THE EVALUATION OF RETAINED AUSTENITE IN THE CARBURIZED LAYERS

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Abstract

During heat treatment of carburized steel parts, the transformation of austenite to martensite occurs and a certain amount of retained austenite remains in the hardened layers. The higher content of retained austenite in the carburized layer adversely influences hardness and frictional characteristics and increases susceptibility to fatigue damage. Furthermore, retained austenite can transform into deformation induced martensite during service, which provokes changes in properties of the carburized layers. From the technological viewpoint, the content of retained austenite is nearly always monitored but obtained results can be different depending on experimental methods.

In this work, the amount of retained austenite in carburized layers of manganese-chromium steels was evaluated by image analysis method, by X-ray diffraction and also by EBSD. The largest amount of retained austenite was measured using the technique of X-ray diffraction, i.e. 28 %. The amount of retained austenite measured by image analysis of metallographic images was 13 %. However, the results of image analysis greatly depend on the metallographic preparation of samples, digital image processing and the grayscale level settings for the calculation of the retained austenite content. The lowest amount of retained austenite was determined by EBSD, i.e. 5 %. Even in this case the values depended on the method of scanned data processing. No method used to determine the amount of retained austenite in the carburized layer is universal and largely depends on the sample preparation and partial steps performed in individual measurement methods.

Keywords: Retained austenite, carburized layers, image analysis, X-ray diffraction, EBSD

1. INTRODUCTION

The structure of carburized layers after the saturation process of the surface of parts in gaseous, liquid or solid medium and subsequent heat treatment (quenching and low-temperature tempering) is formed by high carbon plate martensite with a certain proportion of untransformed austenite. Austenite, which is preserved in the structure of the hardened steel, is referred to as retained austenite. Its presence after the quenching in a bath of room temperature depends on the temperature range of martensitic area, on the possibility of austenite stabilization and on the austenite grain size. The amount of retained austenite after quenching in a bath of room temperature increases rapidly with the carbon content and alloying elements. The proportion of retained austenite also increases with decreasing austenite grain size and with a decreasing cooling rate, and so the proportion is the greatest at the critical cooling rate [1, 2].

The presence of retained austenite in carburized layers is considered generally undesirable because it reduces the hardness of the hardened layer and can lead to spontaneous conversion to a ferritic-carbidic mixture of a bainitic type which is accompanied by a change of properties, dimensional instability and the local increase of internal stress with the possible formation of cracks. The proportion of retained austenite is, therefore, an important characteristic of the quality of carburized layers [1, 2].

The basic methods of studying the structure of solid crystalline materials include light microscopy (metallographic evaluation), methods using a focused electron beam, e.g. electron backscattered diffraction (EBSD) and X-ray diffraction.

Taking metallographic images in digital format allowed the development of automated image analysis. Individual pixels are disclosed by a single numerical value so-called gray level. 8-bit display, which is equivalent to 256 gray levels, is usually used as the default. Absolutely white is associated with gray level 255 and absolute black gray level 0. From the histogram of gray levels can be seen, frequency appearance of different gray levels in the image. If the histogram of gray levels of images shows at least two maxima, the detection of areas in the image by so-called thresholding is possible. The image is then suitable for quantitative analysis. The quality of the scanned image and other image operations affect the distribution of gray levels in the histogram and therefore affect the thresholding value. The actual value of thresholding then influences the resulting values of the quantitative proportion of phases [3, 4].

The determination of the amount of retained austenite in the hardened and tempered steel and the cemented steel may be obtained by using X-ray diffraction, from the intensity of diffraction peaks which are related to the amount of the existing phases. Martensite and austenite have different diffraction peaks in X-ray diffraction and that is why their intensity peaks can be used to calculate the volume fraction of the phases in the steel. Results of X-ray analysis in hardened steels and in carburized layers may be influenced by the presence of primary carbides. The peak intensity is influenced by e.g. grain, texture, surface tension, surface roughness. Different X-ray diffraction results for the same sample also result from the experimental conditions and the diffractogram analysis itself [4-7].

