Influence of Cryogenic Treatment and Tempering on AISI H13 Hot Work Tool Steel

Melika ÖZER¹, Kemal DAVUT²,³, Alpay ÖZER⁴
¹Gazi University, Faculty of Technology, Metallurgical and Materials Engineering Dept., Teknikokullar/Ankara/Turkey
²Atılım University, Metal Forming Center of Excellence, İncek/Gölbaşı/Ankara/Turkey
³Atılım University, Metallurgical and Materials Engineering Dept., İncek/Gölbaşı/Ankara/Turkey
⁴Gazi University, Technical Sciences Vocational School of Higher Education, Ostim/Ankara/Turkey

Abstract

In this study, the effects of cryogenic and deep cryogenic treatments on microstructure, hardness and toughness of H13 steel were investigated. For this purpose, H13 steel samples were subjected to conventional quenching heat treatment. Afterwards, one group of those samples was cryogenically treated at -76 °C and another group was subjected to deep cryogenic treatment at -196 °C. Finally, all sample groups were tempered at 560 °C for 3 hours. The microstructures of the samples were examined via optical and scanning electron microscopes; moreover their hardness and toughness’s were determined. Microstructures of the samples were composed of evenly distributed carbides on a martensitic matrix. The results indicate that tempered samples exhibit lower toughness values. More importantly, cryogenic treatment slightly improves the impact toughness after tempering; and also makes carbide distribution more homogeneous.

1. Introduction

Chromium hot-work tool steels, including the popular and readily available H13 steel, are most widely used for forging and die casting applications. Although its hot hardness is lower than tungsten and molybdenum hot work tool steels, higher toughness and shock resistance of chromium H-type steels makes them preferable for most hot-work operations [1-4]. After conventional heat treatment practices, the microstructure of H13 tool steel should be composed of fine and evenly distributed carbides in a matrix of tempered martensite and some retained austenite [4]. Since retained austenite is a metastable phase, it may transform into martensite during service [3, 5]. This transformation can cause micro-cracks and distortion. [4, 6-8]. The amount and distribution of carbides and retained austenite should be optimized depending on the requirements of the application. Cryogenic treatment is one way of controlling the final microstructure [9-11], applied after cooling to room temperature in order to decrease the retained austenite fraction and also obtain more homogeneous distribution of carbides [4,12,13]. These microstructural changes can enhance dimensional stability and wear resistance properties. The present study aims at investigating the influence of cryogenic treatment on the microstructure and mechanical properties of H13 hot work tool steel

2. Experimental

In this study, a commercial AISI H13 hot work tool steel, whose chemical composition is given in Table 1, was used. The standard composition range of the H13 tool is steel is also given in Table 1. The present material conforms the standard, in terms of chemical composition. Conventional austenitizing, quenching and tempering treatments were applied to the H13 steel samples. The cryogenic and deep cryogenic treatments were applied to the H13 steel samples. The cryogenic and deep cryogenic treatments were conducted at 0.5°C/min. Cooling and heating rates. Table 2 shows the details of the heat treatments applied and the specimen coding. All of the samples were austenitized at 1040°C for 30 minutes followed by oil quenching. The cryogenic treatment was performed at -76°C and deep cryogenic treatment at -196°C for 8 hours. A group of samples were tempered at 560°C for 3 hours. One specimen from each group was prepared for metallographic examination and then examined under optical and scanning electron microscopes. The average hardness of the matrix of the specimens was determined by taking 10 Vickers micro-hardness measurements from randomly selected regions of the as-polished surfaces of the specimens. For this purpose Zwick / Roell ZHV 10 micro-hardness tester was used with a load of 19.61 N at a test speed of 25 mm/min. The impact toughness of the samples were determined via Charpy V-notch tests.

Table 1. The specimens’ chemical composition and standard composition range (in wt.%) of AISI H13 steel

<table>
<thead>
<tr>
<th>AISI H13</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Mo</th>
<th>V</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>measured</td>
<td>0.39</td>
<td>0.97</td>
<td>0.37</td>
<td>0.020</td>
<td>0.001</td>
<td>4.90</td>
<td>1.25</td>
<td>0.94</td>
<td>Bal.</td>
</tr>
<tr>
<td>standard range</td>
<td>0.37-0.43</td>
<td>0.90-1.20</td>
<td>0.30-0.50</td>
<td>0.025 max.</td>
<td>0.005 max.</td>
<td>4.80-5.50</td>
<td>1.20-1.50</td>
<td>0.90-1.10</td>
<td></td>
</tr>
</tbody>
</table>
Table 2. Sample notations and heat treatment details

