

The effect of deep cryogenic treatment on mechanical properties of 80CrMo12 5 tool steel

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Abstract

Cryogenic treatment can be used as a supplemental treatment that is performed on some tool steels between quenching and tempering as an effective method for decreasing retained austenite and increasing wear resistance. In this research, the effect of deep cryogenic treatment (DCT) on dimensional stability and mechanical properties of 80CrMo12 5 tool steel was investigated. The martensitic transformation start and finish temperatures were also studied using dilatometry tests. The results show that the start and finish temperatures of the martensitic transformation are 254°C and - 87°C, respectively. The hardness increases (untempered) by 3 HRC after DCT. Thus, to decrease or eliminate the amount of retained austenite, cryogenic treatment is necessary. In DCT, impact energy and hardness at all tempering temperatures decreases and increases, respectively. Tempering transformations investigation using dilatometry tests, verified that the DCT increases dimensional stability. This is attributed to the transformation of retained austenite to martensite.

Keywords: Martensitic transformation, Impact energy, Hardness, Deep cryogenic treatment, Dimensional stability.

1. Introduction

In tool steels, the presence of high carbon and high alloy elements is said to influence temperature characteristics of martensite and eventually leads to lowering the start (M_s) and finish (M_f) of martensite transformation temperatures. The latter lies well below the ambient temperature for commercial tool steels. Therefore, conventional hardening treatment of these steels fails to convert a considerable amount of austenite into martensite. The retained austenite, γ_{re} , is soft, and it decreases desirable properties such as hardness and wear resistance ¹⁾. Furthermore, γ_{re} is prone to transformation into martensite at the service conditions of tool steels. The untempered freshly formed martensite is very brittle and hence undesirable. In addition, transformation of austenite to martensite is associated with approximately 4% volume expansion that leads to the components distortion and dimensional changes ^{1,2)}, and even failure in extreme working conditions. If the working temperature lies between 200-350°C, there is a probability of converting the retained austenite to a mixture of ferrite

and cementite. This transformation results in volume expansion and dimensional changes ²⁾. Therefore, one of the major challenges in the heat treatment of tool steels is minimizing or eliminating the amount of γ_{re} . The tempering process reduces the amount of retained austenite. However, this process has its own shortcomings as it leads to excessive softening of the matrix and coarsening of any residual carbide, resulting in lower hardness, mechanical strength and lower wear resistance ^{1,2)}. Alternatively, cryogenic treatment is a supplementary heat treatment that is performed on some tool steels before tempering as an effective method for decreasing retained austenite and increasing wear life ^{2,3)}. Decades ago, the Swiss watch-makers used cryogenic treatment to improve wear resistance and stability of their watch components by keeping these components in the icy mountains and under layers of snow ⁴⁾. Cryogenic treatment is generally classified as either so-called "Shallow Cryogenic Treatment", (SCT), at temperatures down to about -80°C or "Deep Cryogenic Treatment", (DCT), at liquid nitrogen temperatures of -196°C. The greatest improvement in desirable properties using cryogenic treatment will be achieved if the treatment takes place quickly after the quenching process and before tempering ⁵⁾. In the deep cryogenic temperatures, finely dispersed eta (η) carbides are precipitated as well as eliminating retained austenite ⁶⁾. Some researchers have pointed out better carbide distribution and increased carbide particles by using deep cryogenic treatment ⁷⁾.

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Improvement in hardness⁸⁻¹⁰, wear resistance^{11,12}, bending strength¹³, dimensional stability¹⁴, fatigue resistance¹⁰ and fracture toughness¹³ have also been reported by researchers using cryogenic treatment. In this research, the effect of deep cryogenic treatment on the dimensional stability, via phase transformations studies during tempering, and mechanical properties such as hardness and impact energy of the 80CrMo12.5 tool steel is investigated. The martensite transformation start and finish temperatures were also studied using dilatometry tests.

2. Experimental procedure

The chemical composition of the 80CrMo12.5 tool steel used in the present study is 0.8C, 0.85Si, 0.25Mn, 3.06Cr, 0.25Mo (Wt.%). A BAHR 805 A/D dilatometer was used to investigate dimensional stability and to determine martensitic transformation start and finish temperatures. The dilatometry test samples were made according to the SEP1681 standard (dia 4mm × 10mm). Conventional heat treatment (CHT) was used as a reference to evaluate the effect of deep cryogenic treatment on mechanical properties and dimensional stability. The cycle of the CHT was: preheating at 620°C for 20 min and austenitizing at 925°C for 20 min in an argon protection chamber followed by oil quenching.

