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On the Effects of Deep Cryogenic Treatment on Wear Resistance, Hardness and Microstructure of the AISI D2 and D3 Tool Steel

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Abstract: *The present study investigates the effect of cryogenic treatment on AISI D2 and D3 tool steels. This analysis is carried out for several heat treatment cycles that follow various tempering and cryoprocessing sequences that are not analysed erstwhile. Moreover, the effect of these heat treatment cycles on wear characteristics, hardness, and microstructural features of AISI D2 and D3 tool steel is also analysed. It is demonstrated that the cryogenic treatment significantly improves the wear resistance, hardness, and retained austenitic transformation of both types of material specimens under consideration. Furthermore, it is established that single tempering after cryogenic treatment is more effective than double tempering processes carried out in different sequences. It is also revealed that the double tempering sequencing involving cryoprocessing stalls the transformation of retained austenite, leading to higher wear and low hardness in the material*

Keywords: cryogenic treatment, microstructures, wear resistance and hardness

I. INTRODUCTION

The productivity and economy of a manufacturing process is strongly governed by life of the cutting tools. The manufacturing processes that involve blanking, forming, thread rolling, cutting etc. are generally performed using the cold worked tool steel from series D (*die steel*). This steel has high chromium and carbon content that provides it better wear and abrasion resistance as demonstrated by Nanesa et al.(2016) and Korade et al.(2018).

Thornton et al.(2013) asserted that, the typical tempering and quenching treatments in-volved in the making process of these tools leads to the formation of *retained austenite*. This *retained austenite* can transform into *secondary martensite* during the usage of these tools. Therefore, as suggested by Podgornik et al.(2016) and Cardoso et al.(2020), this alteration can result into the formation of cracks, and eventually leads to failure. As de-picted by Thornton et al.(2013) and Li and Wu (2015), this issue can be resolved by making microstructural changes into different stable and metastable states of a material. These microstructural changes can be brought about by the cryogenic treatment (cryotreatment or cryogenic processing), which is a *sub-zero* heat treatment process. Thus, cryogenic treat- ment is effective in improving the wear resistance, and reduce the residual stresses in tool steel, as demonstrated by Firouzdor et al.(2008), and Ray and Das (2017).

The initial study on cryogenic treatment was performed by Barron and Mulhern (1980), and Barron (1982). These studies were focussed on understanding the effects of cryogenic treatment on the abrasive wear resistance of wide range of ferrous alloys. However, each tool material needs to be analysed separately, and an individual making process needs to be established. Cryotreatment is just one process that can be incorporated within the ex- isting process route (Stratton(2007)). It was depicted by Meng et al.(1994) and Molinari et al.(2001) that, the cryogenic treatment can improve the tool properties when applied after the quenching and before tempering process. These results were supported by the study of speed steels after cryoprocessing. This understanding was further improved by Mohan Lal et al.(2001) as they demonstrated that the cryogenic treatment applied soon after quenching results in an increased wear resistance in the tool. Dymchenko and VN (1993) demonstrated that a low-temperature tempering after quenching and cryotreating can avoid the austenite stabilization, which will enable its increased transformation. Similarly, Harish et al. (2009) also demonstrated increase in hardness of *EN31* bearing steel by 14% by adopting deep cryogenic treatment. A novel rapid, cryogenic treatment, which acts as an extended quenching process after a traditional quenching was also proposed by Kamody(1999). Conversely, Yun et al. (1998) demonstrated that, the cryogenic treatment for *M2 high-speed steels* can be performed either after quenching-tempering process or after quenching, as proposed earlier. Therefore, regardless of the approach taken during the heat treatment, the materials demonstrated an improvement in their physical properties. Popandopulo and Zhukova (1980) through their results have demonstrated that tempering should precede the cryogenic treatment to achieve better material properties. Moreover, Molinari et al.(2001) also demon started that cryotreating a material after quenching, followed by the traditional tempering has negligible effect on its properties. Furthermore, Kalin et al.(2006) also supported this study by concluding that, the subsequent tempering after the cryogenic treatment has no significant effect on properties of *M2 high-speed steel*. However, Oppenkowski et al. (2010) asserted that, the parameters such as austenitizing temperature, cooling rate, heating rate, and tempering temperature also influence the properties of tool steel after cryotreatment.

