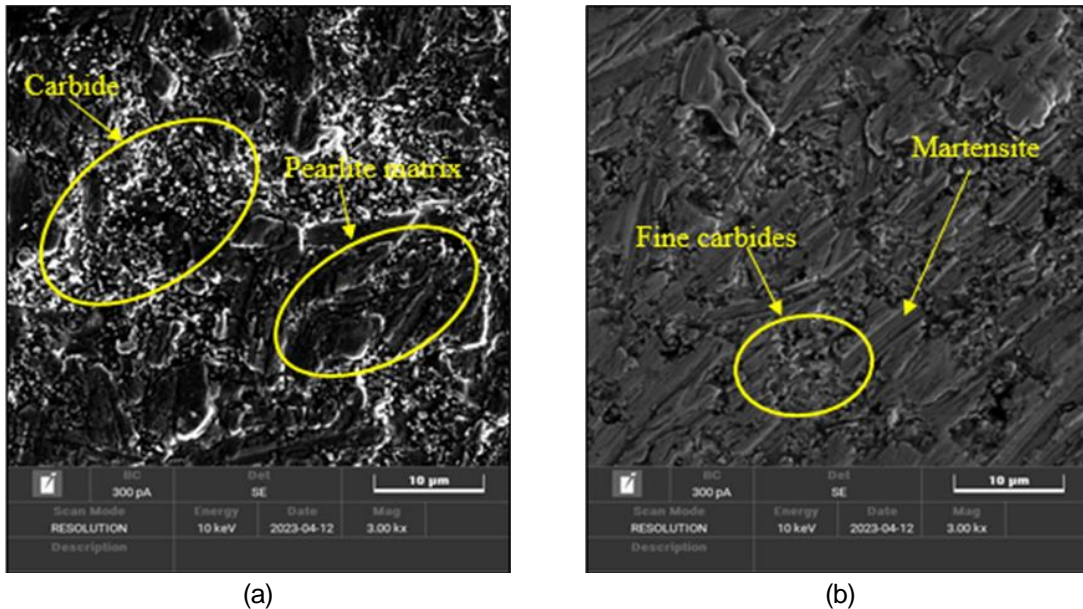


Fig. 2 SEM images of D3 tool steel for (a) As-received (annealed) sample and (b) Heat treated sample, tempered with 160 °C (HT-tempered at 160 °C)

The increases of hardness at this lower tempering temperatures (160 °C) can be supported by referring to result gained by Ghazi and Mashloosh (2015). The authors observed that when D3 tool steel was cryogenically treated at -198 °C for 36 hours and tempered at 200 °C for 30 minutes, this D3 tool steel sample exhibited the maximum hardness of 69 HRC. While for the sample that heat treated with tempering temperature of 426 °C, the hardness of the sample is higher than the untreated sample however it is lower than the hardness of the sample tempered at 160 °C. This is because, a tempering steels at higher temperature will resulting in decreasing its hardness as reported by Saha *et al.* (2012). During tempering, solid-state reactions occur and the as-quenched martensite is transformed into tempered martensite, which, at higher tempering temperatures, is composed of highly dispersed spheroids of cementite (carbides) dispersed in a soft matrix of ferrite, resulting in reduced hardness and increased toughness.

The sample of 440C SS tempered at 160 °C exhibits the highest hardness among those 440C SS sample. The increase in hardness of the stainless steel is because of the transformation of ferrite matrix and primary carbides (in the as-annealed sample of untreated sample) (Kwok *et al.* 2003) to tempered martensite and carbides such as M_7C_3 , $M_{23}C_6$ and M_2C (Salleh *et al.* 2009) that are much harder after undergone heat treatment. However, after tempering at higher temperature, the hardness of the steel is much lower than tempered at lower temperature. In Xu (2012), she study the dissolution of secondary carbide and the coarsening of 13% Cr martensitic stainless steel (MSS) during austenitizing, she found that when the tempering temperature was raised above 280 °C, the hardness of the MSS sample decreased due to over tempering, which was caused by the coarsening of tempered carbide particles.

3.2 Microstructural analysis before corrosion test

One sample of as-received (annealed) D3 tool steel and one sample of heat treated D3 tool steel which was tempered at 160 °C was taken to be analysed to observe the changes of the microstructure inside the steel before and after heat treatment. The tempered D3 steel sample at 160 °C was chosen instead of the other one tempered with higher temperature (426 °C) because this tempered steel exhibits the highest hardness (as shown in Table 3) among those four heats treated samples (all of samples for D3 tool steel and 440C SS). The microstructure analysis was carried out by using scanning electron microscopy (SEM). Figs. 2(a)-(b) shows the SEM images of untreated (as annealed) D3 tool steel, and heat treated D3 tool steel with 3000× magnification.

