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Effect of Microstructure Parameters on Tensile Toughness of Tool Steel after Deep Cryogenic Treatment

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Microstructure of an alloy has a significant effect on mechanical properties. Deep cryogenic treatment extends life of tool steels because of microstructure changes. In this research, effects of microstructural parameters were studied on tensile toughness of a medium carbon-low alloy tool steel. The results showed that the maximum population density of sub-micron sized secondary carbide was obtained after 36 h of soaking time. Also, amount of secondary carbides increased with soaking or tempering times from 2.18 vol% to 12.87 vol%. In addition, high population density and high content of secondary carbides were responsible for tensile toughness enhancement. Therefore, the best results (12-35% improvement in tensile toughness) were obtained for a specimen, which underwent a full treatment cycle consisting of heating, water quenching, soaking at -196ºC for 36 or 48 h and tempering at 200ºC for 1 or 2 h, respectively.

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NOMENCLATURE

DCT = Deep Cryogenic Treatment
PC = Primary Carbide
PD = Population Density
SC = Secondary Carbide
SEM = Scanning Electron Microscope

1. Introduction

In DCT of tool steels, the microstructure mainly consisting of PC and SC is tempered in a martensite matrix.

DCT of AISI D2 high carbon-alloy tool steels causes 10% increase in hardness and wear resistance increases by 370% (wear condition: normal load of 49 N and sliding velocity of 2 m/s).[1] This improvement in properties is due to increased amount and PD of SCs.

When quenching at lower austenitizing temperature, toughness obtained for Cr8-type cold work die steel by DCT can be higher than the conventional treatment.[2] They[2] considered eight sets of specimens at different austenitizing temperature (1140 and 1100ºC) and soaking time (4 and 1 h). Also, tempering (560ºC for 2 h) after and/or before DCT (-196ºC for 8 h) improved impact toughness of H13 hot work tool steel (by about 20%) compared with conventional heat treatment.[3]

For cold work Cr8Mo2SiV and Cr12MoV tool steel specimens, toughness was reduced to one-third[4] in DCT compared to the standard specimens. Only three sets of specimens were soaked at -196ºC for 1, 2 and 24 h and tempered at 210ºC for 2 h. For 815M17 steel, elongation was nearly zero.[5] They[5] performed only one test and their soaking times at cryogenic temperature of -196ºC and tempering temperature of 150ºC were 24 h and 1.5 h, respectively. For 18NiCrMo5 steel, elongation was negligible at 1%. Their soaking times at -196ºC was 1 and 2 h), tempering durations at 180ºC was 2 h and number of set of tests was only 2.[6] Tempering (500 to 600ºC for 1 h) after DCT (-196ºC for 1 h) decreases fracture toughness of AISI M2 tool steel with compared to conventional heat treatment.[7]

In,[2-7] effects of DCT on toughness of high alloy tool steels were studied but microstructure was not studied at all.

The goal of this work was to investigate microstructure effects on tensile toughness of 1.2542 medium carbon-low alloy tool steel and evaluate it using SEM. The tool steel studied by this research contained about 0.5% carbon and 3.7% alloy elements, which made significant difference in microstructures of the tool steels between earlier researchs[1-7] and the present study.

Taquchi Design of Experiment (DOE) method was used to identify and optimize the important parameters in the subzero treatment of 18% Cr martensitic stainless steel.[8] Also, DOE method was used to identify important parameters of DCT of powder metallurgical AISI D2 steel.[9] In addition, austenitizing time was an effective parameter in DCT of D6 tool steel, especially at high load (180 N) and high sliding velocity (0.20 m/s); but, it did not influence the retained austenite.[10] Therefore, these three studies[8-10] investigated eight factors, namely austenitizing, soaking and tempering temperatures, austenitizing, soaking and tempering times and finally heating and cooling rates. In this research, tempering temperature (200ºC), DCT temperature (-196ºC), cooling rate (1.3ºC/min), heating rate (5.5ºC/min) and austenitizing temperature and time (900ºC for 60 min) were kept constant. Thus, tempering and soaking times would be the only parameters which could affect the microstructure.