While the consensus is established over the benefits of cryogenic treatment for tool steels; there is still a disagreement on its position in the sequence of heat treatment routine. More-over, an understanding of the effects of cryogenic treatment on wear characteristics, hardness, and microstructures of AISI D2 and D3 tool steel is still obscure. Therefore, the present study demonstrates the effect of different thermal treatment cycles involving cryogenic treatment over the physical properties of AISI D2 and D3 tool steel. Moreover, the effectiveness of cryogenic treatment for these steels is also analysed in the present study.

This paper is structured as follows. The experimental details and material properties are described in Section 2. The experimental results, and their analysis is discussed in Section3. Finally, the present study is concluded in Section 4.

II. MATERIALS AND EXPERIMENTAL DETAILS

2.1. Materials

The present study is based on the AISI D2 and D3 tool steel which is most commonly used tool steel material for dies having hardness range of 60-64 HRC. AISI D2 cold work tool steel is preferred for the manufacturing of screws, fine blanking sheets of up-to 6 mm thickness, plastic molds, wood cutting tools, cold rolling applications, de-burring processes, and deep drawing dies. On the other hand, AISI D3 tool steel is preferred for applications such as pressing, deep drawing, in blanking sheets of up-to 4 mm thickness, and in cutting dies. The chemical composition of these materials is depicted in Table 1. The chemical composition of AISI D2 and D3 steel is analysed using Optical Emission Spectrometer (*Spectrophoto Analyzer - AS 200 (Switzerland)*).

Material	*C*	*Mn*	*Cr*	*Mo*	*V*	*Si*	*Fe*
AISI D2	1.5	0.36	12.53	0.83	1.04	–	Balance
AISI D3	2.23	0.49	12.31	–	–	0.26	Balance

Table 1: Chemical composition in weight percentage of investigated AISI D2 and D3 cold work tool steel

2.2. Heat Treatment

The materials under consideration are subjected to the various heat treatment routines namely AQCT (following the sequence as *Austenitizing, Quenching, Cryotreating, and Tempering*), AQTCT (following the sequence as *Austenitizing, Quenching, Tempering, Cryotreating, and Tempering*), AQCTT (following the sequence as *Austenitizing, Quenching, Cryotreating, Tempering, and Tempering*), AQC (following the sequence as *Austenitizing, Quenching, and Cryotreating*), and AQT (following the sequence as *Austenitizing, Quenching, and Tempering*). Fig. 1 demonstrates the temperature and duration of various heat treatment process involved in each material treatment routine that is employed in the present study.

The initial hardening (austenitizing) and quenching process is similar for all the five material treatment sequences in this study. Both material specimens of the D2 and D3 tool steel are preheated in two stages of 650$^{\circ}C$ and 850$^{\circ}C$ for 20 min. This is followed by the hardening process carried out at 1020$^{\circ}C$ for 20 min. Subsequently, the hardened tool steel is quenched uniformly to room temperature using cold oil.

Tempering process is carried out at 520$^{\circ}C$ for 90 min with the help of a vacuum furnace. In the present study, tempering is performed after the quenching and cryogenic treatments for AQT and AQCT routine, respectively. Moreover, it is performed before and after cryogenic treatment in AQTCT routine. Furthermore, a double tempering is also carried for AQCTT routine.

The cryogenic treatment of the tool steels is performed using a microprocessor controlled cryogenic unit which uses liquid nitrogen as a medium. The material is cryotreated at 185$^{\circ}C$ with a soaking time of 12Hr. The temperature of nitrogen bath is lowered gradually (3-4$^{\circ}C$) to avoid thermal shocks. The cryotreated specimens are allowed to warm-up in an insulated enclosure for 16-24 Hr to reach the room temperature.

