POSSIBILITIES OF PREDICTION OF THE MARTENSITE VOLUME FRACTION IN DUAL-PHASE MICROALLOYED LOW-CARBON STEELS

Miroslav DŽUPON, Viera HOMOLOVÁ, Iveta SINAIOVÁ, Lucia ČIRIPOVÁ
Institute of Materials Research SAS, Watsonova 47, 040 01 Košice, Slovak Republic,
E-mail: mdzupon@imr.saske.sk

Abstract
Advanced high strength steels - AHSSs, involve a group of dual-phase steels. Dual-phase ferrite-martensite low-carbon steels are mainly intended for cold forming. A suitable combination of the ferrite and martensite volume fractions makes it possible to achieve higher strength and sufficient plasticity parameters of these steels. The strength properties are mainly influenced by the volume fraction of martensite, and the ferrite matrix provides the required plasticity of steel. The paper analyses the effect of the intercritical annealing temperature on the formation of the dual-phase ferrite-martensite microstructure. The phase transition temperatures $\left( A_{c1}, A_{c3}, A_{r3}, A_{r1} \right)$ were determined dilatometrically for the microalloyed steel $C_{3.734A1}$, $C_{4.892A3}$, $C_{0.838A3r}$, $C_{8.695A1r}$, and the critical equilibrium temperature $C_{690A1}$ between ferrite–cementite and ferrite-austenite and the critical equilibrium temperature $C_{0.846A3}$ between ferrite and ferrite-austenite were calculated using Thermo Calc software. The calculation was made at quasi-equilibrium temperatures from 773 K for the Fe-C-Mn-Si-Al-Ni-Cr-Ti-V-Nb-Mo system and the bcc, fcc, liquid, MX, XM2, CM3, CM6, 37CM, 623CM phases. The correlation between the predicted volume fraction of martensite, as a function of the intercritical annealing temperature, and the volume fraction of martensite in experimentally prepared dual-phase microstructures was determined.

1. INTRODUCTION

In designing the procedure of preparation of microalloyed low-carbon dual-phase steels using the intercritical annealing method, the information on the volume fraction of austenite and the dynamics of change of its volume fraction in the furnace (or the sample) temperature measurement uncertainty interval is crucial for the reproducibility of this process. Austenitization and modelling of the kinetics of formation of austenite in low-carbon steels are described in a number of theoretic articles. The analysis of the austenitization processes has been based on combined and separate models to solve the issues of diffusion processes and the processes of austenitization and growth of the austenite grain [1-4]. The simulation of the diffusion-controlled phase transformation is one of applications of Thermo-Calc software [5].

Microalloyed steels (HSLA) are high-strength low-alloyed steels which have better mechanical properties and can have higher atmospheric corrosion resistance than conventional steels and have the carbon content from 0.05 to 0.5% C. The main alloying element is manganese with the content of up to 2.0%. The microalloying elements Nb, V and Ti participate in the formation of carbonitride precipitates and are one of the main hardening mechanisms of microalloyed steels. The role of microalloying elements in ferrite-pearlite steels is to refine the ferrite grain and to contribute to the precipitation hardening.

The aim of this work was to obtain information on the dynamics of austenitization of the low-carbon microalloyed steel in the temperature interval $A_{c1} - A_{c3}$ and on the volume fraction of austenite present in the microstructure at $t \in (A_{c1}, A_{c3})$ using Thermo Calc software and to compare the predicted volume fractions of austenite and martensite in the experimentally prepared low-carbon dual-phase steels.
2. EXPERIMENT

For experiments, samples taken from commercially produced hot-rolled sheet of structural Nb-Ti microalloyed steel with the fine-grained structure and the thickness of 2.95 mm (marked as M) were used. The chemical element composition of the experimental material was determined by optical emission spectral analysis (Tab.1).

Table 1 Chemical composition of experiment material [% by weight]

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Al</th>
<th>Cu</th>
<th>Ni</th>
<th>Cr</th>
<th>Ti</th>
<th>V</th>
<th>Nb</th>
<th>Mo</th>
</tr>
</thead>
<tbody>
<tr>
<td>M</td>
<td>0.077</td>
<td>1.049</td>
<td>0.006</td>
<td>0.008</td>
<td>0.004</td>
<td>0.039</td>
<td>0.020</td>
<td>0.012</td>
<td>0.016</td>
<td>0.018</td>
<td>0.002</td>
<td>0.048</td>
<td>0.003</td>
</tr>
</tbody>
</table>

The phase transition temperatures \((Ac_1, Ac_3, Ar_3, Ar_1)\) were determined using the dilatometric method. This method utilizes volume changes occurring during phase transformations in steel. The M steel samples were analyzed using the Ernst Leitz Wetzlar optical dilatometer at the heating temperature interval of 500-1000 °C and the cooling temperature interval of 1000-650 °C. The temperatures of transformation of \(\alpha + Fe_3C\) into austenite \((Ac_1, Ac_3)\) and back transformation of austenite into \(\alpha + Fe_3C\) \((Ar_1, Ar_3)\) were determined at the points of characteristic bends on the dilatometric curve. The sample heating and cooling rates were 70°C/h at the \(\gamma / Fe_3C \leftrightarrow \alpha \) transformation temperature interval. The phase transition temperatures were: \(Ac_1 = 734.3°C\), \(Ac_3 = 892.4°C\), \(Ar_3 = 838.0°C\), \(Ar_1 = 695.8°C\).

