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EFFECTS OF AUSTENITISATION TEMPERATURE AND MULTIPLE TEMPERING ON THE MICROSTRUCTURE AND IMPACT TOUGHNESS OF A 5 WT. % CR COLD WORK TOOL STEEL

Abstract

The microstructure and properties of a 5 wt.% Cr cold work tool steel were studied after austenitisation at 1020°C, 1050°C or 1075°C followed by single, double and triple tempering treatments at 525°C. The microstructures were investigated with scanning electron microscopy and X-ray diffraction and phase transformations were studied by dilatometry. Furthermore, hardness and Charpy un-notched and V-notched impact toughness testing was performed and results were correlated to observed microstructures.

With higher austenitisation temperature, the martensite and bainite start temperatures were lowered resulting in microstructures containing a higher volume fraction of retained austenite. Retained austenite transformed into martensite on cooling from the tempering temperature. Specimens that were austenitised at 1050°C or 1075°C and tempered twice contained fresh martensite. Applying a third tempering was therefore required to guarantee a fully tempered microstructure. The second tempering resulted in an increase of the un-notched impact energy while the third tempering did not have a pronounced effect.

A triple tempering procedure could be preferable when austenitising at high temperatures to avoid undesirable fresh martensite in the tool microstructure.

Keywords

Cold work tool steel, microstructure, martensite, retained austenite, toughness, X-ray diffraction, scanning electron microscopy, dilatometry.

1. Introduction

Tool steels are alloys of mainly iron, carbon, and carbide forming elements such as chromium, molybdenum, vanadium, and tungsten [1]. Processes that occur below 200°C are usually referred to as cold work processes, e.g. cutting, punching, rolling and pressing etc. [1, 2]. Steels that are used in tools for these applications are therefore called ‘cold work tool steels’.

The microstructure of high-carbon high-chromium cold work tool steels contains large undissolved carbides [1, 3]. Therefore, the steel has high hardness and high wear resistance but does not have enough toughness to withstand the common tool failure mechanism of cracking [2,

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Medium carbon cold work tool steels with 5 wt.% of chromium have lower amounts of undissolved carbides which reduce the risk of fracture initiation at inhomogeneities. These steels have high hardness and good toughness to cut, punch and form advanced high strength steels.

The microstructure of 5 wt.% Cr cold work tool steels contains some retained austenite after austenitisation. The steel is then tempered at secondary hardening temperatures, typically at 525°C, resulting in decreased amounts of retained austenite. Literature suggests that the retained austenite can transform to ferrite and carbides during holding at the tempering temperature [4, 5, 6] or to martensite [7, 8] or bainite on cooling [9]. Therefore, the resulting microstructure and properties can vary depending on the steel composition and heat treatment parameters. Hence, it is necessary to understand the retained austenite transformation to optimise the tempering treatments.

The present study investigates the phase transformations during austenitisation and multiple tempering of a 5 wt.% Cr cold work tool steel. The microstructure after the first, second and third tempering treatments are characterised and correlated to impact toughness of the steel. The phase transformations during austenitisation and tempering were studied with dilatometry. Microstructures were characterised with Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD). The results are discussed aiming at a better understanding of the influence of the microstructure on the impact toughness after multiple tempering treatments.

2. Experimental Procedure

2.1 Material

The cold work tool steel ‘Uddeholm Caldie’ was studied. The chemical composition of the steel is listed in Table 1.

Table 1 Chemical composition (wt. %) of the investigated steel.

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Mo</th>
<th>V</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.7</td>
<td>0.2</td>
<td>0.5</td>
<td>5.0</td>
<td>2.3</td>
<td>0.5</td>
</tr>
</tbody>
</table>

2.2 Heat treatments

Specimens with dimensions of 17x17x10 mm (l x b x h) were taken from the centre of a soft annealed bar and heat treated in a temperature controlled vacuum furnace. Heat treatments consisted of austenitisation at 1020°C, 1050°C or 1075°C followed by multiple tempering treatments at 525°C. Each tempering treatment was for 2 hours.