<table>
<thead>
<tr>
<th>Sample notation</th>
<th>Treatment sequence</th>
<th>Austenitizing and quenching</th>
<th>Cryogenic treatment temperature and duration</th>
<th>Tempering temperature and duration</th>
</tr>
</thead>
<tbody>
<tr>
<td>HT-1</td>
<td>conventional quenching</td>
<td>1040 °C / 30 min. oil quench</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>HT-2</td>
<td>conventional quenching, tempering</td>
<td>1040 °C / 30 min. oil quench</td>
<td>---</td>
<td>560 °C 3 hr.</td>
</tr>
<tr>
<td>HT-3</td>
<td>conventional quenching, cryogenic treatment</td>
<td>1040 °C / 30 min. oil quench</td>
<td>-76°C 8 hr.</td>
<td>---</td>
</tr>
<tr>
<td>HT-4</td>
<td>conventional quenching, cryogenic treatment</td>
<td>1040 °C / 30 min. oil quench</td>
<td>-76 °C 8 hr.</td>
<td>560 °C 3 hr.</td>
</tr>
<tr>
<td>HT-5</td>
<td>conventional quenching, deep cryogenic treatment</td>
<td>1040 °C / 30 min. oil quench</td>
<td>-196 °C 8 hr.</td>
<td>---</td>
</tr>
<tr>
<td>HT-6</td>
<td>conventional quenching, deep cryogenic treatment</td>
<td>1040 °C / 30 min. oil quench</td>
<td>-196 °C 8 hr.</td>
<td>560 °C 3 hr.</td>
</tr>
</tbody>
</table>

3. Results and Discussions

Figure 1 shows optical micrographs of the H13 steel samples subjected to different heat treatments. The microstructures of HT-1, HT-3, and HT-5 coded samples are composed of primary carbides on a martensitic matrix. The volume fraction of primary carbides of the specimens HT-3 and HT-5 are higher than HT-1 sample which was not subjected to any cryogenic treatment. The carbide fraction is the highest for HT-5 specimen, which has been subjected to cryogenic treatment at -196 °C. The microstructures of those untempered HT-1, HT-3 and HT-5 specimens show that as the cryogenic treatment temperature decreases the ratio of carbides increases.

The specimens HT-2, HT-4, and HT-6 have been tempered at 560°C for 3 hours. This tempering not only changed the matrix into tempered martensite, but also caused formation of secondary carbides in addition to the existing primary carbides. Therefore, the tempered specimens contain much higher fraction of carbides. Moreover, the cryogenically treated samples contain much higher fraction of carbides. Similar to the untempered specimens, as the cryogenic treatment temperature decreases the ratio of carbides increases. The microstructures also reveal that the cryogenically treated HT-4 and HT-6 samples have much finer carbides which have distributed more homogeneously than the conventionally treated samples.

Koneshlou et al. [4] reported that tempering after deep cryogenic treatment increases the volume fraction of carbides, whereas refines their size. This behavior was attributed to the transformation of retained austenite into martensite and precipitation of secondary carbides. Similar results were also reported by Perez et al. [11]. The microstructural changes observed in the present study agree well with those reported studies.

The micro-hardness test results of the specimens are given in Figure 2. Specimens HT-2, HT-4, and HT-6 exhibit lower hardness due to tempering treatment. The micro-hardness test results reflect mainly the matrix hardness, which decreases due to the tempering of the martensite. The cryogenic treatment also influences the softening during tempering. The cryogenically treated samples contain more martensite, and therefore they soften more rapidly upon tempering. On the other hand as-quenched HT-2 sample contains more retained austenite, which can transform into martensite during tempering. The un-tempered HT-1, HT-3, and HT-5 specimens have higher hardnesses as expected. HT-3 exhibits the highest hardness, which can be attributed to transformation of retained austenite into martensite and carbides. Nevertheless, the differences between HT-1, HT-3, and HT-5 are very small considering the standard deviations and experimental uncertainties. It should also be noted that the micro-hardness results reflect the hardness of martensitic matrix, which is mainly influenced from the carbon content.
Figure 1. Optical micrographs of the specimens taken at 1000x magnification under bright field illumination

Figure 2. Micro-hardness of the specimens

Figure 3. Charpy V-notch impact toughness test results of the specimens
Figure 3 shows the Charpy V-notch impact test results of the specimens. The impact toughness of HT-1, HT-3 and HT-5 specimens are very close. The tempered HT-2, HT-4 and HT-6 specimens, on the other hand show lower toughness. The tempering treatment increased the volume fraction of carbides. Moreover, it can also cause transformation of the tougher and more ductile retained austenite into martensite. HT-4 and HT-6 specimens, which have been cryogenically treated and then tempered exhibit higher toughness values than HT-2 specimen. When the starting structure is composed of almost fully martensite, then tempering will soften this matrix and hence improve the toughness as in the case of HT-4 and HT-6. Special care in tempering is required for the samples containing significant amounts of retained austenite, as it can transform into harder and brittle martensite during tempering.

4. Conclusions

The influence of cryogenic treatment on the microstructure and corresponding mechanical properties of H13 tool steel has been investigated. The following conclusions can be drawn:

1. The microstructure of conventionally treated HT-1, HT-3 and HT-5 coded specimens is composed of primary carbides on a martensitic matrix. Tempering causes formation of secondary carbides in addition to primary carbides and the tempered martensitic matrix. Cryogenically treated specimens were found to contain much finer and homogeneously distributed carbides than the ones treated conventionally.

2. The un-tempered specimens have insignificant differences in hardness, and cryogenic treatment does not change the hardness of martensitic matrix. On the other hand HT-2, HT-4 and HT-6 specimens exhibit lower hardness due to softening during tempering. The balance between softening of pre-existing martensite and formation of never martensite due to transformation of metastable austenite determines the hardness.

3. The cryogenic treatment also influences the tempering response. The lower the cryogenic treatment temperature the higher the toughness values after tempering.

5. References