2. Materials and Methods

Table 1 presents chemical analysis of 1.2542 tool steel used in this study. The temperature-time history for the DCTed specimens is depicted in Fig. 1(a) and the flow chart of experimental procedure is illustrated in Fig. 1(b).

The DCT work followed a procedure outlined in Fig. 1(b), using a programmable cryogenic processor. Considering the required tempering temperature of 200ºC, the cryogenic processor provided an added facility for the in situ tempering of the DCT specimens at this tempering temperature for the required time durations. The time from end of the hardening to the beginning of DCT was 17 min. However, the DCT specimens were tempered in situ, which resulted in a minimum time gap between DCT and tempering steps as 2 min.

The specimens had to be machined to the required sizes prior to their treatment, which was necessary as the increased hardness and wear resistance of metal specimens after the DCT would render their machining extremely difficult and very costly to perform. Preparation of the specimens was carried out using a CNC Machine.

The specimens were given codes for easy identification during and after the experiment. The coding procedure was as follows:

(a) Cryogenically treated specimens (water quenched): The first two digits of the code for a cryogenically treated specimen indicated soaking time (in hour) at -196ºC. The last digit indicated tempering time (in h) at 200ºC. Therefore, code 361, for example, indicated a cryogenically treated specimen: soaked at -196ºC for 36 h and tempered at 200ºC for 1 h.

(b) Hardened and tempered specimens (no DCT): Water quenched specimen 002 was tempered at 200ºC, but no DCT was performed.

The uniaxial tensile test (F-ΔL) was carried out on a standard specimen, 5 mm in diameter and 50 mm parallel length, at room temperature according to ISO 6892-1.[11] The strain rate was 0.00166 S^{-1}. Each test was repeated three times and the average value was obtained. The test results are presented in Table 2. Tensile toughness is the amount of work per unit volume that can be done on the material without causing it to rupture.[12] Tensile toughness is considered to be the total area under stress-strain curve.[12] It is calculated and listed in Table 2.

The SEM specimens were 15 mm in length and 12 mm in diameter. To calculate each phase size and content, at least five SEM images

Table 1 Chemical analysis of the 1.2542 tool steel (weight %)

Element	(%)	Element	(%)	Element	(%)	Element	(%)
C	0.48	S	0.02	W	1.57	Cr	1.12
Mn	0.34	Fe	95.41	Si	1.00	P	0.06