Then, tempering was carried out at 6 different temperatures: 150°C, 250°C, 350°C, 450°C, 550°C and 650°C, in an argon atmosphere for 3 h. The DCT consisted of cooling to -196°C after oil-quenching, holding the specimens at this temperature for 48 hours and heating them slowly to room temperature. In order to avoid thermal shock and micro crack formation in rapid cooling and heating processes, the cooling and heating rates were set at 1°C/min.

After the deep cryogenic treatment, the specimens were tempered in similar temperatures as in conventional heat treatment. Schematic diagram of the research methodology is shown in Fig.1. The phase analyses were conducted by an X-ray diffractometer (Philips PW3710 diffractometer) with Cu K α radiation. The volume fraction of retained austenite was estimated in accordance with ASTM E975-00¹⁵. HRC evaluation of the specimens were carried out using EMCO 4U750 test apparatus. In order to measure the impact energy, Charpy impact samples were prepared according to the ASTM E23¹⁶ and the impact energy was measured by Zwick 5111 apparatus.

Field emission scanning electron microscope (FSEM JEOL2010) was employed after etching the samples in a 100ml ethanol, 100 ml HCl, and 5gr CuCl₂ solution. For investigation of dimensional stability of the DCT and CHT specimens, the specimens were tempered in a dilatometer at a temperature range of 25-640°C with a heating rate of 200°C/h. Using the dilatometer built-in software, the changes in length, tempering temperature

and the linear thermal expansion coefficient, α , were extracted.

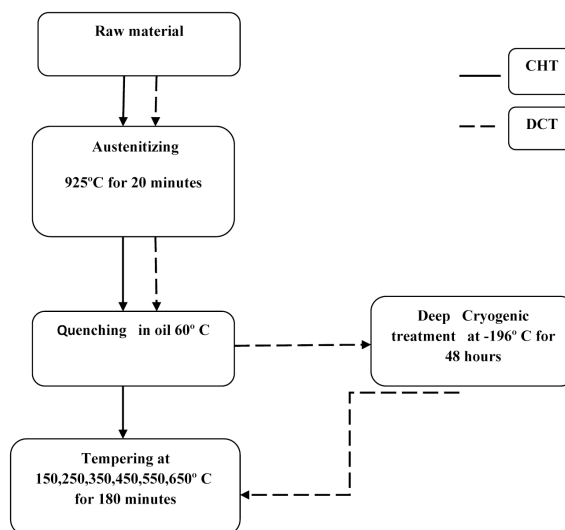


Fig.1. Research methodology.

3. Results and discussion

3.1. Characterization of martensite transformation

In the dilatometry tests, the length and temperature variations of the specimens were printed during cooling and heating processes simultaneously in order to study phase transformations. According to Fig. 2a, after heating the specimen, the length of the specimen increases due to thermal expansion; however, when a phase transformation of α + cementite to α + γ occurs, the specimen length decreases. The start and finish temperatures of this transition are 797°C (AC_1) and 851°C (AC_3), respectively.

By increasing the temperature, the specimen length increases again. Holding the specimen in 925°C does not cause its length to change. Upon quenching, the length starts to decrease due to thermal contraction. When the martensitic transformation begins, the length suddenly increases at the temperature called M_s .

During cooling, the length increases continuously until the transformation finishes (M_f) at -87°C. The martensitic transformation region is shown in Fig. 2b. The M_s and M_f temperatures of the steel were 254°C and -87°C, respectively. If transformation of this steel had been interrupted by holding at some temperatures between M_s and M_f , martensite transformation might not have occurred¹. However, a certain percentage of soft austenite is retained when the specimen is quenched to room temperature. As a result, to decrease or eliminate the amount of retained austenite, the DCT process is necessary.