First, the specimens were austenitised for 30 minutes. They were then quenched with a cooling time of 300 s between 800°C and 500°C. Tempering treatments were performed in an electric oven followed by cooling to ambient temperature with a linear cooling rate of 2 °C/s. Single and double tempering treatments were carried out for all austenitisation temperatures. A triple tempering was only carried out on specimen austenitised at 1050°C. The heat treatments are shown in Table 2.
Table 2 Heat treatments.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Q_{20}</th>
<th>T_{20x1}</th>
<th>T_{20x2}</th>
<th>Q_{50}</th>
<th>T_{50x1}</th>
<th>T_{50x2}</th>
<th>T_{50x3}</th>
<th>Q_{75}</th>
<th>T_{75x1}</th>
<th>T_{75x2}</th>
</tr>
</thead>
<tbody>
<tr>
<td>Austenitisation temp. (°C)</td>
<td>1020</td>
<td>1020</td>
<td>1020</td>
<td>1050</td>
<td>1050</td>
<td>1050</td>
<td>1050</td>
<td>1075</td>
<td>1075</td>
<td>1075</td>
</tr>
<tr>
<td>Number of tempering treatments at 525 °C</td>
<td>0</td>
<td>1</td>
<td>2</td>
<td>0</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>0</td>
<td>1</td>
<td>2</td>
</tr>
</tbody>
</table>

2.3 Microstructural characterisation

Scanning Electron Microscopy (FEI Quanta 600F) was used for microstructural investigations. Specimen surfaces were prepared by grinding with abrasive paper of grit size 350, 500 and 1200 mesh and followed by polishing with 3 μm and 1 μm diamond suspensions. The as-quenched specimens were etched with 2% Nital for 1 minute. Tempered specimens were etched first in Picral for 1 s and then with 2% Nital for 5 s.

Surfaces of XRD specimens were prepared by electrolytic polishing with a Struers Tenupol. XRD (Seifert XRD-3003) was carried out with a current of 35 mA and a voltage of 40 kV with unfiltered Cr Kα radiation. The retained austenite content was estimated using the integrated intensities of (111)_γ, (200)_γ, (220)_γ peaks of austenite and (110)_α, (200)_α, (211)_α peaks of martensite/ferrite. The use of these numbers of peaks allows avoiding possible bias due to crystallographic texture [10]. The volume fraction of retained austenite was determined by performing a pseudo Voigt fitting of the x-ray peaks with Rayflex Version 2.408 (GE sensing & Inspection Technologies). The accuracy of measurements for retained austenite content is ± 2%.

Soft annealed cylindrical specimens with a diameter of 4 mm and a length of 10 mm were heat treated in a dilatometer (Dil 805 A/D, Baehr-Thermo). A thermocouple was welded at the middle of the longitudinal surface of the specimen and heat treatments were performed as described in section 2.2. Dilatometry data during isothermal tempering and cooling was used to construct two different types of plots:

i) Plots of relative length change of the specimen as a function of time during isothermal tempering at 525°C for two hours. These curves have been plotted together in one graph with a numberless y-axis for ease of comparison.

ii) Plots of length change against specimen temperature during cooling where transformation temperatures were evaluated by calculating the derivative of length change as a function of specimen temperature. The transformation start temperatures were defined as the initial deflection in the derivative (see Fig. 1).

Thermo-Calc, a software for thermodynamics was used to calculate the equilibrium chemical composition of austenite for the investigated steel. Uddeholms internal database ‘tooling 11’ was used to perform calculations.

Retained austenite, fresh martensite and bainite are referred to in the text with abbreviations as RA, FM and B, respectively.

2.4 Mechanical testing

Rockwell C hardness was measured with a Zwick/Roell ZHR hardness tester. Each hardness value is an average of five measurements.

The Charpy impact tests were carried out with an Amsler/Roell RKP 150 equipment at room temperature for un-notched and V-notched specimens with a dimension of 55 x 10 x 10 mm (l x b x h). These specimens were taken from the centre in transversal direction of the bar and
tested in radial direction. The impact energies presented in this paper are an average of five measurements.

3. Results

3.1 Thermodynamical calculations

The equilibrium composition of austenite for the investigated steel was calculated at 1020°C, 1050°C and 1075°C as shown in Table 3. It can be seen that the amount of alloying elements in the austenite increases at higher temperatures, i.e. C, Cr, Mo and V contents.