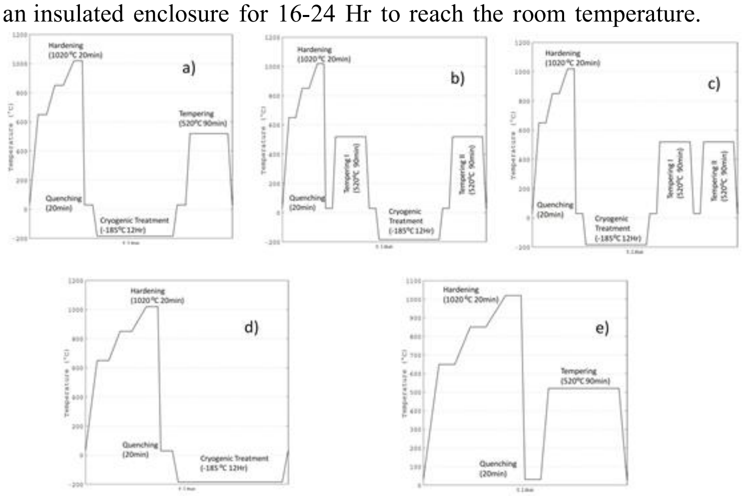

Figure 1: Time-Temperature diagram for a) AQCT, b) AQTCT, c) AQCTT, d) AQC, and e) AQT processroutines illustrates the heat treatment employed for AISI D2 and D3 tool steel.

2.3. Hardness Measurement

The hardness test on the samples from each group was carried out using the *Vickers Hardness Tester* (Model-MVI PC, Make-FIE). This test was performed by applying the load of 10 kgf with the dwell time of 10 sec, and measurements were an average of three random indentations.

2.4. Wear Measurement

To investigate the wear characteristics of the specimens, pin-on-disc wear test machine (Ducom: TR- 20LE-PHM-400) was used. The test is carried out in accordance to ASTM G99-05 by sliding the stationary specimens of diameter 10 mm and length 30 mm for 60 min against a rotating disc. The rotating disc had a diameter of 165 mm with a hardness of 61 HRC. The normal load of 6kg and 10kg was applied with the constant sliding velocity of 1 *m/s* and 2 *m/s* at room temperature. The wear measurements were repeated three times to evaluate the average of wear characteristics. Moreover, a new, circular, abrasive disc was used during the testing of each specimen. After each test, the samples and circular disc were cleaned in an acetone bath to remove surface contaminants.

2.5 Microstructure Visualization

Microstructure analysis was carried by *inverted optical microscope* (Make-CARL ZEISS Germany, Model-AL350) at the magnification of 450X. The specimens of diameter 10 mm and length 10mm are prepared, and were first surface levelled on an endless emery belt (80/0) paper. Furthermore, samples were subjected to polishing on emery paper (grit 240, 400, 600, 800 and 1000) so as to make surface free from scratches. The polished specimen were then lapped on a polishing machine with velvet cloth, and periodic application of alumina suspension. Moreover, these polished specimens were then etched using 3% Nital and 5% Picral solution for microstructural examination. The freshly prepared etchant *Nital* of composition approximately 3*ml* Nitric acid with 100*ml* ethyl alcohol and Picral of composition 5*g* picric acid in 100*ml* ethanol, was used for revealing micro constituents of tool steel. Microstructures were then recorded by image analyzer system. The *Energy Dispersive Spectroscopy* (EDS) analysis has been carried out to establish the presence of fine carbides in the specimens. These carbides are divided in two categories i.e *primary carbides* (size > 5 microns) and *secondary carbides* (size within 1 micron to 5 microns).