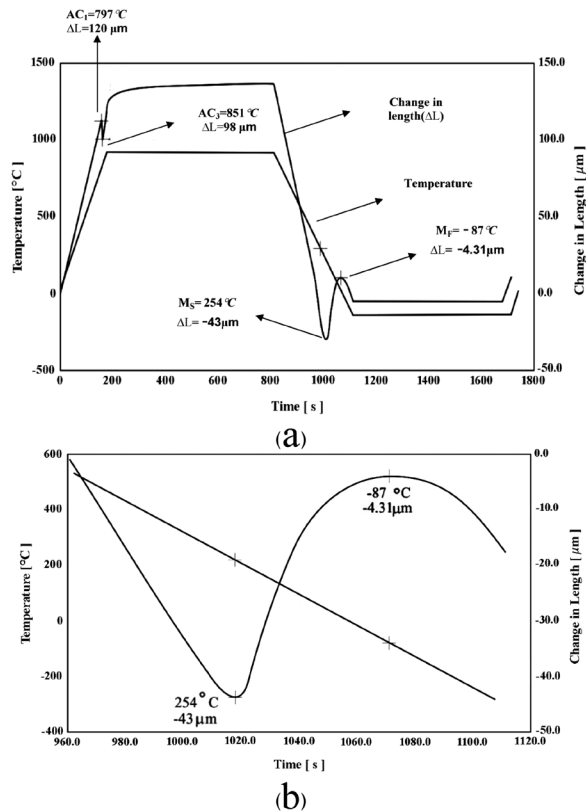


Fig. 2. (a) Heat treatment cycle for determining M_s and M_f , (b) martensitic transformation region.

3. 2. Dimensional stability

To study the effect of deep cryogenic treatment on dimensional stability, the specimens (CHT, DCT) were tempered in a dilatometer at a temperature range of 25-640°C. In Fig. 3, the relation between the tempering temperature and α (linear thermal expansion coefficient) is shown for both types of specimens. It is clear that the value of α at all temperatures is lower in the DCT process than for the CHT process. Therefore, deep cryogenic treatment samples have higher dimensional stability than conventional heat treatment samples. Usually α rises with increasing temperature (normal thermal expansion). But deviation from normal thermal expansion is due to the phase transformations that occurred during the tempering process. This phase transformation in the steel occurs in five different regions (Fig. 3) as follows^{17,18}:

Region 1: α increases because of normal thermal expansion (pure thermal effect).

Region 2: α decreases due to redistribution of carbon atoms by: (a) segregation to lattice defect, (b) clustering of carbon atoms.

Region 3: α decreases due to precipitation of (η/ϵ) transition carbides.

Region 4: α increases due to decomposition of retained austenite to ferrite and cementite.

Region 5: α decreases due to conversion of the segregated carbon and transition carbides to cementite and alloy carbides.

As shown in region 4 of Fig. 3 (at a temperature range of 190-420°C), α is lower in the DCT specimen compared to the CHT specimen because of the small amount of retained austenite. Retained austenite in the CHT specimen transforms to a mixture of ferrite and cementite in a temperature range of 190-420°C. Also, the retained austenite is unstable and transforms into martensite at the service conditions of tool steels. The freshly formed martensite is untempered. So, it is brittle and also accompanied by 4% volume expansion which causes dimensional instability. Deep cryogenic treatment induces the continued transformation of retained austenite to martensite below room temperature. In the DCT samples, because of the small amount of retained austenite, less change in length and α is observed; therefore, the dimensional stability is much higher than that in the CHT samples.

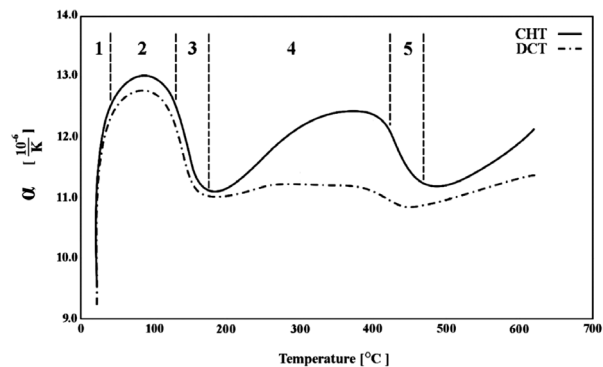


Fig.3. The effect of tempering temperature on linear thermal expansion coefficient (α) for DCT and CHT treatments.

3.3. Impact energy

The effect of tempering temperature on impact energy for CHT and DCT specimens is illustrated in Fig. 4. As can be seen, the lower impact energy after DCT can be due to an increase in the amount of martensite in comparison to CHT. Fig.4 also shows that impact energy decreases in CHT by increasing the tempering temperature to 400°C. This behavior is known as "tempered martensite embrittlement" which is due to austenite decomposition to ferrite and interlath films of M₃C carbides at prior austenitic grain boundaries¹. By increasing the tempering temperature from 350°C, impact energy increases in both DCT and CHT samples. Tempered martensite embrittlement is not seen in DCT due to small amount of retained austenite. Reduction in impact energy after DCT in comparison to CHT is in excellent agreement with other researchers works^{10, 19}.