Fig. 2(a) shows the microstructure of as received (annealed) D3 tool steel, with the matrix composed of ferrite containing globular carbides. Under these supply conditions, it can be observed that this sample contains a pearlite matrix that has clearly defined grain boundaries and is surrounded by massive and fine primary carbides (Alza 2020). Pearlite is a two-phased, lamellar (or layered) structure composed of alternating layers of ferrite and cementite (Chandra 2021). From Fig. 2(a), this pearlite matrix is surrounded by fine primary carbides. According to Naravade *et al.* (2013) the microstructure of untreated tool steel displays non-uniform distribution of large, elongated white regions of primary chromium carbides and uniform distribution of smaller, nearly spherical secondary chromium carbides. The annealed state of the steel exhibits ferrite, pearlite, and carbides of various compositions, which cause the steel to become soft, ductile, and to have a low tensile strength (Bhateja *et al.* 2012). The result for hardness revealed that as-received (annealed) D3 tool steel shows low value of hardness compared to other heat treated D3 tool steel samples.

Comparing Figs. 2(a) and (b) reveals that the larger carbides present in Fig. 2(a) were significantly reduced in size, transforming into much finer carbides after undergoing heat treatment

Table 4 Hardness of D3 tool steel and 440C SS after corrosion test

Samples	Hardness (HRC)	
	D3 tool steel	440C SS
As-received (annealed)	14.3	13.5
HT-Tempered at 160 °C	52.8	50.1
HT-Tempered at 426 °C	50.8	50.2

with a tempering temperature of 160 °C. After completing the heat treatment process consisting of normalizing, quenching, cryogenic, and tempering, the microstructure observed contains small amount of retained austenite, martensite, and cementite. According to Salih *et al.* (2011), after quenching the phase presence in the steels is martensite with small amount of retained austenite. Chandra *et al.* (2021) concluded that the austenite formed during hardening before quenching will decompose into some medium carbon martensite and fewer pearlite during quenching to room temperature which the martensite form in this stage is too brittle and the steel cannot be use directly after the quenching process for any type of applications. Thus, tempering needs to be applied on the steels for the purpose of increasing ductility and toughness of the steels. In Fig. 2(b) it is clearly seen very fine carbides in the region of martensite in the steel structure. This finding aligns with the research conducted by Nykiel and Hryniewicz (2014), where they investigated the microstructure of D3 tool steel after tempering at 200 °C. In their study, they observed the formation of very fine plate-shaped carbides within the martensite region following the tempering process.

3.3 Hardness after corrosion test

After 30 days of immersion in a 3.5 wt% NaCl solution for corrosion testing, the hardness of three types of D3 tool steel samples was re-evaluated. Table 4 demonstrates that after the immersion test, all samples experienced a slight reduction in hardness values. After the corrosion test, the hardness of the as-received (annealed) sample for D3 tool steel decreases from 18.5 HRC (refer Table 3) to 14.3 HRC (Table 4), while the hardness of the HT-tempered at 160 °C sample decreases from 54.1 HRC to 52.8 HRC. Similarly, for D3 steel, the hardness of the corroded HT-tempered at 426 °C sample is reduced to 50.8 HRC afterward a 30-day immersion test. For 440C SS, after immersing samples in 3.5 wt% NaCl solution for 30 days, hardness values decreased to 13.5 HRC for untreated samples, 50.1 HRC for HT-tempered at 160 °C and 50.2 HRC for HT-tempered at 426 °C.

The reducing in hardness of those steel (D3 tool steel and 440C SS) after immersion test are because of the presence of the chloride ions. Chloride ions are known to be aggressive enough to attack steel and initiate pitting. The Cl ion's aggressiveness stems from its small size, high diffusivity, and strong acidic anionic nature. Sundjono *et al.* (2017) conducted research on the corrosion of mild steel in sea water of the Indonesia by referring to ASTM G1-81. When corrosion happen, the metal is being eaten away by chemicals, and it cause the metal to lose its strength and hardness over time. In the case of steel, the formation of iron chloride results from the reaction of the chloride ions in the NaCl solution with the iron already present in the steel. This iron chloride is a brittle compound that, if it were to flake off the surface of the steel, would leave the steel with less strength and a lower hardness. The amount of corrosion that takes place is proportional to the strength of the NaCl solution, the duration of the steel's exposure to the solution, and the temperature