The dual-phase low-carbon steels were prepared experimentally using the intercritical annealing method. Samples with the dimensions of 30 x 170 x 2.99 mm taken from the hot-rolled low-carbon steel sheet in the rolling direction were first heat-treated using the normalizing process at 900°C/(10 minutes heating to the annealing temperature + 5 minutes dwelling at the annealing temperature)/air cooling, and then were subjected to intercritical annealing at 720°C, 740°C, 760°C, 780°C, 800°C, 820°C, and 850°C. The annealing time for all the states was 12 minutes (7 minutes heating to the annealing temperature + 5 minutes dwelling at the temperature). After dwelling at the intercritical annealing temperature, the samples were cooled down in a 10% NaOH water solution with the temperature of 22-24°C. In a furnace with the Ar 5.0 protective atmosphere, two samples were heated together. Thus, various states were achieved, having various volume fractions of martensite. Each experimental group consisted of eight samples.

3. RESULTS AND DISCUSSION

The microstructures in all the groups of samples (720°C to 850°C) were analyzed using light microscopy. In each group, seven samples were analyzed. On each sample, eleven image fields were measured. Each image field consisted of 3x3 pictures. On each picture, seven measurements of the area fraction of martensite \(V_m\) and the mean ferrite grain size \(d\) were made, i.e. 693 measurements of \(V_m\) and \(d\) were made on each sample. The area fraction of martensite was determined using the point method. The following was determined: arithmetic means of \(V_m\) and \(d\) and the standard deviations of \(V_m\) and \(d\) by sample, which show how \(V_m\) and \(d\) differ from the arithmetic means of \(V_m\) and \(d\) (Fig. 1-6, Tab.2).
Table 2 Area fraction of martensite \( V_m \) and ferrite grain size \( d \)

<table>
<thead>
<tr>
<th>Temperature [°C]</th>
<th>720</th>
<th>740</th>
<th>760</th>
<th>780</th>
<th>800</th>
<th>820</th>
<th>850</th>
</tr>
</thead>
<tbody>
<tr>
<td>( V_m ) [%]</td>
<td>Ave</td>
<td>3.8</td>
<td>6.7</td>
<td>8.9</td>
<td>12.5</td>
<td>29.9</td>
<td>45.5</td>
</tr>
<tr>
<td>StDev</td>
<td>0.3</td>
<td>0.5</td>
<td>0.5</td>
<td>0.5</td>
<td>3.5</td>
<td>3.5</td>
<td>4.5</td>
</tr>
<tr>
<td>( d ) [μm]</td>
<td>Ave(^{(i)})</td>
<td>8.40</td>
<td>7.35</td>
<td>8.09</td>
<td>8.13</td>
<td>8.20</td>
<td>7.84</td>
</tr>
<tr>
<td>StDev(^{(ii)})</td>
<td>0.45</td>
<td>0.16</td>
<td>0.14</td>
<td>0.60</td>
<td>0.57</td>
<td>0.66</td>
<td>~0.08</td>
</tr>
</tbody>
</table>

