INFLUENCE OF THE RETAINED AUSTENITE VOLUME FRACTION ON THE PHASE TRANSFORMATIONS DURING TEMPERING IN HIGH CARBON ALLOY STEEL

Rafał DZIURKA, Kamil GÓRECKI, Mateusz KOPYŚCIAŃSKI, Łukasz Frocisz

AGH University of Science and Technology, Faculty of Metals Engineering and Industrial Computer Science, Cracow, Poland, EU, dziurka@agh.edu.pl

Abstract

The hereby work presents the influence of the retained austenite volume fraction on the phase transformations during tempering. Two different austenitising temperature 830 and 1050 °C were selected. For each austenitising temperature two samples were hardened in dilatometer. One sample immediately after hardening was tempered in dilatometer. The other before tempering was cooled in liquid nitrogen for 1 hour.

The investigations of phase transformations during tempering of the investigated steel were made on the ground of dilatometric curves recorded during heating from as-quenched state. Dilatometric investigations were performed by use of the DT 1000 dilatometer made by Adamel, and L78 R.I.T.A. (Rapid Intensive Thermal Analysis) dilatometer made by Linseis. The methodology of the dilatometric investigation and the method of the characteristic points determination were described.

The microstructure analyses were performed using FEI Nova NanoSEM 450 and light microscopy made by Zeiss. The effect of austenitising temperature and sub-zero treatment on the retained austenite volume fraction was studied. During tempering the amount of retained austenite can significantly affect the final properties of the steel. High volume fraction of retained austenite may increase temper brittleness effect caused by the creation of a large amount of fresh martensite, therefore stability problem of retained austenite is very important.

Keywords: alloy steels, phase transformations, dilatometric investigations, retained austenite, transition carbides, cementite carbides, sub-zero treatment

1. INTRODUCTION

The issue of retained austenite influence on mechanical properties in the steels, its stability and stabilization mechanisms is a very interesting subject. The phenomenon of austenite mechanical stabilization has also practical significance upon heat treatment in the majority of tool steels. Such steels usually have low Ms temperature therefore after heat treatment in room temperature there usually remains a big amount of retained austenite which, depending on its morphology, stability and fraction, may have a considerable influence upon such steels properties. One of the major problems associated with the content of retained austenite, is that it is soft phase and unstable at low temperature and transforms into brittle martensite during service. [1-7].

The retained austenite phase in the unalloyed structural steels is characterized by a low mechanical stability. Under an influence of tensile stresses it is easily mechanically destabilized and transforms into martensitic, regardless of the previous thermal stabilization [8].

There is strictly the relation between the carbon content and the retained austenite volume fraction on the mechanical stability of this phase. In the work [8] the retained austenite thermal and mechanical stabilization was considered. Due to the thermal stabilization after the heat treatment more retained austenite remained in C65 steel. However, this austenite has a smaller mechanical stability and easier undergoes destabilization than retained austenite in C45 steel, characterized with a smaller sensitivity to tensile stresses. The
mechanical destabilization does not allow for the complete elimination of retained austenite from the microstructure of the investigated steels. Regardless of the carbon content, after the application of very high stresses close to 3100-3200 MPa, 4-5 % of the retained austenite volume fraction remained in samples of the investigated steels [4,8].

The investigations on the influence of individual elements on the stability of the austenite are very important, particular the impact of chromium (because it is the most common addition to steel and strongly stabilizes the austenite). The issue of chromium interaction with other elements is important and its influence on the phase transformation during cooling of austenite [9].

The cryogenic treatment is used for minimizing the amount of RA in tool steels. This treatment has been claimed to improve wear resistance of several steels and has also been implemented recently in cutting tools [10-13], its effective in reducing the content of retained austenite in high carbon steels, but it cannot completely transfer the RA into martensite. Cryogenic treatment accelerates the precipitation of small secondary carbides, increases their volume fraction and promotes uniform distribution of the carbides in entire bulk of material [13,14].