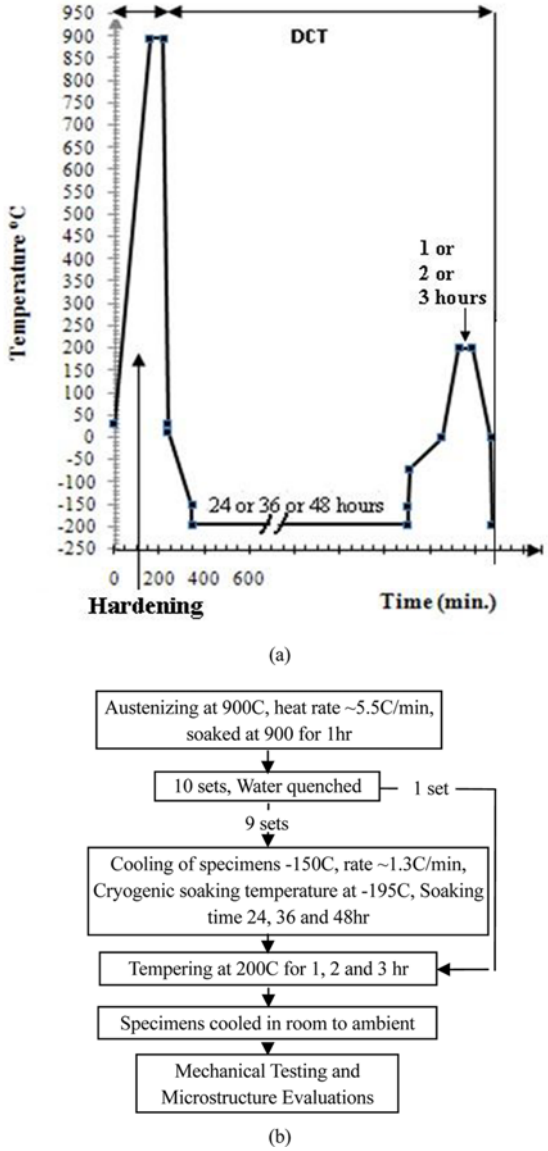

Fig. 1 Cryogenic treatment (a) cycle (b) flow chart

Table 2 Results of tensile tests at room temperature for 1.2542 tool steel

Specimen No.	σ_{uts} (MPa)	e_f (%)	Tensile toughness (MJ)[12,13] $U_T = 2\times\sigma_{uts}\times e_f / 3$
002	2229±65	5.5±1.5	81.7±0.65
241	2279±21	4.75±0.75	72.2±0.65
242	2265±31	2±1.0	30.2±0.65
243	2137±53	6±1.5	85.5±0.65
361	2268±65	7±1.5	105.9±0.65
362	2201±65	5±1.5	73.4±0.65
363	2245±65	5±1.5	74.8±0.65
481	2244±64	6.2±0.3	92.8±0.65
482	2206±65	7.5±1.5	110.3±0.65
483	2249±28	6.2±0.8	93.0±0.65

Table 3 Content, size and PD of PC and SC

Specimen No.	PC Content 0.4±0.2	SC Content ±10%	PC length Average (min to max) (μm)	SC diameter Average (min to max) (μm)	PC PD (mm^{-2}) ±5%	SC PD (mm^{-2}) ±2%
002	0.42	1.8	0.60 (0.3to1.5)	0.35 (0.13to0.5)	64000	200000
241	0.42	2.2	0.50 (0.3to1)	0.22 (0.065to0.5)	63000	660000
242	0.47	2.4	0.55 (0.3to1)	0.23 (0.065to0.5)	65000	630000
243	0.37	3.8	0.70 (0.6to0.9)	0.28 (0.065to0.7)	60000	600000
361	0.57	4.6	0.65 (0.5to0.8)	0.30 (0.065to1)	64000	894000
362	0.60	7.0	0.70 (0.4to1.7)	0.32 (0.065to0.7)	63000	750000
363	0.34	9.0	0.70 (0.4to1.5)	0.33 (0.065to0.6)	62000	726000
481	0.35	10.0	0.70 (0.3to1.4)	0.44 (0.065to0.7)	65000	707000
482	0.25	12.6	0.70 (0.4to1.4)	0.45 (0.065to1)	62000	650000
483	0.24	12.8	0.70 (0.4to2)	0.46 (0.065to1)	65000	620000

were taken from five different scanned regions in $\times 10^4$ magnification. The reported values were average ones. The SC and PC size and content were measured using phase analysis software, OLYSIA m3. It was calibrated for images with 2048×1536 pixels. Table 3 presents content, size and PD of PC and SC. The precipitation of SC initiates during cryogenic soaking itself, rather it takes place during tempering treatment. Carbide which is not dissolved after austenitization treatment is termed as PC.

3. Results and Discussion

SEM micrograph in Fig. 2 reveals that SCs are fine and distributed fairly uniformly. Also, SCs are sub-micron sized.

As shown in Fig. 3, content of the PC was approximately constant (about 0.4±0.2 V%) in different specimens. Also, average size of the PC remained almost constant with increase in soaking or tempering times (especially, for long soaking times of, for example, 48 h). Also, PD of the PC was constant for all the specimens, which was because specification of PC is dependent on the austenitization treatment parameters which were kept constant in this research. As a whole, the PC particles were in the range of 0.3 to 2 μm.

