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Structural characterization of low-carbon multiphase steels merging advanced research methods with light optical microscopy

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ABSTRACT

The paper presents new approach to characterizing low-carbon multiphase steels structure by means of concurrent application of optical and scanning electron microscopy with the emphasis on the latter one. Limitations of optical microscopy in material structure analysis, and the possibilities of information interchange between both methods have been discussed. The microstructure characterization is mostly based on the results obtained by means of scanning electron microscopy with wide application of EBSD method. As part of the work, fundamental constituents of multiphase steels structure with particular focus on consideration of the possibility of influencing their morphological features during thermal treatment have been systematized and described. Referring to the sheets of TRIP 700 and DP 600 steel, a capability of combining the FEG SEM analysis with EBSD method to characterize their structure was presented. EBSD method enables not only distinction of phases of various crystalline structure. Owing to analysis of parameters such as IQ, FIT, CI, KAM and misorientation angle, it is possible to differentiate constituents having different crystal structure. The method of IQ distribution curve deconvolution allows estimation of the fraction of structure constituents representing the same crystal structure. Existence of characteristic ranges of misorientation angles for selected structure constituents was proven.

1. Introduction

Low-carbon multiphase steels are characterized by higher strength and ductility, as well as capability of absorbing energy during collision as compared to conventional steel or HSLA grades. To this category belong advanced high strength steels (AHSS) used in automotive industry. Characteristic feature of these steels, as compared to other steels applied in automotive industry, is their high strength. Proof stress and tensile strength of these steels are higher than 300 MPa and 600 MPa, respectively [1,2]. Mechanical properties of these steels depend on the fraction, morphology and mechanical properties of their constituents [3,4]. Structural constituents of the final products made of multiphase steels include: ferrite, bainite, martensite and retained austenite. These

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constituents, depending on chemical composition of steel, and carbon segregation during ferritic and bainitic transformation, as well as heat treatment conditions may develop various morphological forms [5–7].

The study presented in this paper was aimed at detailed characterization of particular constituents of low-carbon multiphase steels structure with special focus on the effective use of the capabilities of optical microscopy in combination with high-resolution scanning electron microscopy and EBSD methods. Thus, the morphological features and conditions of formation of various structural constituents of multiphase steels were precisely examined.

2. Material and the experimental procedure

Research materials comprised 1 mm thick cold rolled sheets of steel produced from laboratory heat specified as 243. The total reduction during cold rolling of these sheets was 50%. Structure was composed of ferritic and pearlitic grains elongated in the cold rolling direction. Moreover, 2 mm thick sheets manufactured in the industrial continuous annealing process of DP 600 and TRIP 700 steels were also investigated. Chemical composition of the examined steels is presented in Table 1.

The capabilities of the developed methodology of the structure analysis by means of optical microscopy were verified on industrially manufactured DP 600 steel. For this purpose, the sample was etched using nital, Le Pera and Klemm's etchant. This material and the sample taken from sheet of TRIP 700 were additionally investigated by FEG SEM and EBSD to present the advantage of combining both methods in the research of multiphase steel structure. The EBSD examination on samples prepared in the same manner as samples for FEG SEM observation was conducted at 20 kV, and sample inclination angle of 70.5°. Analyses were conducted on the area of 57.3 μm × 57.3 μm with analysis step of 0.25 μm for TRIP 700 steel and on the area of 17.3 μm × 17.3 μm with step of 0.12 μm for DP 600 steel. In order to obtain different constituents of multiphase steels, the heat treatment simulations by means of dilatometer were performed. Samples machined from the sheets made of heat marked as 243 for dilatometric examinations, having dimensions 1 mm × 1 mm × 7 mm were subject to heating by means of dilatometer DIL 805A/D at a rate of 3 °C/s to temperatures in the range 780–950 °C. Some of samples were held at the defined temperature for 10–20 s. Cooling was mostly carried out in one step to ambient temperature at rates in the range of 1–70 °C/s. The structure investigation of dilatometric samples was performed using FEG SEM method. The structures of all the samples for FEG SEM investigations were revealed by means of electrolytic etching using the TenuPol-5 device and electrolyte A8 produced by Struers® of the temperature 8.5–10 °C under the voltage of 58 V and polishing time of around 40 s. Observations were conducted by means of high resolution scanning microscope Inspect F equipped with EBSD detector.

This paper presents only the selected results which allows to describe the morphology of different constituents of multiphase steels. The specification of material, details of the thermal profiles applied in the course of investigation which allowed to obtain the different components of structure are given in Table 2. Table 2 is related to Fig. 2 which presents the results of structure investigation.

3. Results and discussion

Due to complexity, small size and fine substructure of structural constituents, the capabilities of examination of the steels by means of optical microscopy are very limited. However, a lot of valuable information can be obtained by using this method which define the field of interest while proceeding to much more complex investigation with FEG SEM and EBSD. The first stage of such an analysis involve etching to reveal the structural constituents and there are various etchants used for this purpose. Examples illustrating the effect of etching method using different etchants are presented in Fig. 1. Commonly, nital is used for revealing the structure constituents of steels (Fig. 1a). Whilst, distinction of ferrite from bainite and martensite is relatively easy (Fig. 1a), it is almost impossible to distinguish martensite/bainite from austenite using nital etching. In such a case, etchants allowing for differentiation of structural constituents based on different colouring, including, e.g. Le Pera and Klemm's ones, are more successful in revealing minor constituents. Le Pera etchant facilitates differentiation of martensite, bainite and retained austenite (M/RA) islands, which are appearing as bright areas in the pictures (Fig. 1b) [5,8]. On the other hand, Klemm's etchant downgrades the ferrite appearance by covering of thin film of ferrum sulphides. White areas correspond to retained austenite (Fig. 1c).

More precise analysis of the structure is conducted by means of scanning electron microscopy FEG SEM and EBSD [9,10]. High-resolution electron microscopy – FEG SEM facilitates identification of structure constituents based on their crystallography and morphology. Examples of the typical structures of multiphase steels investigated by means of FEG SEM are presented in Fig. 2. Description of the samples along with heat specification and its heat treatment variant is presented in Table 2.

Typically polygonal ferrite constitutes matrix of the AHSS structure which is presented in Fig. 2a. Pearlite is composed of colonies, where cementite and ferrite plates are arranged parallely (Fig. 2b). Although avoided in the final structure of

Table 1 – Chemical composition of the materials subject to microstructure characterization (wt.%).

Heat	C	Mn	Si	P	S	Cr	Ni	Mo	Ti	V	Al_t
243	0.09	1.42	0.10	0.011	0.010	0.35	0.01	0.02	0.001	–	0.043
DP 600	0.10	1.57	0.19	0.012	0.004	0.45	0.040	0.008	0.003	0.007	0.044
TRIP 700	0.22	1.50	0.09	0.014	0.005	0.03	0.02	0.01	0.004	0.005	1.7