III. RESULTS AND DISCUSSION

3.1. Microstructural Features

The microstructural features of AISI D2 tool steel when subjected to various heat treatment sequences is demonstrated in Fig. 2. AQT sequence provides 55% retained austenite whereas, AQC has 95% *unstable* austenite. As AQC is not subjected to tempering the untampered martensite proportion is greater, as compared to AQT which demonstrates a 10% untempered martensite. AQT demonstrates irregular distribution of carbides having large globular (9μ) and nodular (11 4μ) shapes. On the other hand, deep cryogenic treatment in AQC provides more uniform distribution of carbides having 7% proportion. Similarly, AQCT sequence as in Fig. 2a also provides uniform distribution of carbides. However, single tempering carried out after cryogenic treatment increases the carbide content to 10%, and reduces the untempered martensite to 2%. Moreover, tempering treatment also reduced the retained austenite content to 20%. Howbeit, the carbide size is observed to increase after tempering. Furthermore, the double tempering treatment after cryogenic processing (AQCTT) is observed to increase the retained austenite content to 50%. However, the content of secondary carbides increases to 12% after performing AQCTT sequence. If tempering is carried out before and after the cryotreatment, as in AQTCT (Fig. 2b), the content of retained austenite remains similar to AQCTT (50%). Moreover, the secondary carbide and untampered martensite content also reduces to 7% and 4%, respectively. Therefore, cryotreating D2 tool steel can provide better transformation of retained austenite to martensite. Moreover, tempering cycle after cryotreatment is a effective way to increase the carbide content.

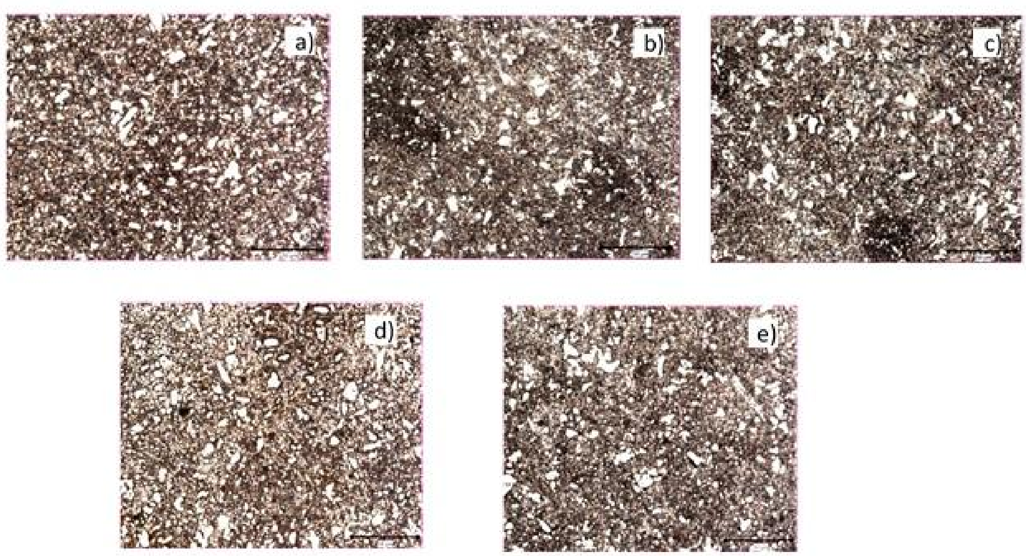

Figure 2: Microstructures of AISI D2 steel subjected to a) AQCT, b) AQTCT, c) AQCTT, d) AQC, and e) AQT process routines.

The microstructural features of AISI D3 tool steel when subjected to various heat treatment sequences is demonstrated in Fig. 3. Comparison between AQC and AQT process routines shows that, cruogenic treatment uniformly distributes the carbides. Moreover, AQT demonstrates large nodular secondary carbides (3 6μ) having its content of 20%; whereas, AQC demonstrates globular and erratic large secondary carbides having content of 4%. On the other hand AQT demonstrates 20% retained austenite whereas, AQC provides 95% of *unstable* austenite. Furthermore, the untempered martensite content is greater in AQC as compared to AQT sequence. Tempering D3 tool steel after cryogenic treatment provides complete transformation of retained austenite, as observed in sequence AQCT, AQCTT and AQTCT. However, sequence involving double tempering (AQTCT and AQCTT) reduces the carbide content to 10% as compared single tempering sequence (AQCT) where carbide content is 15%. Moreover, the untempered martensite content is maximum in AQCTT sequence as compared to AQTCT (5%) and AQCT (2%).