Fig. 4 shows that content of the SC increased with soaking or tempering times from 2.18 V% to 12.87 V%. As shown in Fig. 4(a), increase in tempering time resulted in slight precipitation of SC (50% in average) due to the diffusion phenomenon. Also, as shown in Fig. 4(b), increase in soaking time resulted in more precipitation of SC (500% in average) due to the defect population density in phase matrix that increased with increase in the soaking time, which facilitated precipitation of SC.

At constant tempering temperature of 210°C, constant tempering time of 2 h for different soaking times, the amount of the PC precipitated in AISI D2 was constant (about 7 V%).[1] However, they[1] reported that increase in DCT soaking time for a DCTed specimen increased SC from 7.5 V% to 10 V%.

As demonstrated in Fig. 4(a), with increase in the tempering time, size of the SC had a slight increase (10% in average) due to the diffusion phenomenon. Also, in the specimens with similar soaking times, PD of the SC was slightly reduced (10% in average) with increase in the tempering time.

As demonstrated in Fig. 4(b), with increase in soaking time, size of the SC also increased (90% in average).

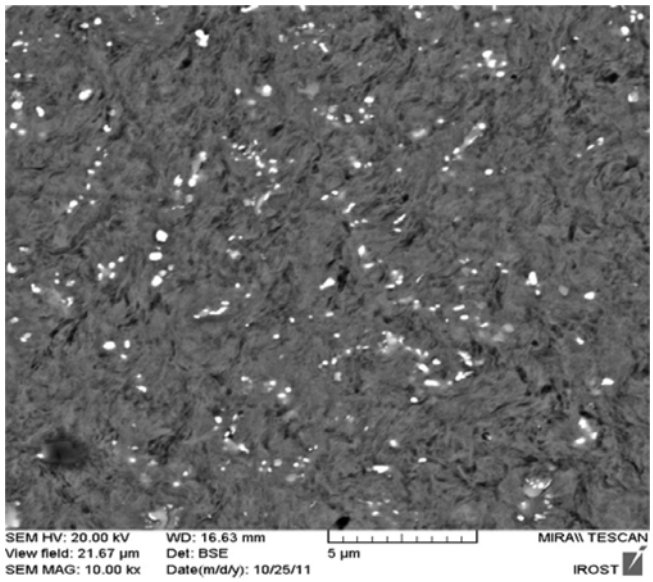

Fig. 2 SEM micrograph of specimen 241, 10^4

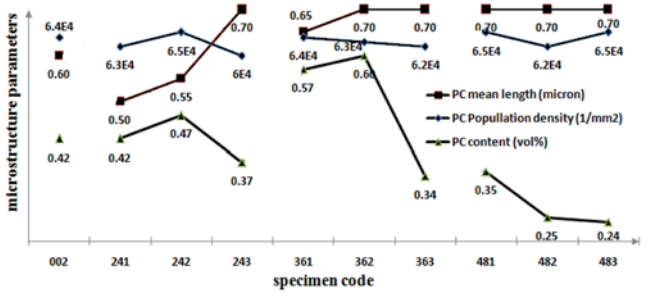

Fig. 3 Size, PD and content of PC, tempering time trends

The comparative assessment of characteristics of SCs of specimen 361 with specimen 002 made it possible to infer that contents of SCs increased by 125% (Fig. 4) and PD of SCs increased by 350% (Fig. 4) were associated with reduction in their mean diameter by 30% (Fig. 4). These results evidently indicated that DCT refined the SCs.

The above finding confirmed the findings by Das et al.,[1] indicating that contents of SCs of DCTed specimens with respect to no DCT specimens increased by approximately 17% and PD of SCs increased

