QUANTIFICATION OF MICROSTRUCTURAL PARAMETER FERRITIC-MARTENSITE DUAL PHASE STEEL BY IMAGE ANALYSIS

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Abstract
Method of optical microscopy with combination of digital record of image and with software for image analysis present productive tool, with which is possible receive new information about parameters of microstructure dual phase steel. Condition of using image analysis is encompassment of preparation of metallographic cut so that microstructure was convenient for further processing. Necessary condition is convenient technique of etching so that individual structural ingredients were equally contrast and convenient for consistent image analyses. Were tested different techniques of etching and different types of enchants, which were documented with optical microscopy. Consequently were investigated possibilities of image analyses for determining of volume fraction of martensite and grain size of ferrite. On interpretation of these microstructural parameters was used software called Image J, which is develop for scientific public and it is freeware. Program is offering general spectrum methods for digital processing and consistent image analyses. Image J provides possibility of specific work with picture of metallographic cut and make it possible to determinate grain size, volume fraction of individual phases but also largeness of various elements their form, distribution and morphology.

Target this jobs was examine various types of enchants or combination of enchants and technique of etching for dual phase steel. Primary target was find optimal techniques of etching for achievement sufficient contrast for digital processing of image microstructure ferritic-martensite dual phase steel with tools of image analyses.

1. Introduction
The structure of dual phase steel consists of ferritic matrix and islands of martensite. Martensite increase strength of basic material and ferritic matrix provide good plastic properties. In the combination high strength, good ductility and not expressive yield strength, dual phase steels are ideal material for automobile industries [1, 2]. They are used on production parts of bodyworks, chassis and wheels. These are parts, which are susceptible to absorb crash energy. In the present time we know two methods of production dual phase ferritic-martensite steels. It is method of intercritical annealing (between $A_{C1}$ and $A_{C3}$) or technology of controlled forming and cooling (as rolled), susceptible to reach required volume fraction of ferrite and martensite [3-4].

Image analysis dealt with acquisition of quantitative information about various parameters of microstructure at material on his 2D cut, such as determination of percentage fraction of phase in the matrix or size of phase in structure [5]. The main problem of image analyses is right contrast setting. In the case, right level of contrast setting for individual components of structure, we reach by image analyses more accurately results. The most important factor for using of image analyses is preparation of sample, so as individual components of structure were sufficiently
contrast, what we can reach by applicable etching technique. Table 1 summarizes various etching techniques and etchants that have been used by several investigators [6-8].

Table 1. Etching techniques for dual-phase steel [6-8]

<table>
<thead>
<tr>
<th>Etchant</th>
<th>Result of etching</th>
<th>Author of etchant</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 g(NH₄)₂S₂O₈, 2 ml HF, 50 ml CH₃COOH, 150 ml H₂O</td>
<td>Martensite etches dark, retained austenite lighter than the ferrite matrix</td>
<td>Rigsbee [7], Vander Arend</td>
</tr>
<tr>
<td>Solution A: 7 g Na₂S₂O₅, 100 ml H₂O Solution B: 5 g picric acid, 100 ml C₂H₅OH Solution C: 4 ml HNO₃, 95 ml C₂H₅OH</td>
<td>Ferrite is blue, martensite and austenite is yellow, bainite is brown</td>
<td>Bandoh [8]</td>
</tr>
<tr>
<td>Solution A: 1% Na₂S₂O₅ v H₂O Solution B: 4% picric acid with C₂H₅OH</td>
<td>Martensite white, bainite black, ferrite khaki</td>
<td>LePera [6]</td>
</tr>
<tr>
<td>Picral, Sodium Bisulphate (NaHSO₄)</td>
<td>Stains ferrite grey, martensite white, bainit black</td>
<td>LePera, Goodhart, Aichbaumik,</td>
</tr>
<tr>
<td>Picral, alkaline chromate (2g CrO₃, 40g NaOH, 72ml H₂O)</td>
<td>Stains new ferrite white, old ferrite grey, martensite black</td>
<td>Huppi, Matlock [7]</td>
</tr>
</tbody>
</table>

Target of this job was to find an optimal technique of etching for achievement a sufficient contrast for digital processing of image for ferritic-martensite dual phase steel microstructure with tools of image analyses and then a determination of microstructure parameters by software called Image J.

2. EXPERIMENT

2.1 Material

On experimental work was used DP 600 steel, which is produced in U.S. Steel Kožice. Chemical element’s analyse of received material is at Table 2. Microstructure of this steel consists of ferrite and martensite, let us say bainit. Mechanical properties DP 600 steel were determined on samples, which were parallel with direction of rolling, yield tensile strength \( R_m = 620 \) MPa, yield strength \( R_{p0.2} = 410 \) MPa, ductility \( A_5 = 29.5\% \), relationship yield strength to yield tensile strength \( R_m/R_{p0.2} = 0.65 \).

Table 2. Chemical elements’ analyse [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>Cr</th>
</tr>
</thead>
<tbody>
<tr>
<td>DP 600</td>
<td>0.065</td>
<td>1.10</td>
<td>0.021</td>
<td>0.045</td>
<td>0.004</td>
<td>0.025</td>
<td>0.56</td>
</tr>
</tbody>
</table>

2.2 Etching techniques

Samples for optical microscopy were prepared by convention technology of preparation metallographic cut (they were wet grinding by using of sand paper with
granularity 180 ñ 800 and then they were polishing by diamond paste with granularity 1 μm. For etching of microstructure DP 600 steel was used these practises:

1. etching by Nital (2% solution HNO$_3$ in ethyl alcohol),
2. combined etching: 40-60 second in 4% picric acid in ethyl alcohol and then 5 second by Nital (2% solution HNO$_3$ in ethyl alcohol),
3. two-step etching: 5 second by 3% Nital (3% solution HNO$_3$ in ethyl alcohol) and then 30 second by 10% aqueous solution of sodium metabisulfite (Na$_2$S$_2$O$_5$).