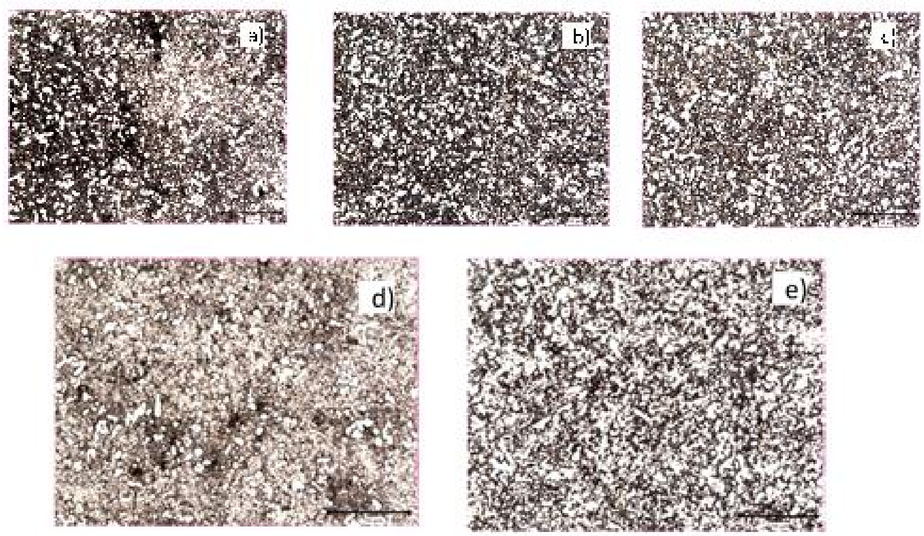

Figure 3: Microstructures of AISI D3 steel subjected to a) AQCT, b) AQTCT, c) AQCTT, d) AQC, and e) AQT process routines.

3.2 Wear Characteristics

The wear characteristics such as wear volume, wear rate, and wear resistance are analysed for AISI D2 and D3 tool steels in the present study. Fig. 4 demonstrates the wear volume (mm^3) of D2 tool steel for various heat treatment routines when subjected to different normal load and sliding velocities. It is observed from the comparison of AQT and other routines involving cryotreatment that, the cryogenic treatment reduces the wear volume of the D2 tool steel. The sequence AQCT demonstrates the least wear as compared tothe other cryotreated sequences (AQCTT and AQTCT) as most (80%) of the retained austenite was transformed. Moreover, there occurs a transformation of retained austenite to martensite at the surface even at the static loads of wear test as stated by Cardosoet al. (2020). This transformed martensite combines with the existing secondary carbidesto reduce wear (Cardoso et al. (2020)). It is to be noted that AQCT has higher content of secondary carbides as compared to AQCTT and AQTCT.

Fig. 5 demonstrates the wear volume (mm^3) of D3 tool steel for various heat treatment routines when subjected to different normal load and sliding velocities. Similar to D2 steel, cryogenic treatment is also observed to reduce the wear in D3 tool steel by complete trans- formation of retained austenite. Moreover, the sequence AQCT is also found effective inreducing the wear in D3 tool steel. This is owing to the complete transformation of retained austenite, and ~ 15% secondary carbide content. This provides the tool steel subjected to AQCT routine more hardness as compared to AQCTT (~ 10% carbide content) andAQTCT (~ 10% carbide content), as will be discussed further in Section 3.3.