Electron backscattered diffraction (EBSD) is an advanced technique for the characterization of the microstructure and analysis of the crystalline materials, which is based on analyzing Kikuchi lines projecting from the surface of a strongly inclined sample in SEM chamber. Many structural parameters, on which the behavior and properties of materials depend, may be derived from EBSD data, e.g. grain size, phase composition, mechanical anisotropy and residual deformations. The analysis of the retained austenite in the martensitic structure is problematic. The measurement results may be distorted due to the transformation of retained austenite to strain-induced martensite in the surface layers during sample preparation. The same problem may occur when using X-ray measurements of retained austenite but with a lesser extent due to increased penetration of X-rays beneath the surface of the sample [7, 8].

This paper deals with the evaluation of the volume fraction of retained austenite in carburized layers by using digital image analysis of metallographic images, X-ray diffraction method or EBSD method.

2. EXPERIMENTAL METHODS

A cylindrical body with a diameter of ∅ 34 mm made of steel ČSN 14 220 (equiv. to ČSN 1.7131, 16MnCr5) was used for the evaluation of retained austenite. The sample was case hardened at 930 °C in a vacuum furnace at regular alternation of carburized and diffusion periods. Carburized period lasted for 5 minutes (total 45 minutes), the diffusion period gradually rose from 5 to 90 minutes (total 330 minutes). Hardening was carried out directly at the temperature of 930 °C, by the nitrogen pressure of 5 bars. The sample was tempered at 200 °C.

High carbon plate martensite and retained austenite formed the microstructure of the hardened layer, as is shown in **Figure 1**. The etching of the microstructure was performed in Nital.

Hardness and depth of the hardened layer were measured by microhardness tester Leco AMH 2000. The graphic dependence on the distance below the surface of hardness HV1 is represented by the curve shown in **Figure 2,** where is highlighted the contracting hardness value of 550 HV1, for which a depth of diffusion layer CHD 1.07 mm was set (CHD means Case Hardening Depth).

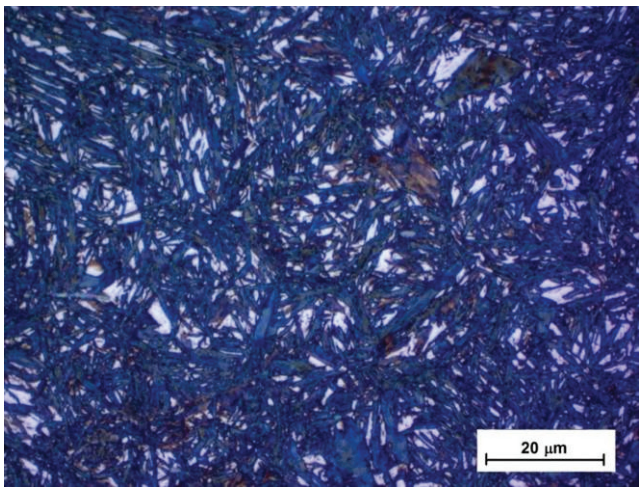

Figure 1 The microstructure of the carburized layer in the depth of 0.1 mm below the surface. Etch. Nital

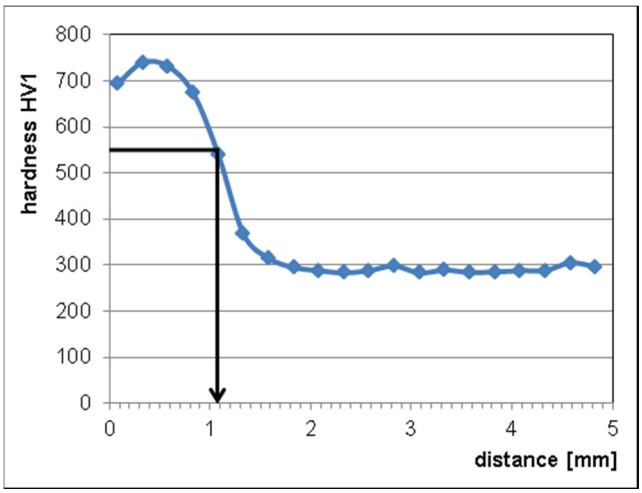

Figure 2 The process of hardness HV1 in the depth of 0.1 mm below the surface. CHD 550 HV1 = 1.7 mm