2.2 Image analyses

For image processing and image analyses was used image analyzer ImageJ [9]. This software can define count object in picture, analyze their surface, length, boundaries, moments, count of grains and grains size, volume fraction of phase and to deposit their results of analyses to data files.

On measurement grains size of ferrite and martensite islands were used function Analyse particles by which is possible to determine perimeter and count of grains. By application of this function we can in the picture to determine defined particles (such as inclusion), which form is defined by two parameters ñ size in pixels and circularity of object. The volume fraction of martensite is possible to determine by function Area fraction. Principle is in determining of surface fraction of chosen object (in our case it is fraction of martensite to ferrite) to whole picture. Applied function determine surface fraction after thresholding of picture by function Threshold which is determined by red colour to general surface of picture and after installing macro Measure area fraction we can to determine surface fraction of martensite in percentage. By software ImageJ we can determine also others parameters of microstructure such as:

- Perimeter in the case of closed polygon it is a perimeter, in the case of opened polygon (broken line, straight line) it is a length of line calculated as sum of Euclidian distance of individual fender points along counter,
- Circularity is defined as: \( \frac{4 \pi \times \text{surface}}{\text{perimeter}^2} \). This value is from interval \(<0,1>\) and this value 1.0 is a round, but at the praxis this value can be higher as 1.0,
- Ferret's diameter is average of round, which is having equal surface as surface of observed object

Very interesting function is function Surface plot which draw by intensity of grey colour a picture his 3D cut. The function Surface plot" can offer a basic vision about morphology of observed surface [9].

3. RESULTS AND DISCUSSION

By etching DP 600 steel in Nital (Fig. 1-2) and by combined etching in picral and Nital was reached suitable contrast between ferrite and martensite for determination a volume fraction of martensite (Fig. 3-4). The grains boundaries of ferritic grains and islands of martensite were ñ displayed as compact, which brought inaccuracy to determination of ferritic grains size and volume fraction of martensite at application of some tools software ImageJ, such as Threshold". The best contrast between ferrite and martensite, let us say bainit (Fig. 5-6) we are reached by two-step etching 5 second by 3% Nital (3% solution HNO$_3$ in ethyl alcohol) and then 30 second by 10% aqueous solution of sodium metabisulfite (Na$_2$S$_2$O$_5$).
Medium size of ferritic grains $d_F$ and volume fraction of martensite $V_m$ was determined. Ferritic grains size was determined by linear method, which is described
in STN 42 0462 [10] (Tab. 3) and volume fraction of martensite was determined by grids method (square sculptured glass at measurement 15 cm x 15 cm with grid 1 x 1 cm throughout whole surface). The achieved parameters were then compared with parameters of microstructure, which were achieved by image analyses. Results of image analyses for individual types of etchants are in Table 4. The microstructural parameters achieved by image analyses on samples, which were etched by various techniques of etching show big differences (Tab. 4), which is caused by not-etching of some grain boundaries and by insufficient contrast between individual phases. If we compare this with results, which were achieved by standard methods by STN 0462 (Tab. 3), so comparable results we are reached by two-step etching 5 second by 3% Nital (3% solution HNO₃ in ethyl alcohol) and then 30 second by 10% aqueous solution of sodium metabisulfite (Na₂S₂O₅).

Table 3. Microstructural parameters

<table>
<thead>
<tr>
<th>Material</th>
<th>Volume fraction of martensite Vₘ %</th>
<th>Ferritic grain size d_F [μm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>DP 600</td>
<td>22</td>
<td>8,3 μm</td>
</tr>
</tbody>
</table>

Table 4. Microstructural parameters - Image J

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</tr>
</thead>
<tbody>
<tr>
<td>DP 600</td>
<td>1</td>
<td>99</td>
<td>194,5</td>
<td>34,9</td>
<td>4,7</td>
<td>12,5</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>199</td>
<td>85,4</td>
<td>22,8</td>
<td>3,7</td>
<td>22</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>492</td>
<td>7,8</td>
<td>8,4</td>
<td>2,8</td>
<td>22</td>
</tr>
</tbody>
</table>

4. CONCLUSION

The conditions of application image analyses is encompassment of preparation metallographic cut so, that individual structural components were sufficiently contrastive.

In this work was attest availability applications three types of etchants on activating microstructure of DP 600 steel. The microstructural parameters achieved by image analyses on samples, which were etched by various techniques of etching show big differences (Tab. 4), which is caused by not-etching of some grain boundaries and by insufficient contrast between individual phases.

Suitable contrast between ferrite and martensite, let us say bainit was reached by two-step etching 5 second by 3% Nital (3% solution HNO₃ in ethyl alcohol) and then 30 second by 10% aqueous solution of sodium metabisulfite (Na₂S₂O₅). After this etching islands of martensite was show as compact surface blocks and this is convenient for consecutive application of image analyses by software ImageJ.
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LITERATURE