Table 3 The calculated chemical composition of austenite (wt.%) at 1020°C, 1050°C and 1075°C.

<table>
<thead>
<tr>
<th>Austenitisation temperature (°C)</th>
<th>C</th>
<th>Cr</th>
<th>Mo</th>
<th>V</th>
<th>Si</th>
<th>Mn</th>
</tr>
</thead>
<tbody>
<tr>
<td>1020</td>
<td>0.54</td>
<td>4.34</td>
<td>2.10</td>
<td>0.33</td>
<td>0.20</td>
<td>0.50</td>
</tr>
<tr>
<td>1050</td>
<td>0.61</td>
<td>4.62</td>
<td>2.23</td>
<td>0.40</td>
<td>0.20</td>
<td>0.50</td>
</tr>
<tr>
<td>1075</td>
<td>0.67</td>
<td>4.80</td>
<td>2.28</td>
<td>0.46</td>
<td>0.20</td>
<td>0.50</td>
</tr>
</tbody>
</table>

3.2 Dilatometry and XRD

3.2.1 Hardening

Dilatometry curves showing length change as a function of specimen temperature after austenitisation at temperatures of 1020°C, 1050°C or 1075°C are presented in Fig. 1. Bainite and martensite formation occurred during cooling and transformation start temperatures were identified from changes in the derivative of length (Fig.1a).

![Figure 1](image1.png)

Figure 1 a) Length change as a function of specimen temperature during quenching. Identification of Ms and Bs temperatures (arrows) using the derivative of length change (black line) is illustrated for the specimen austenitised at 1020°C. b) A section of Fig. 1a showing that the bainite and martensite start temperatures (arrows and dashed arrows, respectively) were lowered when austenitising at higher temperatures.
The martensite and bainite start temperatures decrease with higher austenitisation temperatures as presented in Fig. 1b and Table 4. The martensitic transformation did not reach its finish temperature for any of the austenitisation temperatures. XRD results showed that an increasing austenitisation temperature resulted in higher volume fractions of retained austenite. These results are presented in Table 4.

Table 4 Fraction of retained austenite and martensite and bainite start temperatures after austenitisation at 1020°C, 1050°C or 1075°C.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Fraction of RA (vol. %)</th>
<th>Martensite start temperature Ms (°C)</th>
<th>Bainite start temperature Bs (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Q20</td>
<td>19</td>
<td>180</td>
<td>230</td>
</tr>
<tr>
<td>Q50</td>
<td>24</td>
<td>150</td>
<td>200</td>
</tr>
<tr>
<td>Q75</td>
<td>28</td>
<td>140</td>
<td>190</td>
</tr>
</tbody>
</table>

3.2.2 Tempering

First tempering

Dilatometry curves for specimens that were austenitised at 1020°C, 1050°C or 1075°C are presented in Fig. 2a. They show a decrease in length change (contraction) for all specimens as a function of time during the first tempering treatment at 525°C. The total contraction for all specimens was similar, i.e. approximately 0.007%.

Figure 2 Dilatometry curves for the a) first and b) second tempering treatments showing specimen contraction. a) The contraction was 0.007% for all austenitisation temperatures. b) The contraction increased with austenitisation temperature and was 0.001%, 0.002% and 0.003% for specimens that were austenitised at 1020°C, 1050°C or 1075°C, respectively.

When cooling to ambient temperature after the first tempering retained austenite transformation occurred for all specimens. The transformation temperatures were evaluated and the fraction of retained austenite was measured at room temperature. These results are presented in Table 5. It can be noticed that a higher austenitisation temperature resulted in a decreased Ms temperature and higher amounts of retained austenite.
Second tempering

The contraction of specimens varied with the austenitisation temperature during the second tempering treatment, i.e. specimens with a higher austenitisation temperature showed a larger contraction as shown in Fig. 2b.

During cooling from the second tempering, there was observable retained austenite transformation only for specimens that were austenitised at 1050°C or 1075°C. The Ms temperatures are presented in Table 5. Notice that the Ms is higher than for first tempering. XRD measurements revealed that the microstructures did not contain significant amounts of retained austenite for any of the specimens after the second tempering treatment.

