



Tutorial

Differentiation of grain orientation with corrosive and colour etching on a granular bainitic steel



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A B S T R A C T

This study presents a detailed verification of the etching methods with Nital and Klemm on a granular bainitic steel. It is shown that both methods allow the identification of the crystal orientation, whereas Klemm etching enables also a quantification of the apparent phases, as also retained austenite can be distinguished from the other bainitic microstructures. A combination of atom probe tomography with electron-back-scattered-diffraction showed that both etching methods emphasize the bainitic {100} crystal orientation. However, a cross-section produced by focused ion beam evidenced that Klemm etching leads to the formation of a topography of the different oriented bainitic crystals that directly affects the thickness and therefore the apparent colour of the deposited layer formed during etching.

1. Introduction

It is a well-known fact that the mechanical properties of steels are influenced by the solid-solid phase transitions during heat treatment and the corresponding evolution of the microstructure (Eck et al., 2014a, 2014b; Eisenhüttenleute, 1984). Bainitic steels have grown in importance because of their material properties close the gap between conventional martensitic steels and ferritic-perlitic steel grades (Raedt et al., 2017; Bhadeshia, 2001). Due to the related higher costs it is often not possible to accomplish a fully bainitic microstructure by an isothermal heat treatment in steel producing industries. A more favourable variant to achieve a bainitic microstructure is a continuous cooling heat treatment. For this heat treatment variant, the final microstructures strongly depend on the cooling rate (Mazancova and Mazanek, 1997; Wang and Yang, 1992). If the cooling rate is in a certain range, a granular bainitic microstructure is formed. A special feature of this microstructure is often the lack of carbides (e.g. cementite), because the carbon is partitioned from bainitic ferrite to the remaining austenite and stabilizes the austenite (Bhadeshia 2001; Mazancova and Mazanek, 1997). Thus, the final microstructure consists of bainitic ferrite sheaves/platelets which are separated by thin films of retained austenite and blocky islands of retained austenite or high carbon enriched martensite, which influences strongly the mechanical properties. To predict the macroscopic material properties the global and local quantification of the different apparent phases is important (Schemmel et al., 2015). X-ray diffraction (XRD) techniques offer a possibility to determine macroscopic quantities of the apparent phases

via the crystal orientation (Wiessner et al., 2014). However, due to the geometric extension of the excitation (i.e. excitation both on the surface and in the bulk of the material) this technique does not easily allow the determination of the local phase distribution; local investigations are possible, but difficult and expensive.

Etching is one of the most powerful metallographic techniques to identify phases. In most cases corrosive or colour etchants only allow to distinguish between the phases but they do not reveal grain orientation (Angeli et al., 2004). For the determination of grain orientations electron-backscatter-diffraction (EBSD) is commonly used (Pereloma et al., 2014). However, EBSD investigations are on the one hand time consuming (cost intensive) and on the other hand have to be tuned to the expected size of the microstructural effects. With the currently available equipment either a large region of several $\sim 100 \mu\text{m}^2$ is scanned with a low resolution ($> 300 \text{ nm}$ pixel size) or a small region ($\sim 1 \mu\text{m}^2$) is scanned with a high resolution ($< 20 \text{ nm}$ pixel size). Here, etching techniques have an advantage because it is a cheap and simple characterization method and with the optical light microscope (OLM) both large regions ($\sim 1 \text{ mm}^2$) and small regions ($\sim 1 \mu\text{m}^2$) can be investigated in short time on the same sample. Furthermore, correlative microscopy offers the possibility to investigate an etched surface with even higher resolution, e.g. by combining OLM and scanning electron microscopy (SEM).

Therefore, in this paper we present a technique to determine the grain orientation of a granular bainitic microstructure by means of corrosive and colour etching in OLM and evidence that no local chemical differences are responsible for the different etching attack,

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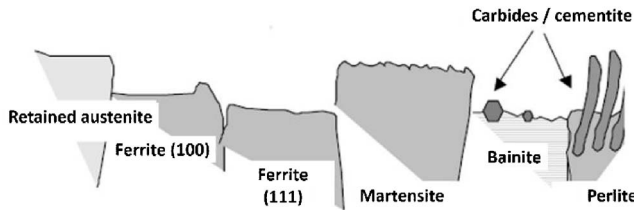


Fig. 1. Model of corrosive etching in multiphase steel taken from Angeli et al. (2004).

by means of EBSD and atom probe tomography (APT). Furthermore, the layer thickness after colour etching was determined to explain the different colours appearing on each grain orientation in the OLM.

2. Fundamentals of etching

2.1. Corrosive etching

The classical corrosive etching strongly depends on chemical composition, crystal orientation and lattice defects. In Angeli et al. (2004), they developed a model for corrosive etching in multiphase steels. They concluded that carbon rich phases (e.g. austenite) act as anodes in the electrolyte and that the carbon poor phases (e.g. ferrite) represent the cathode. Subsequently, the higher the carbon content of a phase, the higher is its resistance to the removal by etching, as sketched in Fig. 1. The influence of the crystal orientation on the chemical removal is particularly strong in carbon poor phases. In Engell (1958), the author showed in his experiments with dilute sulfuric and nitric acid on α -iron, that a crystal surface with a {111} orientation is removed two times faster than a surface with a {100} orientation. The reason for this behaviour lies in the different anodic parts of the current density potential curves of both crystal surfaces, which is more positive at a {100} surface.