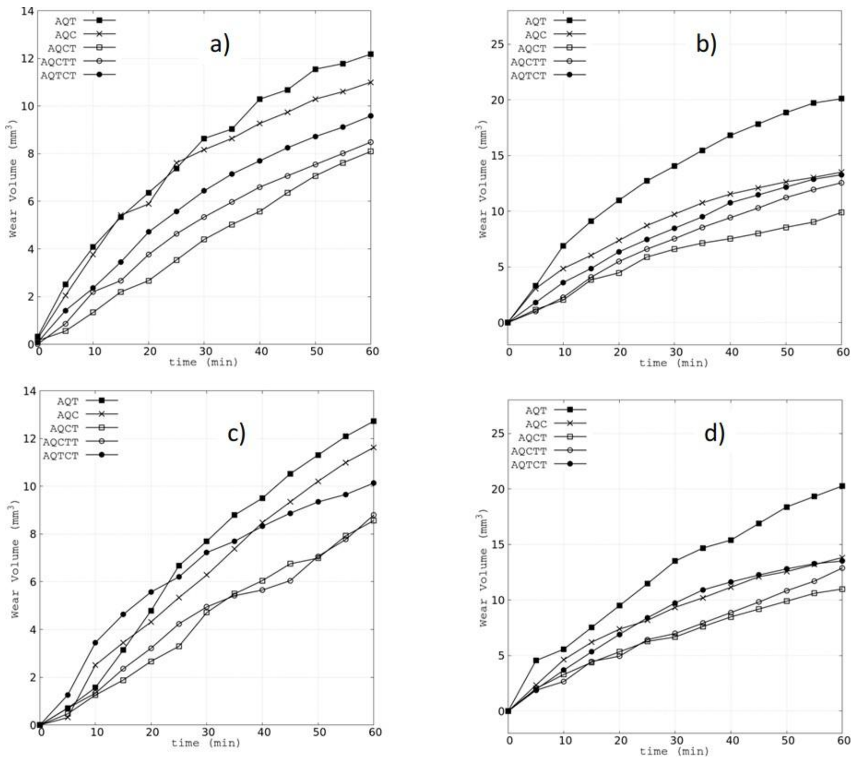

Figure 4: Wear volume (mm^3) of AISI D2 steel subjected to normal load and sliding velocity of a) 6*kg* and 1*m/s*, b) 6*kg* and 2*m/s*, c) 10*kg* and 1*m/s*, and d) 10*kg* and 2*m/s* during the wear test.

Fig. 6 and Fig. 7 depict the wear rate (mm^3/m) and wear resistance for AISI D2 and D3 tool steel when subjected to various normal load and sliding velocities during the wear test.

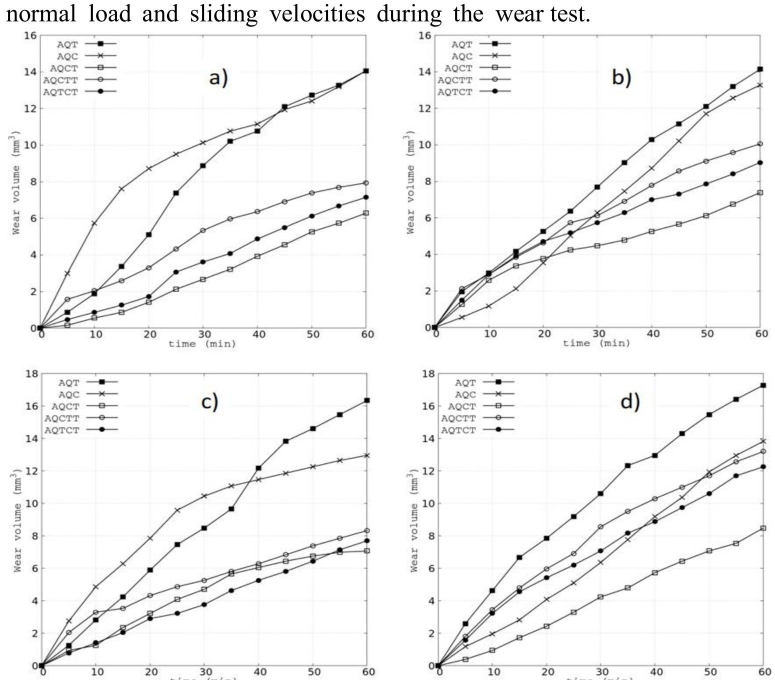

Figure 5: Wear volume (mm^3) of AISI D3 steel subjected to normal load and sliding velocity of a) 6kg and 1m/s, b) 6kg and 2m/s, c) 10kg and 1m/s, and d) 10kg and 2m/s during the wear test.