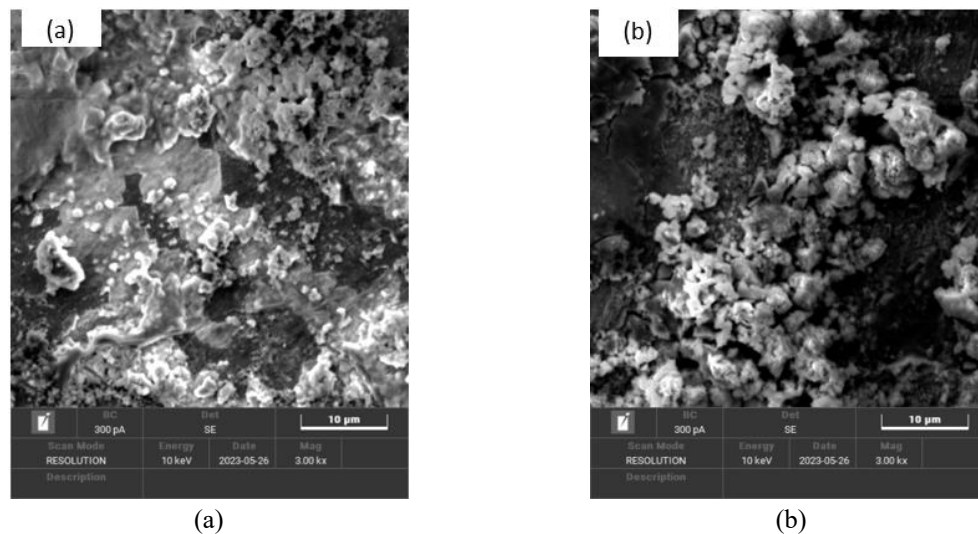


Fig. 3 SEM images of (a) corroded D3 tool steel HT-tempered at 160 °C and (b) corroded 440C SS HT-tempered at 426 °C after corrosion immersion test in 3.5wt% of NaCl solutions (3000x magnification)

Table 5 Corrosion rate of D3 tool steel and 440C SS steels

Samples	Corrosion rate (mm/year)	
	D3 tool steel	440C SS
As-received (annealed)	0.2899	0.3198
HT-Tempered at 160°C	0.3131	0.3120
HT-Tempered at 426°C	0.3401	0.2808

of the solution.

3.4 Microstructural analysis after corrosion test

Fig. 3(a) shows the corroded surface of D3 tool steel which had been HT-tempered at 160 °C, while Fig. 3(b) shows corroded surface for 440C SS after HT-tempered at 426 °C. Those two samples were chosen for microstructural analysis because these samples show the highest value of hardness among heat treated samples after corrosion test. After 30 days of immersion test, we can see the surface of corroded steel is slightly different from the microstructure of this sample before corrosion test. Fig. 3(a) shows the pitting corrosion surface for D3 tool steel after tempered at low temperature (160 °C). As compared to microstructure for 440C SS after tempered at higher temperature, which is 426 °C (Fig. 3(b)), pitting corrosion attack more on lower temperature, which is 160 °C. In Fig. 3(a), it is observed that the surface of D3 tool steel tempered at 160 °C has an outer rust layer structure that is composed of granules of corrosion products like flaky particles. Pitting corrosion also attacks at grain boundaries of 440C SS but not much compared to sample D3 tool steel which have been tempered at lower temperature. This is proved by result for corrosion rate (in Table 5) which stated that sample D3 steel HT-tempered at 160 °C have higher corrosion rate (0.3131 mm/year).

Corrosion occurs on the steel due to the interactions of the metal surface with the acidic chloride ion compounds contained in the 3.5% NaCl solution (Saefuloh *et al.* 2020). Based on the results calculated for the corrosion rate of D3 tool steel and 440C SS samples (as-received and heat treated), the corrosion rate analyzed for D3 tool steel HT-tempered at 160 °C exhibited a higher corrosion rate compared to 440C SS HT-tempered at 426 °C (discussed in section 3.5). However, the hardness of D3 tool steel tempered at lower temperature exhibits the highest hardness among all the samples after the corrosion immersion test, and 440C SS exhibits the lowest hardness among all. According to the research conducted by Atapek *et al.* (2013), it is believed that steel with the highest hardness level should offer superior corrosion resistance when compared to steel with lower hardness. Surprisingly, the findings from this current study contradict this notion. Contrary to the expectations based on the earlier research, the results from this study indicate that the heightened hardness of the steel leads to an increase in its corrosion rate.