2.1. Digital image analysis

Samples for evaluating the proportion of retained austenite were taken by way that the surface of the cylindrical body was oriented parallelly to the surface of the metallographic sample. The hot pressed samples were grinded to a depth of 0,1 mm, polished and etched repeatedly in a solution of 4% HNO_3 in ethanol (Nital) to achieve the best contrast between the martensite and retained austenite. The quality of metallographic samples plays an important role in the proper evaluation of the image by image analysis. The documentation of the carburized layer microstructure was taken via metallographic optical microscope Zeiss Axio Observer A1M and software Axio Vision 4.8 at a magnification of 1000x. The portion of retained austenite was evaluated from photographs, which were edited using a delinearising filter (5 x 5 matrix, the threshold level for Sobel operator 35). The lowest difference of retained austenite proportion depending on the gray level for thresholding was found when using the delinearising filter. The software Image-Pro Analyzer 7.0 was used to determine the proportion of retained austenite. The picture was there converted into 8-bit scale level in 256 gray levels. The thresholding gray level was determined from the average of the two peaks on the histogram (major peak corresponds to martensite, minor peak corresponds to retained austenite) - **Figure 3** and **Figure 4**. The calculation of the retained austenite was carried out by the selected function "per area", which calculates the proportion of the area of objects to the total area of the image. 12 randomly selected fields of view were evaluated.

2.2. X-ray diffraction analysis

The proportion of retained austenite was evaluated by X-ray diffraction analysis. The samples were evaluated in not etched condition. The measurement of the retained austenite was carried out on a Bruker-AXS D8 Advance with a 2θ/θ measurement geometry and position sensitive detector LynxEye under following conditions: CoKα radiation/Fe filter, voltage 40 kV, current 40 mA, divergent aperture of 0.28°, step mode with a step 0.014° 2θ, with a total time of 1.25 seconds at a step (summation of five measurements with a step of 0.25 s) and digital processing of the result data. Computer programs Bruker Diffrac Suite were used for the measurement and evaluation. The diffraction data database PDF-2, Version 2011 (International Centre for Diffraction Data, Pennsylvania, USA) was used for quality assessment. The software Bruke Topas version 4.2 with a slightly modified Rietveld method of the structural analysis of powder diffraction was used for quantitative analysis.

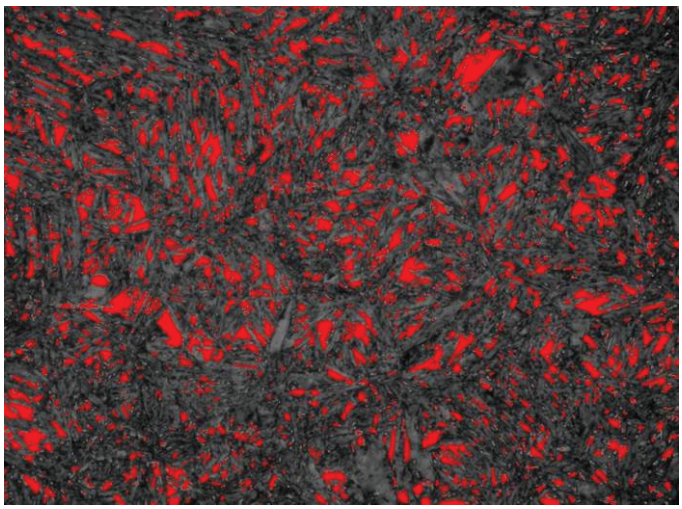

Figure 3 The structure after image edit, delinearising filter (the threshold level for Sobel operator 35), gamma, 1.0, gray level 127