Table 5 Martensite start temperatures and the fraction of retained austenite after the first and second tempering treatments for specimens austenitised at 1020°C, 1050°C or 1075°C.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Fraction of RA (vol. %)</th>
<th>Martensite start temperature Ms (°C)</th>
<th>Sample</th>
<th>Fraction of RA (vol. %)</th>
<th>Martensite start temperature Ms (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T20x1</td>
<td>&lt;2</td>
<td>190</td>
<td>T20x2</td>
<td>&lt;2</td>
<td>-</td>
</tr>
<tr>
<td>T50x1</td>
<td>7</td>
<td>170</td>
<td>T50x2</td>
<td>&lt;2</td>
<td>180</td>
</tr>
<tr>
<td>T75x1</td>
<td>12</td>
<td>160</td>
<td>T75x2</td>
<td>&lt;2</td>
<td>180</td>
</tr>
</tbody>
</table>

3.3 Microstructures

The microstructure of the investigated steel after austenitisation at 1050°C was representative for all three austenitisation temperatures. The as-quenched microstructure after austenitisation at 1050°C is presented in Fig. 3a. It shows light and dark regions together with

Figure 3 SEM micrographs of microstructures. a) The as-quenched microstructure of the specimen austenitised at 1050°C showing blocky light regions and dark regions b) The microstructure after tempering at 525°C for two hours contains blocky light regions and regions with precipitation of carbides.
with undissolved carbides. Regions which were more heavily etched appeared dark while regions which were largely un-etched appeared light and had a blocky shape.

The microstructure after the first tempering is presented in Fig. 3b. It shows undissolved carbides, light blocky regions and dark regions showing precipitation of carbides.

The microstructures after the second and third tempering treatments are presented in figs. 4a and b, respectively. Both microstructures appear to be similar, i.e. consisting of undissolved carbides and dark regions with precipitated carbides. However, one difference in the appearance is the existence of blocky regions which were present only in the microstructure that was tempered twice (i.e. Fig. 4a).

3.4 Hardness and toughness

The hardness of the tempered specimens increased from 60 HRC to 61 HRC and 62 HRC when austenitised at 1020°C, 1050°C and 1075°C, respectively. There were no significant differences in hardness when comparing samples tempered one, two or three times.

The results from the impact toughness testing of Charpy un-notched and V-notched specimens which were austenitised at 1050°C and tempered at 525°C once, twice or three times are shown in Fig. 5. The impact energies for un-notched specimens had comparatively lower values after the first tempering. Specimens tempered two or three times showed higher impact energies. However, the difference in impact energies after the second and the third tempering treatments was very small.

Charpy impact toughness of V-notched specimens did not vary significantly with the number of tempering treatments. However, it is noticeable that the impact energies after the third tempering were very consistent showing a very small scatter.
Figure 5 Charpy un-notched and V-notched impact energies after one, two or three tempering treatments at 525°C for specimens austenitised at 1050°C. The un-notched impact toughness for two and three tempering treatments is higher than after tempering one time.

4 Discussion

4.1 Microstructure

The as-quenched microstructure consisted of etched regions and blocky largely un-etched regions (Fig. 3a). A rough estimation suggests that the amount of blocky regions in the as-quenched microstructure is higher than the fraction of retained austenite measured by XRD (compare Fig. 3a and Table 4). Therefore, it is reasonable to believe that all the blocky regions in the as-quenched microstructure were not retained austenite but that also some un-tempered martensite may appeared as blocky regions. One possible explanation could be that martensite with certain crystallographic orientations was etched more heavily than martensite with other orientations [11]. After the first tempering treatment the dark regions showed precipitation of small carbides which is a result of tempering of bainite and martensite. This supports the assumption that the etched regions in the as-quenched microstructure consists of bainite and martensite.

The amount of blocky regions in the microstructure after the first tempering was similar to the amount in the as-quenched condition. However the fraction of retained austenite was significantly lower (Table 5). For example, for the specimen austenitised at 1050°C, the retained austenite content decreased from 24% to 7% when tempering the first time. For the specimen tempered twice at 525°C, only a few blocky regions were found (Fig. 4a) while after the third tempering treatment, the blocky regions were completely gone (Fig. 4b). This suggests that the blocky regions in the tempered microstructure consisted of fresh martensite and retained austenite in varying proportions.