Prior to this, it is reported that the corrosion rate of steel was also influenced by the presence of carbon content (Peral *et al.* 2023). This is because the carbon atoms in steel form carbides, which are more prone to corrosion than the iron matrix. During the tempering process, steel is heated within a specific temperature range to enhance its mechanical properties and relieve internal stresses. The diffusion and reaction of carbon atoms with iron atoms during tempering lead to the formation of carbides. The amount of carbon present in the steel significantly influences the extent of carbide formation, with higher carbon content resulting in more available carbon atoms for carbide transformation (Uygur *et al.* 2015). Consequently, steel with increased carbide content, owing to higher carbon levels, offers more potential sites for pitting corrosion to happen on D3 tool steel.

3.5 Corrosion behavior of D3 tool steel and 440C SS

Table 5 provides data on the corrosion rates (in mm/year) for two types of steel, D3 tool steel and 440C SS, under different conditions. The corrosion rate of D3 tool steel and 440C SS were calculated using Eq. (1) as mentioned at section 2.5. In their as received (annealed) state, D3 tool steel exhibits a corrosion rate of 0.2899 mm/year, while 440C SS has a slightly higher corrosion rate of 0.3198 mm/year.

When subjected to a heat treatment process followed by tempering at 160°C, both steels experience an increase in corrosion rates. The corrosion rate of D3 tool steel rises to 0.3131 mm/year, and 440C SS shows a corrosion rate of 0.3120 mm/year. Further heat treatment at a higher tempering temperature of 426 °C leads to a notable increase in the corrosion rates of both steels. D3 tool steel experiences a corrosion rate of 0.3401 mm/year, while 440C SS displays the lowest corrosion rate of 0.2808 mm/year.

These results indicate that heat treatment, particularly at higher tempering temperatures, can significantly affect the corrosion resistance of both D3 tool steel and 440C SS. The data suggests that the corrosion rates generally increase with increasing temperatures, with the effect more pronounced for D3 tool steel compared to 440C SS. D3 tool steel HT-tempered at 426 °C exhibited the highest corrosion rate due to the amount of carbon presence in this steel. Based on the composition analysis of D3 tool steel, the carbon content in D3 tool steel is 0.91% which is higher than 440C SS, 0.841%. Higher carbon content (increased pearlite content) causes an increase in the cementite-to-ferrite area ratio in pearlite, which contributes to an increase in corrosion rate (Katiyar *et al.* 2018).

The corrosion rate of 440C SS for HT-tempered at 426°C exhibited lowest corrosion rates due to the chromium (Cr) composition in 440C SS sample for this study that is much higher than D3 tool

steel. By comparing the results of the composition analysis, the Cr composition in 440C SS is 16.48% which is much higher than in the D3 tool steel which have just 12.28% of Cr. This higher Cr contents or alloying elements which sacrifices corrosion for iron (Al-Qawabah *et al.* 2020) provides the 440C SS with more corrosion resistance through producing a passive oxide film of Cr_2O_3 on the steel surface (Xu 2012).

In addition, Ibrahim *et al.* (2009) investigated the corrosion behaviour of austenitic stainless steels through the utilization of potentiodynamic polarization technique in a 2.5 M NaCl solution. The study revealed that the corrosion resistance of the stainless steels was enhanced with the increase in molybdenum (Mo) and chromium (Cr) content in their alloys (Ibrahim *et al.* 2009). In this research, the results obtained for the corrosion rate are in agreement with all the research mentioned above. The corrosion rate of 440C SS HT-tempered at 426 °C have the lowest corrosion rate which indicates that this steel has higher corrosion resistance compared to other samples.

4. Conclusions

The effect of heat treatment on D3 tool steel and 440C SS were investigated for the purpose of blade application. Received samples were as received (annealed) and heat-treated samples with different conditions of tempering temperatures to investigate effect of heat treatments on the mechanical properties, microstructural also corrosion behavior of D3 tool steel and 440C SS. The conclusions from this study are summarized as follows:

- In as-received annealed condition, the microstructure of these steel composed of pearlite and carbide. After completing the heat treatment process consisting of normalizing, quenching, cryogenic, and tempering, the microstructure observed are a small amount of retained austenite, martensite, and cementite. The heat treatment contributes to the transformation of pearlite and carbide in the annealed condition to a new phase resulting to the increased of the hardness of the samples.
- Heat treated D3 tool steel sample (HT-tempered at 160 °C) exhibited the highest hardness value among other samples with the value of 54.1 HRC.
- The immersion test for 30 days results indicated that the 440C SS sample which has been normalized, quenched, cryogenic and HT-tempered at 426 °C is the most effective corrosion resistant treatment compared with the other treatments in this study. It has a slightly better corrosion resistance with the lowest corrosion rate value, 0.2808 mm/year.
- For blade application, the 440C SS will be chosen which exhibits acceptable value of hardness and have an excellent corrosion resistance to be use in an aggressive environment.

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