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The results of wear resistance and wear rate support the earlier discussion made on wear volume. Thus, it is observed that the AQCT sequence improves the wear resistance of AISI D2 steel by 31% to 93% as compared to the traditional AQT sequence of heat treatment. On the other hand, AQCT is also observed to be effective in improving wear resistance and reducing wear rate in AISI D3 tool steel by 72% to 111%, as compared to AQT. it also demonstrated that, the double tempering treatment after cryoprocessing, and tempering before and after cryoprocessing deteriorates the wear resistance and increases the wear rate in both D2 and D3 tool steel. However, cryoprocessing in general improves the wear characteristics as compared to traditional AQT process in the tool steel materials presently under consideration

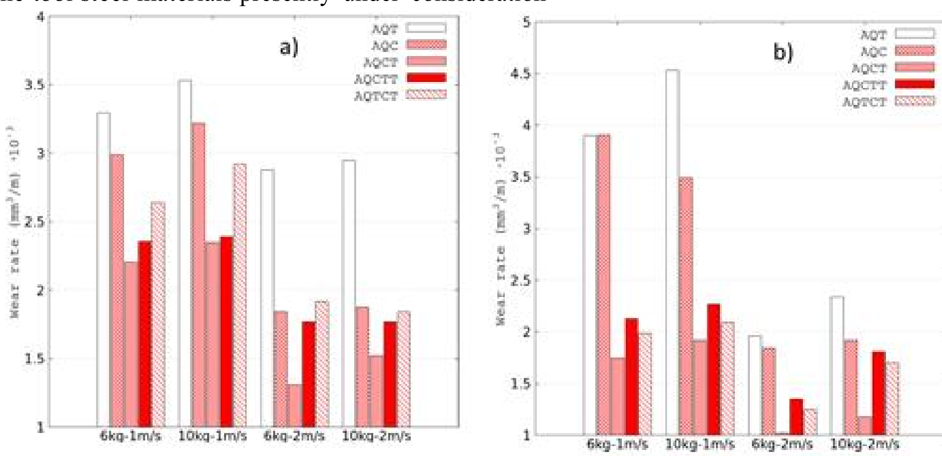

Figure 6: Wear rate (mm^3/m) for various normal loads and sliding velocity during the wear test of a) AISI D2 and b) AISI D3 tool steel

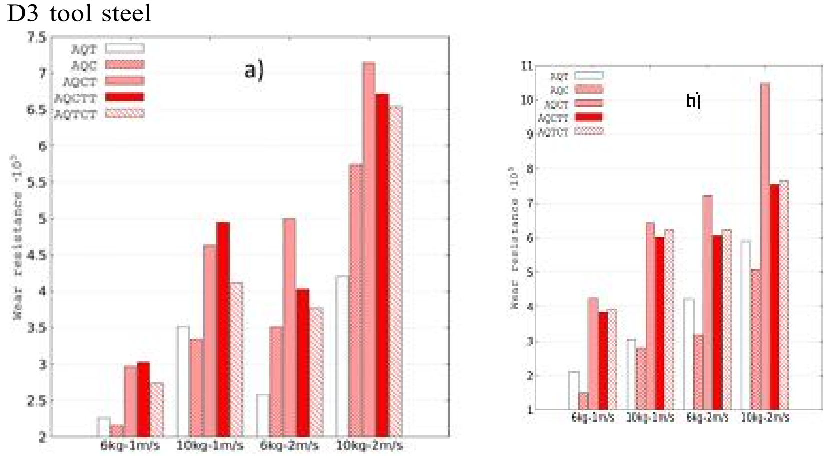

Figure 7: Wear resistance for various normal loads and sliding velocity during the wear test of a) AISI D2 and b) AISI D3 tool steel.

