Micron 81 (2016) 1–7

Contents lists available at ScienceDirect

Micron

journal homepage: www.elsevier.com/locate/micron

Correlative microscopy of a carbide-free bainitic steel

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ARTICLE INFO

Article history:
Received 8 August 2015
Received in revised form 22 October 2015
Accepted 27 October 2015
Available online 1 November 2015

Keywords:
Carbide-free bainite
Martensite–austenite (M–A) constituent
Transmission Kikuchi diffraction (TKD)
Transmission electron microscopy (TEM)

ABSTRACT

In this work a carbide-free bainitic steel was examined by a novel correlative microscopy approach using transmission Kikuchi diffraction (TKD) and transmission electron microscopy (TEM). The individual microstructural constituents could be identified by TKD based on their different crystal structure for bainitic ferrite and retained austenite and by image quality for the martensite–austenite (M–A) constituent. Subsequently, the same area was investigated in the TEM and a good match of these two techniques regarding the identification of the area position and crystal orientation could be proven. Additionally, the M–A constituent was examined in the TEM for the first time after preceded unambiguous identification using a correlative microscopy approach. The selected area diffraction pattern showed satellites around the main reflexes which might indicate a structural modulation.

1. Introduction

Recently, a new method to investigate electron transparent samples by electron diffraction in a conventional scanning electron microscope (SEM) has been developed. There, the Kikuchi diffraction patterns from the transmitted electrons are collected on an electron backscatter diffraction (EBSD) detector. Besides transmission-EBSD (t-EBSD) also the terminology transmission Kikuchi diffraction (TKD) has established and will be used in this study for diffraction patterns formed by transmitted electrons in the SEM. Compared to conventional EBSD an absolute spatial resolution down to 5–10 nm can be reached due to the lower excited specimen volume in a thin foil (Babinsky et al., 2014; Keller and Geiss, 2012; Trimby, 2012).

The easy accessibility of EBSD, its automated data collection and, therefore, the much less time-consuming investigation of larger areas, are the most pronounced advantages. Also, the analysis and post-processing of the data with a variety of options (e.g. crystal structure, grain size, crystallographic orientation relationship) subsequent to the data collection due to the digitalization of the results is a significant benefit compared to transmission electron microscopy (TEM). Especially the characterization of nanostructured assemblies as e.g. present in advanced high strength steels (AHSS) is greatly facilitated by the new, powerful TKD technique with improved resolution. Birosca et al. (2015) and Trimby et al. (2014) investigated the structure of ultrafine-grained steels after high deformation. Also, the phase transformation in transformation- and twinning-induced plasticity steels was studied by TKD to improve the fundamental understanding of the microstructure–property relationship (Gazder et al., 2015; Yen et al., 2015). Nonetheless, the still higher spatial resolution of TEM and the direct imaging of the microstructural features, as e.g. dislocations and precipitates, are reasons to expand the effort to a more time-consuming examination in the TEM (Zaefferer, 2011).

In this work both complementary techniques, TKD and TEM, were applied to the very same position of a thin foil of a carbide-free bainitic steel, which belongs to the third generation of AHSS, e.g. see (Caballero et al., 2013; Grajcar et al., 2012). This steel is transformed isothermally and consists of bainitic ferrite laths and a great amount of retained austenite which is stabilized by C during the isothermal holding time. Depending on the stability of the retained austenite, parts of it may transform to martensite upon quenching to room temperature forming so-called martensite–austenite (M–A) islands. They have a negative impact on the mechanical properties of the material because they act as crack initiation sites and, therefore, reduce the fracture toughness (Lambert et al.,

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http://dx.doi.org/10.1016/j.micron.2015.10.008
0968-4328/

2000). Moreover, the decrease in retained austenite volume fraction diminishes the transformation-induced plasticity effect (Gao et al., 2014; Timokhina et al., 2003). M–A islands also play a crucial role in steel welds after multipass welding (Bonnevie et al., 2004; Lambert et al., 2000; Mohseni et al., 2012). During reheating austenite nucleates along prior austenite grain boundaries, becomes enriched in C and, therefore, forms the M–A constituent upon cooling.

According to microprobe analyses (Biss and Cryderman, 1971; Mohseni et al., 2014) and atom probe tomography (APT) (Hofer et al., 2015) the M–A constituent has an elevated C content compared to the nominal composition. Its outer regions are partly austenitic as shown by EBSD in (Hofer et al., 2015; Santofimia et al., 2014) as a result of the C diffusion out of the bainitic ferrite while the center is mainly composed of plate martensite consisting of micro-twins as shown by TEM (Biss and Cryderman, 1971; Lambert et al., 2000; Lan et al., 2014; Mazancova and Mazanec, 1997). The observation of micro-twinning allowed the conclusion that the corresponding area was part of the M–A constituent. However, other phases, as e.g. lath martensite, austenite and carbides, could also be part of the M–A constituent but could not be assigned to the M–A area without doubt since an unambiguous identification in conventional TEM samples solely based on the contrast in bright field mode is not possible.