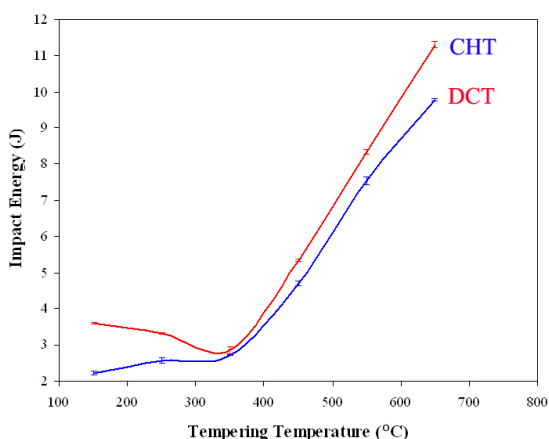


Fig.4. The effect of tempering temperature on impact energy after DCT and CHT treatments.

3.4. Hardness

A comparison of hardness in CHT and DCT samples (untempered) indicates that the DCT samples exhibit 4.6% higher hardness than the CHT samples (Table 1). The increase in hardness by cryogenic treatment, compared to conventional heat treatment, is attributed to the complete transformation of soft retained austenite to hard martensite. The amount of retained austenite has been estimated by XRD technique whose detailed procedure has been published earlier²⁰. The microstructure of the 80CrMo12 5 tool steel consists of austenite, martensite, Cr₃C, and Si₃C₃. The X-ray diffraction (XRD) analysis of the samples shows that performing the deep cryogenic on the 80CrMo12 5 tool steel eliminates the retained austenite (Fig.5). The retained austenite percentage was 12 % after the conventional heat treatment, but it was totally eliminated in the DCT48 sample (Table 1).

Table1. The retained austenite and hardness of the 80CrMo12 5 tool steel after heat treatment.

| Sample | Volume fraction of retained austenite (%) | Hardness (HRC) |
|--------|---|----------------|
| CHT | 12% | 64±0.1 |
| DCT | 1% > | 67±0.3 |

The effect of tempering temperature on hardness for CHT and DCT specimens is shown in Fig.6. Hardness in DCT specimens was greater than that in CHT specimens at all tempering temperatures. This improvement can be attributed to the increase in the amount of martensite in DCT and precipitation of very tiny secondary carbides in the tempering process after deep cryogenic treatment. This higher amount and more homogenized distribution of the carbides are due to the increase in density of dislocations and vacancies during deep cryogenic treatment. The supersaturation of dislocations and vacancies enhance the diffusion coefficient of carbon. Carbon atoms are driven to segregate dislocations by the interaction of stress field around dislocations with interstitial carbon atoms. This phenomenon induces the precipitation of tiny carbides during tempering after deep cryogenic treatment²¹. The microstructures of both CHT and DCT specimens, tempered at 150°C, are shown in Fig. 7. It is clear that finer and more homogeneous distributed carbide particles are achieved in DCT. However upon increasing the tempering temperature, hardness decreases in both treatment types. This is attributed to excessive softening of matrix and coarsening of carbide particles. It should be noted that other researchers have reported increase in hardness during cryogenic treatment too^{10, 11, 19}.