2. TEST MATERIAL

The investigations were performed on high carbon Cr-Mn-Mo model alloy steel. Its chemical composition is presented in Table 1. The material was supplied as bars of a rectangular cross-section of dimensions 35x20 mm after soft annealing at 650 °C for 10 hours after forging. Before the beginning of examinations a soft annealing was applied to have the material in a state near the equilibrium.

Table 1. The chemical composition (wt. %) of the investigated alloys

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>V</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>WIII</td>
<td>0.99</td>
<td>1.51</td>
<td>0.13</td>
<td>0.009</td>
<td>0.012</td>
<td>1.88</td>
<td>0.01</td>
<td>0.27</td>
<td>0.17</td>
<td>bal.</td>
</tr>
</tbody>
</table>

After this process the microstructure was consist of the cementite and ferrite. The critical points were determined on the basis of the dilatometric investigations and they are equal Ac1s = 750 °C, Ac1f = 780 °C and Acem = 1020 °C. Based on these results two extreme different austenitising temperatures were selected T_A1 = 830 °C and T_A2 = 1050 °C. The first temperature was in the range of austenite with cementite carbides and the second one in the range of homogeneous austenite. These two temperatures ranges significantly influence the phase transformation quenching.

The samples ware quenched from corresponding austenitising temperatures in the dilatometer with the cooling rate of 10 °C/s. During quenched from T_A1 the M_s temperature was equal 270 °C. For samples quenched from higher temperature (T_A2) the M_s temperature have decreased to 160 °C. This is due to dissolution of cementite which resulted in the enrichment of carbon and other elements in the structure. As expected the differences in austenite and M_s temperatures influenced the volume fraction of retained austenite. The volume fraction of RA was measured with magnetic method as a result quantitative amount of this phase was given. One sample from each austenitising temperature immediately after hardening was cooled in liquid nitrogen for 1 h. After these we have two samples from each austenitising temperature one after quenching end second after quenching end cryogenic treatment.

Figure 1 presents the photos of the microstructure of the samples after previously mentioned heat treatment, photos were taken using the scanning electron microscope FEI Nova NanoSEM 450 in BS mode.
Microstructure of the samples quenched from the 830 °C consists of martensite and many not dissolved during austenitising cementite carbides. The diameter of cementite carbides are in the range from 0.4 to 1.4 μm. Cryogenic treatment did not affect significantly on the microstructure only on the volume fraction of RA.

Higher austenitising temperature resulted in complete dissolution of cementite carbides. The microstructure after these quenching consists of plate martensite with a large volume fraction of retained austenite. Cryogenic treatment affect significantly on the microstructure especially on the volume fraction of RA. It is the output state for research of influence of the volume fraction of RA on the phase transformations during continuous heating from as-quenched state.

3. EXPERIMENTAL PROCEDURE AND RESULTS

After quenching and quenching with cryogenic treatment samples ware heated in the dilatometer with heating rate equal 0.1 °C/s to the 700 °C and then cooled to the room temperature. Dilatometric
investigations were performed by use of the L78 R.I.T.A. (Rapid Intensive Thermal Analysis) dilatometer made by Linseis and DT1000 made by Adamel.

Figure 2 shows the dilatometric curves with the corresponding differential curves of heating the samples with 0.1 °C/s heating rate to the 700 °C.

Fig. 2. Dilatometric curves with the corresponding differential curves of heating the samples with 0.1 °C/s heating rate to the 700 °C of the samples quenched from temperature a, b) 830 °C and c, d) 1050 °C and then cryogenic treated b, d) in liquid nitrogen for 1 h

The analysis of the curves has shown that in each case, the similar effects can be pointed of the same phase transformation. For each dilatometric curve first negative dilatation effect is associated with transition carbides precipitation. For samples quenched from $T_{A1}$ temperature this effect is less intensive as for samples hardened from a $T_{A2}$ temperature. This is due to dissolution of cementite which resulted in the enrichment of carbon and other elements in the structure. In each case the temperature of transition carbides precipitations start is equal 70 °C. Also in each case the maximum intensity of transition carbides precipitations occurs in 150 °C temperature. A slight difference occurs at the end of this phase transformation for samples quenched from different temperatures.