Identification in the SEM is facilitated by the reduced etching attack as a consequence of the higher C content as well as the characteristic shape of the M–A islands. Therefore, Mohseni et al. (2014) carried out TEM investigations of the M–A constituent after site-specific preparation in the focused ion beam (FIB) microscope, thus, ensuring that the right areas were investigated. However, the bombardment of the sample with focused Ga ions can lead to the transformation of meta-stable retained austenite even at low voltage and current. These authors also observed twinned martensite but no retained austenite, which could either lie outside of the sample volume or could have transformed to martensite during preparation.

As a consequence in the present study a novel approach for the unambiguous identification and study of the M–A constituent in a carbide-free bainitic steel via TKD was applied. Subsequently, exactly the same area was investigated in the TEM, providing additional information on this highly dislocated constituent. Finally, the advantages and limitations of these two methods, which represent an example of a correlative microscopy, are discussed.

2. Experimental

In this work a carbide-free bainitic steel with the nominal composition of Fe-0.2C-1.5Si-2.5 Mn (in mass%) was investigated. The increased Si content prevents cementite precipitation due to its low solubility for Si and the elevated Mn content ensures hardenability. The material was provided in form of cold-rolled sheets with a thickness of 1.2 mm. Samples of $10 \times 10 \times 1.2\,mm^3$ were inductively heat treated in a dilatometer DIL805 A from TA Instruments (Germany) and the temperature was controlled using a type S thermocouple. After austenitization at 900 °C for 1 min the samples were quenched with a cooling rate of 100 K/s with He gas to the isothermal holding temperature of 400 °C which is about 50 °C above the martensite start temperature (Hofer et al., 2015). There, the samples were held isothermally for 1000 s in the region of the bainitic transformation and then quenched to room temperature with a cooling rate of 100 K/s.

For TKD and TEM studies the heat treated sheets were mechanically thinned to a thickness of 100 μm, and then disks of a diameter of 3 mm were punched out. Subsequently, electropolishing was carried out on a TenuPol-5 twin-jet polisher from Struers (Germany) using a solution of 4 vol% perchloric acid ($HClO_4$) and 96 vol% acetic acid (CH_3COOH) applying a voltage of 15 V with a flow rate of 10 at room temperature.

Prior to the TEM investigations the samples were examined via TKD in a field-emission gun SEM Quanta 450 from FEI (USA) equipped with a Hikari XP EBSD system from EDAX (USA) to identify the M–A constituent. A tilt angle close to 0° is recommended but the actual sample tilt depends on the set-up in the microscope to obtain the diffraction pattern center on the EBSD screen (Keller and Geiss, 2012). In our case an angle of −10° to the incident electron beam was found to give the best results with the actual set-up in the SEM chamber shown in Fig. 1. Therefore, an adapted TEM sample holder with a pre-tilt of −30° was tilted to 20°, resulting in an effective angle of −10° to the incident electron beam. TKD scans were carried out at a working distance of 6 mm with an acceleration voltage of 30 kV, a spot size of 5, a step size of 60 nm, and a 4×4 binning. The data evaluation was performed with the TSL OIM Analysis 7 software without using any data clean-ups.

Subsequently, the same region on the sample was investigated in a JEM-2200FS TEM from JEOL (Japan) with a C_s corrector for the objective lens operated at a voltage of 200 kV. Imaging was carried out using bright field and scanning-TEM (STEM) mode. The schematic spot diffraction patterns were simulated with the software JEMS.

3. Results and discussion

Thin areas close to the hole of the TEM sample were examined by TKD. Since the M–A islands formed during the last cooling step of the heat treatment and they are highly dislocated, the crystal lattice inside these areas is locally distorted leading to a “smearing” of the Kikuchi pattern and, therefore, a decrease in the diffraction pattern quality (Wu et al., 2005). The diffraction pattern quality can be visualized in image quality (IQ) maps, where a greyscale value is attributed to every data point according to its diffraction pattern quality, whereby a darker greyscale value corresponds to a lower pattern quality. Based on an IQ map several areas with M–A constituents could be identified in the bainitic matrix as illustrated in Fig. 2a. As can be seen in the inverse pole figure maps (IPF) of the ferrite in Fig. 2b and the austenite in Fig. 2c, which, in these images, are superimposed with the corresponding IQ map, the outer regions of the large, distinctively shaped M–A area are partly austenitic and the center consists of individual martensitic laths. Therefore, the individual microstructural constituents could be unambiguously identified by TKD based on their different crystal structure for bainitic ferrite and retained austenite and by means of IQ for the M–A constituent. In this study a step size of 60 nm was employed for the TKD scan shown in Fig. 2 to identify the M–A

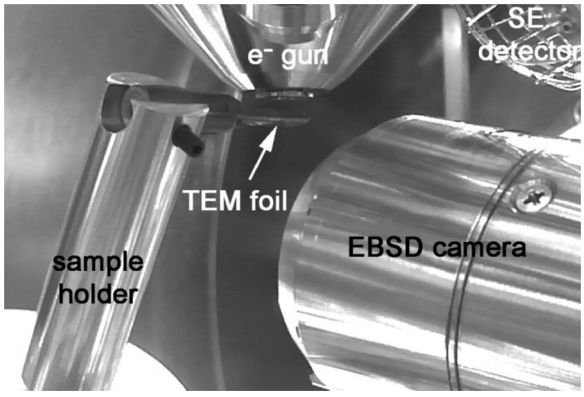

Fig. 1. Set-up in the SEM chamber for the TKD measurements of the TEM foil.