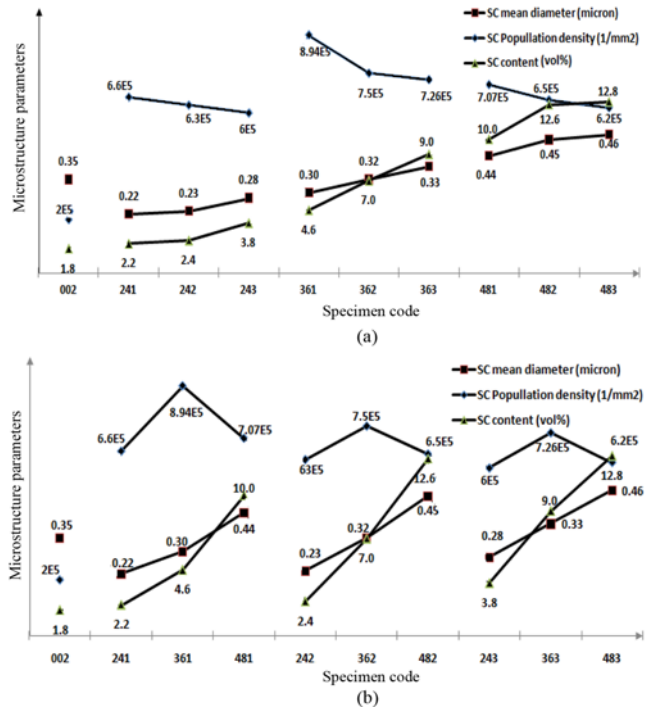

Fig. 4 Size, PD and content of SC (a) tempering time trends (b) soaking time trends

by almost 225%, which was associated with reduction in their mean diameter by approximately 29%.[1]

As shown in Fig. 4(b), at specific tempering time, maximum PD of the SC occurred in specimens with 36 h of soaking time. Accordingly, specimens with 1 h of tempering time showed maximum PD of the SC. The above finding confirmed the findings by Das et al.,[1] indicating that, at constant tempering temperature of 210°C, constant tempering time of 2 h and for 0 to 84 h DCT soaking times, size of SC increased by about 30% and maximum PD of the SC (200% and 300% increase for large and small, respectively) occurred in specimens with 36 h of soaking time after DCT of AISI D2. The reason for this observed phenomenon is explained below:

Volume of martensite contracts and their lattice parameters shrank, which could reinforce precipitation of carbon atoms to decorate the nearest dislocation. The decorated regions increased due to precipitation of more carbon atoms.[4,14-16] At constant tempering time, with an increase in soaking time, content of martensite contracts or lattice parameters shrank, which could reinforce precipitation of carbon atoms to decorate the nearest defect. The decorated regions increased due to precipitation of more carbon atoms. With increase in the precipitation of carbon atoms, the region in which precipitation of carbon atoms increased also developed and the particles became bigger. Also, at constant tempering time, with an increase in the soaking time, the defect population density increased due to the difference in the coefficient of thermal expansion of the phases. With increase in the defect population density, the region in which the defect population density increased also developed. As long as these regions did not meet (namely, for 36 h of soaking time), population density of the SC also increased. In the case of meeting these developing regions, the particles grew in size; however, population density decreased, which occurred at 48 h of soaking time.

Table 2 presents about 12-35% improvement in tensile toughness for specimens 361 and 482 compared to specimen 002. This improvement in properties could be contributed by pull-out toughening mechanism of SC.[17,18] Micro-voids were formed in the matrix by de-cohesion of secondary carbides while primary carbides were broken.[17,18]

The above finding confirmed the findings in,[2,3] which indicated that DCT could improve toughness of steels.

The difference between results of the present work and those reported in[4-7] could be due to the point that fewer number of set of tests performed at cryogenic and tempering temperatures in the work[4-7] was not sufficient for determining suitable microstructure.

Fracture toughness of DCT specimens was slightly lower (by about 7%) compared to that of conventionally treated AISI D2 tool steel.[17,18] In the first view, AISI D2 was high alloy tool steel containing about 13% alloy elements; conversely, low alloy tool steel contained about 3.7% alloy elements in this study, which made big difference of microstructure between their research[17,18] and the present study. In the second view, they studied fracture toughness; conversely, tensile toughness was considered in this study. Also, impact toughness of DCT treated specimens was lower when compared to that of conventionally treated AISI 4340 steel.[19] According to chemical composition of AISI 4340 steel, M_f temperature was -194°C; so, retained austenite must completely transform to martensite at DCT temperature (-196°C); but, they reported 4.2 V% retained austenite after DCT.