4.2 Effect of austenitisation temperature and tempering

It was seen that a higher austenitisation temperature dissolved more alloying elements in the austenite (Table 3). Therefore the martensite and bainite start and finish temperatures were lowered and the resulting microstructures contained higher amounts of retained austenite (Table 4). The higher alloying content in the matrix also resulted in a higher volume fraction of tempering carbides thereby increasing the hardness slightly.
It is clear for this steel that the retained austenite transforms into martensite (partly or fully) during cooling from the tempering temperature. However, literature suggests that the contraction during holding at the tempering temperature was due to the precipitation of carbides in martensite (and bainite) and in the retained austenite. The precipitation of carbides, consuming carbide forming alloying elements, preceded the transformation of retained austenite into martensite on cooling [4, 5, 6]. As a consequence the Ms temperature increased with the number of tempering treatments confirming that the retained austenite was becoming less alloyed.

A rough approximation of the fresh martensite content in the microstructure after austenitisation and multiple tempering is provided in Table 6 combining information from Tables 4 and 5.

### Table 6 Fraction of fresh martensite for heat treated specimens.

<table>
<thead>
<tr>
<th>Sample</th>
<th>As-quenched FM + B (vol. %)</th>
<th>First tempering</th>
<th>Second tempering</th>
<th>Third tempering</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Sample</td>
<td>FM (vol. %)</td>
<td>Sample</td>
<td>FM (vol. %)</td>
</tr>
<tr>
<td>Q_{50}</td>
<td>81 T_{20x1}</td>
<td>19 T_{20x2}</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Q_{50}</td>
<td>76 T_{50x1}</td>
<td>17 T_{50x2}</td>
<td>7 T_{50x3}</td>
<td></td>
</tr>
<tr>
<td>Q_{58}</td>
<td>72 T_{75x1}</td>
<td>16 T_{75x2}</td>
<td>-</td>
<td></td>
</tr>
</tbody>
</table>

The as-quenched samples all contained various proportions of un-tempered martensite, bainite and retained austenite (in total 100 %) resulting in a comparatively large contraction during the first tempering (Fig. 2a). During the second tempering the contraction was smaller mainly due to the presence of lower volume fractions of retained austenite and fresh martensite (Fig. 2b). This is in line with the observation that a higher austenitisation temperature resulted in a larger contraction during the second tempering. For example the contraction was 0.003 % for the highest austenitisation temperature with 28 % FM + RA compared to 0.001 % contraction for the lowest austenitisation temperature with 19 % FM and no retained austenite. What proportion of the contraction that can be attributed to martensite, bainite and retained austenite cannot be estimated from the present study.

### 4.3 Toughness

The lower impact un-notched and V-notch toughness after the first tempering is due to the presence of approximately 17 % of un-tempered martensite in the microstructure (Fig. 5 and Table 6). The impact toughness significantly increased after the second tempering due to the lower amount of fresh martensite (7 %). However, a third tempering treatment did not have a significant effect on the level of toughness although the scatter was reduced for Charpy-V specimens. This observation leads to the conclusion that small volume fractions of fresh martensite are not drastically affecting the un-notched impact toughness of the steel but can have an effect on scatter in impact energies of V-notch specimens. It can be assumed that the negligible effect of a third tempering, once the amount of un-tempered martensite was decreased to low levels, is due to that toughness to a large extent is governed by the undissolved carbides.
Conclusions

A 5 wt. % Cr cold work tool steel was characterised after austenitisation at 1020°C, 1050°C or 1075°C and multiple tempering at 525°C. The following was concluded:

- A higher austenitisation temperature led to a higher alloying content in the matrix and lower Ms and Bs temperatures. Thus, a higher volume fraction of retained austenite was formed.
- Retained austenite transforms into martensite on cooling from the tempering temperature.
- The presence of significant amounts of fresh martensite after the first tempering resulted in low un-notched impact toughness. A second tempering reduced the content of fresh martensite and increased the toughness.
- A triple tempering procedure can be needed when austenitising at high temperatures to decrease the level of fresh martensite in the tool microstructure to ensure sufficient toughness.

References