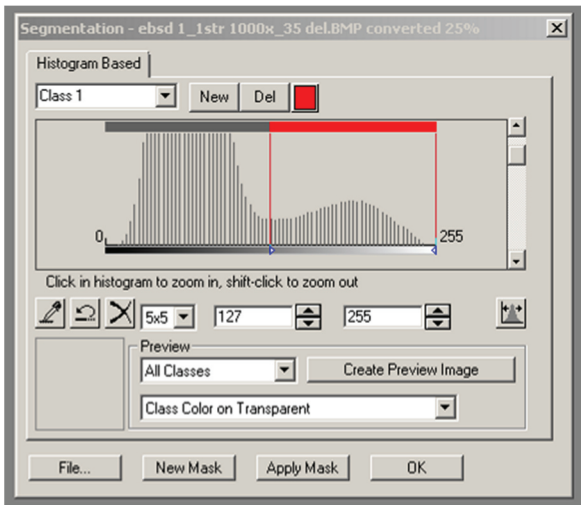

Figure 4 Determining the thresholding gray level 127 from the two peaks of the delinearised image

2.3. EBSD

The measurement was performed on the device Quanta FEG (Field Emission gun) 450 from the manufacturer FEI with the detector HIKARI. The sample was placed in the device and gradually the sample holder was tilting by an angle of 70° to the horizontal plane. The scanning of the selected field of view was performed under following conditions: the analysis time 8 h, step 0.1 μm, speed 65 diffractograms / s, the minimum grain size of 5 pixels.

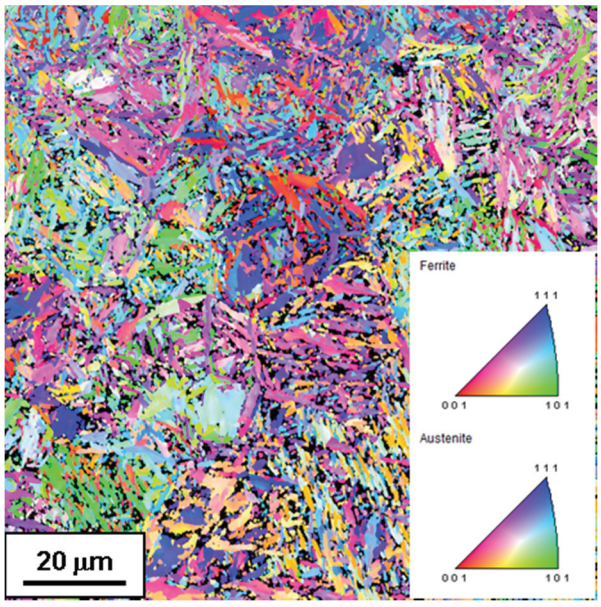

Figure 5 Results of EBSD analysis processed when using the filtration Grain dilation. IPF map for the direction [001] (ND).

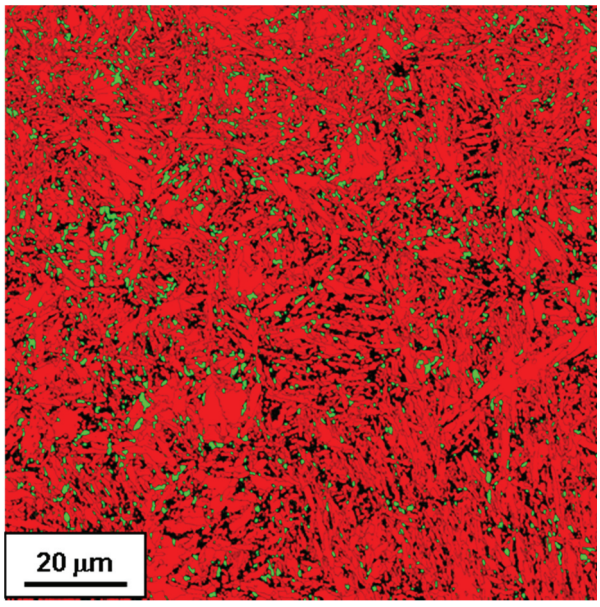

Figure 6 Results of EBSD analysis processed when using the filtration Grain dilation. The phase map, red - martensite, green - austenite.