Occurred positive dilatation effects are associated with the transformation of retained austenite. The temperature range of retained austenite transformation for sample only quenched from 830 °C is from 200 to 320 °C. Similarly for samples quenched from the same temperature but also cryogenic treated. An important difference is the intensity of these effects. For sample cryogenic treated dilatation effect is very weak.

For samples quenched from 1050 °C without cryogenic treatment the retained austenite transformation temperature range is shifted to the range of 220 – 420 °C and for the sample cryogenic treated these range
is narrowed to a range 240 – 300 °C. The dilatation effect of the retained austenite transformation for the sample cryogenic treated is very weak and almost entirely dominated by the formation of cementite.

The second occurred negative dilatation effect for the investigated steel comes from precipitations of cementite. For samples quenched from 830 °C and for quenched and then cryogenic treated the temperature range is the same 230 – 390 °C. For samples quenched from a higher temperature the range of cementite precipitations shifts to 260-490 °C and for the sample additionally cryogenic treated to 210-450 °C.

The last dilatation positive effect is related to the precipitations of the MC carbides. For each sample the temperature start of these phase transformation is equal to 500 °C. The last negative dilatation effect is related to recrystallization.

The figure 3 presented the summary of above described dilatometric curves in order to visualize the differences in the strength of the dilatation effects coming from the phase transformation during continuous heating from as-quenched state.

**Fig. 3.** The dilatometric curves of heating the samples with 0.1°C/s heating rate to the 700°C of the samples quenched from temperature a) 830°C and b) 1050°C

For samples quenched from 830 °C (Fig. 3a) the most visible difference is the positive dilatation effect associated with the transformation of retained austenite. This effect is more intensive for the sample only quench without cryogenic treatment.

For samples quenched from 1050 °C (Fig. 3b) it can be seen a very big difference. The dilatation effects comes from the precipitations of transition carbides and cementite carbides are more intense in cryogenic treated sample. The main reason is that cryogenic treatment has reduced the volume fraction of retained austenite and the effect comes from these phase transformation do not interfere with the positive effects derived from precipitation of carbides. This means that the cryogenic treatment and the volume fraction of retained austenite not impact significantly on the precipitation of carbides during tempering.

4. **CONCLUSIONS**

Influence of the retained austenite volume fraction and cryogenic treatment on the phase transformations during tempering in high carbon alloy steel was investigated. The following results were obtained:

The microstructure of the samples quenched from the 830 °C consists of martensite and many not dissolved during austenitising cementite carbides with diameter in the range from 0,4 to 1,4 μm. Cryogenic treatment did not affect significantly on the microstructure only on the volume fraction of RA.
The microstructure of the samples quenched from the 1050 °C consists of plate martensite with a large volume fraction of retained austenite. Cryogenic treatment affects significantly on the microstructure especially on the volume fraction of RA.

The first negative dilatation effect is associated with transition carbides precipitation. The temperature of transition carbides precipitations start is equal to 70°C and the maximum intensity of its precipitations occurs in 150°C. A slight difference occurs at the end of this phase transformation for samples quenched from different temperatures.

First positive dilatation effects are associated with the transformation of retained austenite. For the samples quenched from 830 °C and for quenched and cryogenic treated the transformation is in range 200 – 320 °C. For samples quenched from 1050 °C without cryogenic treatment the retained austenite transformation temperature range is shifted to the range of 220 – 420 °C and for the sample cryogenic treated these range is narrowed to a range 240 – 300 °C.

The second negative dilatation effect comes from precipitations of cementite. For samples quenched from 830°C and for quenched and then cryogenic treated the temperature range is the same 230 – 390 °C. For samples quenched from a higher temperature the range of cementite precipitations shifts to 260-490 °C and for the sample additionally cryogenic treated to 210-450 °C.

The last dilatation positive effect is related to the precipitations of the MC carbides. For each sample the temperature start of these phase transformation is equal to 500 °C.

ACKNOWLEDGEMENTS

Financial support: The National Centre for Research and Development, project no PBS1/B6/3/2012 Manufacturing technology of monolithic shafts for power and machine-building industry.

LITERATURE