3.3. Hardness

Fig. 8 demonstrates the hardness test results for both D2 and D3 tool steels subjected to various heat treatment sequences. It is observed that the cryogenic treatment improves the hardness level of D2 tool upto 2% to 15% as compared to traditional process AQT. Moreover, improvement in hardness of 1% to 41% is observed in D3 tool steel over AQT. Moreover, cryogenic treatment is more effective in reducing the amount of austenite and increase the number of fine secondary carbides precipitate, which can increase the dispersion strengthening effect, which leads to an improved hardness. Moreover, the decreased grain size after cryotreatment also contributes to the increase in hardness level. The grain size of martensite is smaller than that of the austenite, and

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cryogenic treatment can produce more martensite, which implies a fine grain strengthening effect. Hence, the hardness of AQC, AQCT, AQCTT and AQTCT is higher than that of the AQT samples.

The hardness of AQC samples of both D2 and D3 tool steel is the maximum due to the untempered structure. However, such material is more brittle due to the presence of more untempered martensite which is observed in the microstructure. Hence, tempering becomes a necessity to reduce the brittleness by scarifying some hardness and to relieve the internal stresses. During tempering, martensite rejects carbon in the form of finely divided carbide phases. The results into a fine dispersion of carbides in the *α-iron* matrix.

Thus, the present study demonstrates that, the single tempering process after cryotreatment (AQCT) is effective for maintaining the hardness level and to reduce the retained austenite as compared to other cryotreatment routines considered in this study such as AQC, AQTCT, and AQCTT.

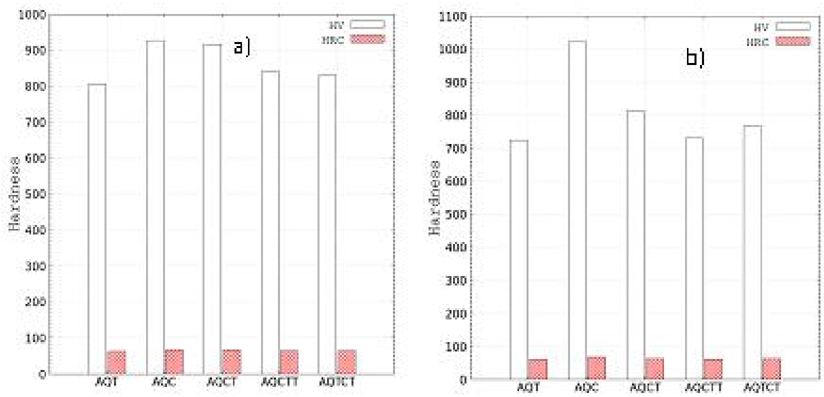

Figure 8: Hardness measurement of a) AISI D2 and b) AISI D3 tool steel using *Vikers* (HV) and *Rockwell* (HRC) hardness test.

IV. CONCLUSION

In the present study, the effect of deep cryogenic treatment on AISI D2 and D3 tool steel in terms of wear characteristics, hardness, and microstructural features is studied. Moreover, the effect of various tempering sequences before and after the cryotreatment is also analysed. It is demonstrated that cryogenic treatment on AISI D2 and D3 tool steel reduces the retained austenite and increase the fine secondary carbide content in the material. Moreover, these characteristics improve the material properties such as wear resistance and hardness, which are essential for tool steel. It is also depicted that the material properties can be further improved by carrying out the single tempering process (AQCT) after cryoprocessing. AQCT routine demonstrates the better transformation of the retained austenite, improved wear resistance and hardness in both D2 and D3 tool steel compared to AQTCT, AQC and AQCTT routines. Furthermore, if tempering is carried out before and after the cryoprocessing (AQTCT), the retained austenite content increases for D2 tool steel, and carbide content decreases. On the other hand, double tempering after cryoprocessing (AQCTT) demonstrates the similar results for D2 steel. However, the order of tempering in the heat treatment cycle does not affect the transformation of the retained austenite in D3 tool steel.

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