The relationship of the chosen direction in the coordinate system of the sample to the significant crystallographic directions of the analyzed crystal is represented by an inverse pole figure which is plotted into the basic stereographic triangle. A color that is unique to the position of the projection of a chosen direction in the coordinate system of the sample to the inverse pole figure can be assigned to each analyzed pixel in the studied area after assigning appropriate colors to different areas of the basic stereographic triangle (**Figure 5**). Applying that color coding on all analyzed points creates the two-dimensional "Orientation Map" or "Inverse Pole Figure Map" IPF (**Figure 5**), which is constructed always for one selected macroscopic direction in the coordinate system of the sample. Problems with an identification of any orientation may occur in the orientation maps, e.g. at the grain boundaries, where there are often diffractograms composed of two superposed diffraction patterns from both crystal grids separated by grain boundaries. This can lead to an incorrect indexation. Through the parameter CI (Confidence Index), an image filtering ("Clean-Up") can be carried out. Confidence index takes values from 0 to 1. The value is -1 for diffractograms that can´t be indexed. The hue of pixels is becoming more vivid (darker) and not indexed pixels have black contrast along with the decreasing reliability of diffractograms indexing. [9, 10] Evaluation of the recorded data was performed in the OIM program (Orientation Imaging Microscopy) 6.0. The average confidence index was 0.25, the number of analyzed points: 1776 649. Three locations on the surface of the sample were analyzed without a filtration, using the filtration Grain IC Standardization and Grain dilation (**Figure 6**).

3. RESULTS AND DISCUSSION

The mean value of retained austenite proportion was determined by the method of digital image analysis from 12 randomly selected fields of view as 13.9 ± 1.5 %. The result value of the retained austenite proportion is very influenced by the actual sample preparation, scanning the image and its editing. There is no universal technique of image editing and settings of gray levels because each image is unique and influenced by sample preparation, microscope setting, image editing techniques and it´s evaluation.

The value of retained austenite proportion of 28.3 % was determined by X-ray diffraction analysis. In the case of the analysis by X-ray diffraction, overestimation of retained austenite might possibly manifest due to the iris used, i.e. the measurements were carried out over the entire width of the sample while only the middle of the sample corresponded to a depth of 0.1 mm below the surface.

The mean value of retained austenite proportion of 4.7 ± 1.4 % was determined by EBSD method with any data filtering (Clean Up). The mean value of retained austenite proportion of 5.1 ± 1.5 % for Grain CI Standardization and of 5.1 ± 1.6 % for Grain dilation were measured when using diffraction data filters. The filtering of scanned data had almost the same results as without the filtering. In this case, the use of filters did not affect the final value of the proportion of retained austenite. The low determined proportion of retained austenite by using EBSD method may be partly related to the fact that in phase maps images occurred areas with a dark contrast, where the diffractograms analysis was unsuccessful, i.e. diffractograms could not be assigned either to the austenite or the ferrite (martensite). These areas were not considered during the quantification of present phases. Problems with the interpretation of diffractograms relate to the nature of the studied sample microstructure - martensite with high dislocation density, high frequency of grain boundaries and interfaces.

4. CONCLUSIONS

This article was focused on comparing the methods used for evaluation of the proportion of retained austenite in carburized layers by methods of digital image analysis, X-ray diffraction, and EBSD.

An important role in the use of image analysis plays the sample preparation, especially the intensity of etching the sample. From the results of the retained austenite, assessment can be concluded that this is a very sensitive method and that there is no universal technique of image editing and setting the gray levels to

evaluate the differently prepared samples. Each image is unique and influenced by sample preparation, setting the microscope, image editing techniques, and evaluation.

The higher value of the retained austenite was determined by X-ray diffraction analysis, which could be due to the fact that the measurements were carried out over the entire width of the sample, not just in the middle, which corresponded to a depth of 0.1 mm below the surface.

Image analysis without correction and using different filters was used for the evaluation of retained austenite by EBSD method. Individual results didn´t differ from each other too much, the usage of filters to determine the proportion of retained austenite did not affect the evaluation.

Observed differences in the proportion of retained austenite correspond to trends that are described in the literature [4, 7, 8]. Usually, the largest proportion of retained austenite is measured by the technique of X-ray diffraction the portion of austenite measured by image analysis of images from light microscopy can be up to 14 % lower.

In conclusion, neither one of the methods were universal for assessing the proportion of retained austenite in the carburized layer of chrome-manganese steel and largely depends on the sample preparation and partial steps performed in the individual measurement methods.

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