As shown in Fig. 4, content of the SCs in specimen 002 (1.8%) was smaller than those in specimens 361 and 482 (4.6% and 12.6%, respectively); also, PD of the SCs in specimen 002 (160000 mm^{-2}) was smaller than those in specimens 361 and 482 (894000 mm^{-2} and 650000 mm^{-2}, respectively). As demonstrated in Fig. 5, the specimens which had high PD and high content of SCs had high tensile toughness as well (specimens 361 and 482). But, its reverse was not correct (specimens 362, 363, 481and 483); i.e., in these specimens, high PD and high content of SCs were two effective parameters for tensile toughness enhancement.

For all ten sets, Table 2 shows that yield and tensile strengths of the specimens had no relative change because, as shown in Table 3 or Fig. 4, very fine SCs or sub-micron sized SC (produced in DCT as almost 0.1 μm) could easily go around at high stress levels.

In this research, specimens 361 and 482 had higher tensile toughness than conventionally treated ones, which showed that increase of DCT duration needed increase of tempering time to obtain high tensile toughness. For example, for 36 h of soaking time at -196°C, 1 h tempering time was needed to obtain the maximum tensile toughness. For 48 h of soaking time at -196°C, the corresponding tempering time was 2 h, which meant that, with increase in the tempering time, a longer soaking time was required for obtaining the maximum tensile toughness.

4. Conclusions

In this research, 10 sets of 1.2542 tool steel specimens were DCT treated at -196°C for 24, 36 and 48 h and were tempered at 200°C for 1, 2 and 3 h.

1) The increased soaking time led to constant increase of content of SCs; however, population density of SCs increased until 36 h of soaking time while being reduced after this period.

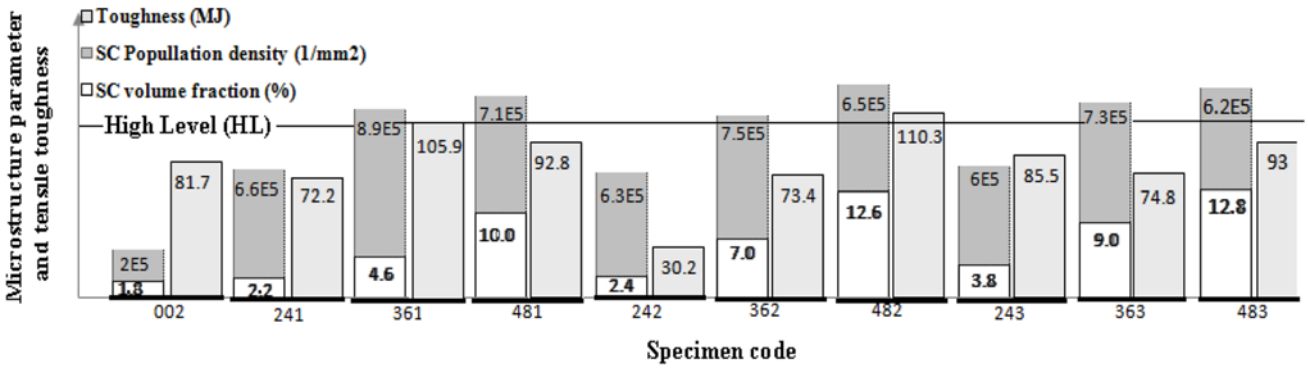

Fig. 5 Tensile toughness of specimens, PD and content of SCs

2) In these specimens, high PD and high content of SCs were two effective parameters for tensile toughness enhancement. Conventionally higher content of SCs reduces toughness in general. But, in this research, SCs were sub-micron sized and distributed uniformly.

3) It was observed that maximum tensile toughness could be obtained by simultaneously increasing tempering duration and soaking time. However, if the reduction in production time and cost are of primary concern, less tempering time and soaking time would seem to be a more feasible option. For example, in this case, specimen 361 was a more feasible option.

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