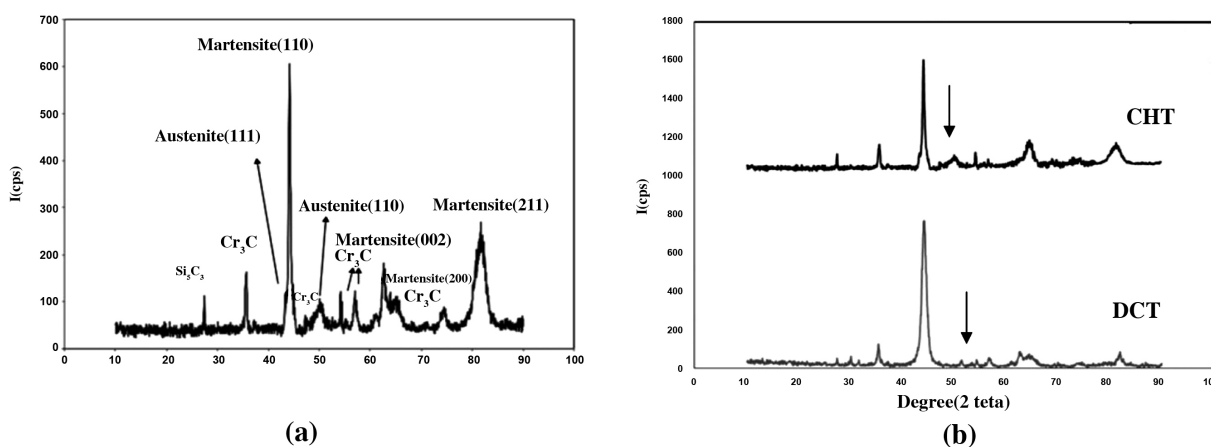


Fig. 5. XRD pattern of the 80CrMo12 5 tool steel: (a) after quenching and (b) after conventional and deep cryogenic treatment.

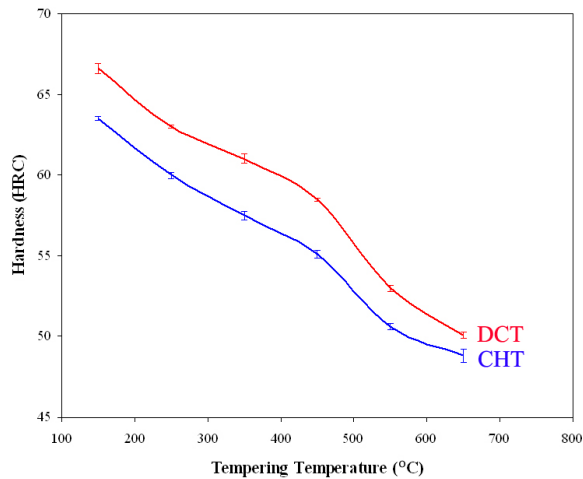


Fig. 6. Hardness of the samples for different tempering temperatures after DCT and CHT treatments.

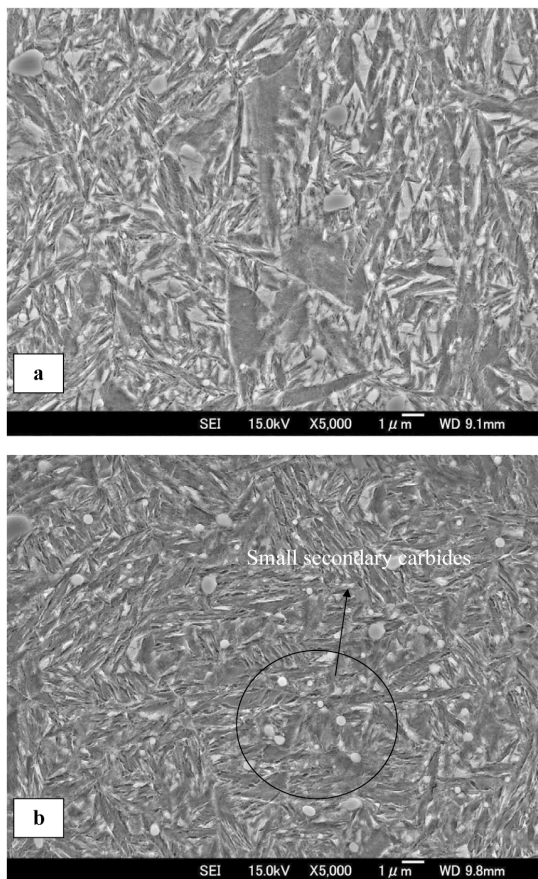


Fig. 7. FSEM micrographs of 80CrMo12.5 tool steel after a) CHT, b) DCT. Note that the carbides in (b) are smaller and homogeneously distributed than (a).

4. Conclusion

- Martensitic transformation start and finish temperatures in 80CrMo12.5 steel were 254°C and -87°C, respectively; therefore, using DCT is necessary to minimize or eliminate the amount of retained austenite.
- In DCT, the amount of retained austenite decreases and precipitation of tiny carbides occurs; therefore, hardness in DCT is higher than that in CHT at all tempering temperatures.
- Dimensional stability is higher in DCT compared to that in CHT. This is attributed to a decrease in the amount of retained austenite and also the amount of α .
- In DCT, impact energy is lower than CHT due to higher amount of martensite. Furthermore, tempered martensite embrittlement is seen in CHT at a temperature range of 190-420°C which is due to retained austenite decomposition, but tempered martensite embrittlement is not seen in DCT due to small amount of retained austenite.

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