\(^{(i)}\) Arithmetic mean, \(^{(ii)}\) Standard deviation by sample

The dependence of the area fraction of martensite on the intercritical annealing temperature, for the intercritical annealing temperatures of 720°C – 850°C, is shown in Fig. 7. Within the 5% error in experimental determination of the area fraction of martensite, the dependence of \( V_m \) on the intercritical annealing temperature \( t \) could be approximated using the linear function (1) for the temperature interval of 720-780°C and the linear function (2) for the temperature interval of 780-850°C (Figs. 8, 9). In the Equation (1), 14.0 is the regression coefficient. It also represents the statistical mean value of the intensity of increment of the volume fraction of martensite in all the four states in the annealing temperature interval \( t = 780 – 850°C \). The intensity of increment of the volume fraction of martensite in the temperature interval \( t = 780 – 850°C \) is characterized by the coefficient 0.86, which is 6-times higher than the coefficient of intensity of increment of the volume fraction of martensite in the temperature interval \( t = 720 – 780°C \).
The information on the kinetics of formation of austenite during intercritical annealing in the temperature interval of existence of $\alpha - \gamma$ phases was obtained using thermodynamic calculations of phase balances. The calculations were made with Thermo Calc software, using the database "A. Kroupa, Internal report of IPM Brno 2006". The following temperatures were calculated: critical equilibrium temperature $A_1 = 690^\circ\text{C}$ between ferrite-cementite and ferrite-austenite, and the critical equilibrium temperature $A_3 = 846.0^\circ\text{C}$ between ferrite and ferrite-austenite. The calculation was made at quasi-equilibrium temperatures from 773 K for the $\text{Fe} - \text{C} - \text{Mn} - \text{Si} - \text{Al} - \text{Ni} - \text{Cr} - \text{Ti} - \text{V} - \text{Nb} - \text{Mo}$ system and the bcc, fcc, liquid, $\text{MX}$, $\text{M}_2\text{X}$, $\text{M}_3\text{C}$, $\text{M}_6\text{C}$, $\text{M}_7\text{C}_3$, $\text{M}_{23}\text{C}_6$ phases. Information was obtained on the volume fractions of the $\alpha$-Fe and $\gamma$-Fe fractions in the $A_1 - A_3$ temperature interval (Fig. 10) and the equilibrium temperatures $A_1$ and $A_3$ of the $\text{M}$ material. For temperatures at which the dual-phase structures were prepared, the predicted values of the $V_\gamma$ volume fraction of the $\text{Fe}_\gamma$ phase and the $V_m$ martensite volume fraction were compared (Fig. 11). In the temperature interval $t = 720 - 780^\circ\text{C}$, the ratio of the calculated volume fraction of the $\text{Fe}_\gamma$ phase to the experimentally determined volume fraction of martensite $V_m$ was approximately $V_\gamma/V_m \sim 2$, and for the temperature $t = 780 - 850^\circ\text{C}$ this ratio was $V_\gamma/V_m \sim 1 - 1.4$. The calculation of the $\text{Fe}_\gamma$ phase fraction for the temperature $t = 720 - 780^\circ\text{C}$ and the temperature $t = 780 - 850^\circ\text{C}$ was conditioned by reaching the quasi-equilibrium temperatures from 773 K for the $\text{Fe} - \text{C} - \text{Mn} - \text{Si} - \text{Al} - \text{Ni} - \text{Cr} - \text{Ti} - \text{V} - \text{Nb} - \text{Mo}$ system.
The diffusion process was the controlling mechanism of austenitization. Under conditions of a quasi-equilibrium thermodynamic process, the kinetic coefficients are a function of temperature. At \( t = 720 - 780^\circ C \), the diffusion of carbon was less intensive as at higher temperatures \( t = 780 - 850^\circ C \). This can explain the different ratios \( V_\gamma / V_m \sim 2 \) and \( V_\gamma / V_m \sim 1 - 1.4 \) for temperatures from intervals \( t = 720 - 780^\circ C \) and \( t = 780 - 850^\circ C \). In the process of real preparation of dual-phase microstructures by intercritical annealing, this process was terminated after short dwelling at the intercritical annealing temperature.

In the process of heat treatment by intercritical annealing, an uncertainty from temperature measurements and from heterogeneity of the furnace thermal field is introduced in the process. The heat treatment process should be considered in a temperature interval around the required temperature. The information on the increment of the austenite volume fraction at a unit temperature change obtained using Thermo-Calc software makes it possible to assess the stability of the whole annealing process from the viewpoint of the chemical composition of the material.

4. CONCLUSIONS

Low-carbon dual-phase steels were prepared experimentally using the intercritical annealing method. The following was determined using light microscopy: arithmetic means of \( V_m \) and \( d \), and standard deviations of \( V_m \) and \( d \) by sample. Within the 5% error in experimental determination of the area fraction of martensite, the dependence of \( V_m \) on the intercritical annealing temperature \( t \) was approximated using the linear function (1) for the temperature interval of 720-780°C and the linear function (2) for the temperature interval of 780-850°C. The intensity of increment of the volume fraction of martensite in the temperature interval \( t = 780 - 850^\circ C \) was characterized by the coefficient 0.86, which was 6-times higher than the coefficient of intensity of increment of the volume fraction of martensite in the temperature interval \( t = 720 - 780^\circ C \). The information on the kinetics of formation of austenite during intercritical annealing in the temperature interval of existence of \( \alpha - \gamma \) phases was obtained with Thermo Calc software using the database “A. Kroupa, Internal report of IPM Brno 2006”. In the temperature interval \( t = 720 - 780^\circ C \), the ratio of the calculated
volume fraction of the $\gamma$ phase to the experimentally determined volume fraction of martensite $V_m$ was approximately $V_\gamma/V_m \approx 2$, and for the temperature $t = 780 – 850^\circ C$ this ratio was $V_\gamma/V_m \approx 1–1.4$. The analysis of the increment of the austenite volume fraction at a unit temperature change obtained using Thermo-Calc software makes it possible to assess the stability of the whole annealing process from the viewpoint of the chemical composition of the material.

ACKNOWLEDGEMENTS

The authors would like to thank the Slovak Research and Development Agency for the financial support of the Project APVV 0629-06.

REFERENCES