2.2. Colour etching

In addition to the traditional corrosive etching methods, colour etching methods are increasingly used in metallographic laboratories because the colour contrast offers a fast and easy way to distinguish different phases in the microstructure. In the case of colour etching methods, a chemical reaction of the etchant with the substrate forms a deposit layer on the surface. Analogous to the corrosive etching methods, the different phases act as anode or cathode and different etchants form either a cathodic (e.g. molybdenum-acid) or an anodic (e.g. Klemm1-etchant) layer on the surface (Angeli et al., 2004), as shown in Fig. 2. Another analogy to the corrosive etching is that the etchant reaction with the substrate scales with the local carbon content of the phase. This time it is not the material removal that leads to a different contrast in the OLM, but the different thicknesses of the surface layer formed by the aforementioned chemical reaction, henceforth called “deposit”.

The apparent colour of the deposit in an OLM image is generated via an interference effect between metal surface and the surface of the deposit. To keep the illustration simple, we consider only a simple reflection, as schematically shown in Fig. 3. This interference effect depends on the deposit thickness d , the reflection index n of the deposit, the wavelength λ and angle of incidence of the light source. When the incident light hits the deposit, a part of the light is reflected at the surface, another part penetrates into the deposit. The penetrating light reaches the metal surface after passing through the deposit and is reflected on the deposit-metal interface. The re-passing of the deposit-air interface results in a path difference between the two reflected light rays. This path difference is proportional to the double of the deposit thickness d and constructive interference occurs when the path difference is equivalent to an integral multiple of half the wavelength

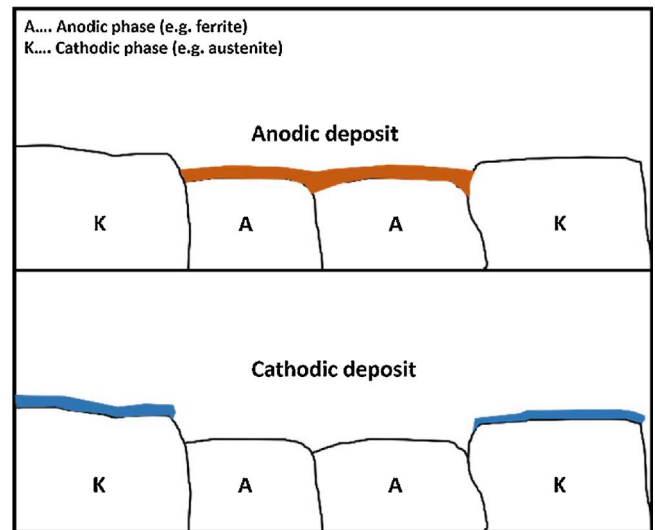


Fig. 2. Model of colour etching with anodic and cathodic deposit.

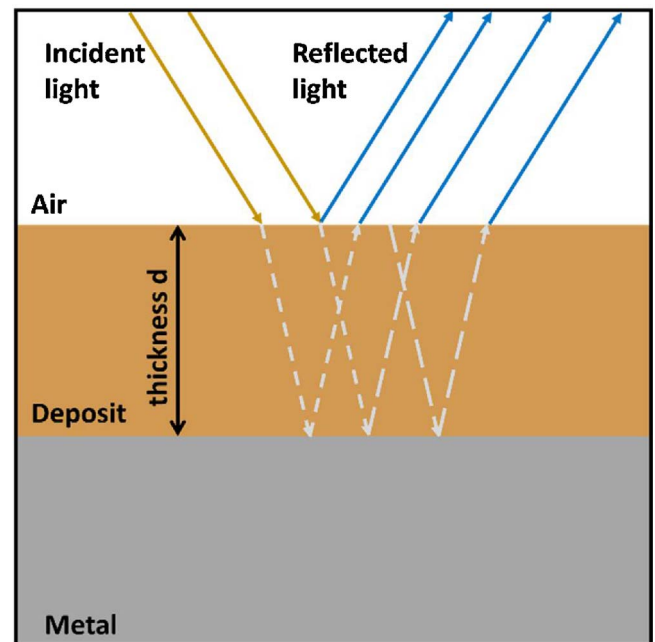


Fig. 3. Schematic diagram for light interference after colour etching.

λ (Meschede, 2015). Destructive interference occurs when the deposit thickness corresponds to an integral multiple of the quarter of the wavelength. In this case a part of the light which corresponds to the deposit thickness is removed from the incident light and the observer sees the complementary colour (Szabo and Kardos, 2010; Szabo and Bonyar, 2012). According to constructive/destructive interference, also the order of interference is important for the observed colour. In Britz et al. (2016) they presented an in situ experiment with a Beraha etchant on a dual-phase steel, which shows the colour evolution as a function of the exposure time. The authors pointed out, that during the experiment the colour passes different orders of interference. In the first order the colour changes from yellow, red, purple to a blue staining. However, in the second-order of interference the colour spectrum ranges from yellow to green. The authors also observed third-order colours, from yellow to slight red. They also concluded that higher-order colours appear brighter and more intense. This is contradictory to the findings by Gahn and Jeglitsch (1981), they demonstrated that first-order colours are the most brilliant and intense colours.

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