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Opening the Door to Fundamental Understanding of Structure and Color Metallography – a Correlative Microscopy Study on Steel

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Steel is still one of the most important engineering materials of our time. Despite over 3000 years of history, steel continues to develop into an increasingly functional material. Besides the chemical composition, its microstructure, tailored by i.e. thermo-mechanical processing, dictates the mechanical properties. A qualitative and quantitative analysis of the microstructure is thus of utmost importance. Standard metallographic procedures were applied accordingly, using chemical etching to contrast microstructures. In industrial practice these etchings were limited to empirical approaches, quickly reaching limits, especially for subsequent quantitative image analyses. Although *Nital* is the most common etching for low carbon steels, color etchings like *Beraha* [1] have enormous potential in practical metallography, lacking, however, in reproducibility and a solid knowledge base.

Based on the work of Füreder [2] and Szabo [3], the present work investigates *Nital* and *Beraha* etching with a focus on their use in current metallography, towards tailored etchings. By combining different microscopy techniques at different scales and with different contrast mechanisms, correlatively with electron backscatter diffraction (EBSD), a parameter study has been carried out to obtain a quantitative insight into the governing chemical mechanisms present during etching and to improve the contrast for even small features. The parameters were the etch composition, temperature and time. The temperature during *Nital* etching, for example, was found to control the contrast formation not only for the second phase, but also for the ferrite matrix depending on the orientation. Also, the composition and duration of a *Beraha etching* can control its nature between structure and/or color etching to get best contrasts for even small features. For a better understanding and a quantitative analysis of *Beraha* etching, a test setup was designed, which, for the first time, allows for in-situ real-time investigation of the contrasting (Figure 1). Correlative EBSD and Atomic Force Microscopy (AFM) revealed the orientation-dependent layer height (Figure 2). Focused Ion Beam (FIB) cross-sections and lamellas were used for a quantitative evaluation of the orientation-dependent thickness, and a qualitative study of the composition and structure of the layer formed by *Beraha* etching (Figure 3).

The parameter study shows how the different key parameters, such as the composition of the etch solution and the temperature, influence *Beraha* and *Nital* contrasting. Furthermore, the combined and time-resolved characterization methods as well as the targeted preparation provide qualitative and quantitative data from the interference layer formation on steels. The present work opens the door to reliable and reproducible etchings, thanks to the knowledge about the governing process, and it is the starting point for further methodological work with practical relevance.

References:

[1] E. Beraha, Practical Metallography, vol.4 (1966) , no. 8, pp. 416-418.
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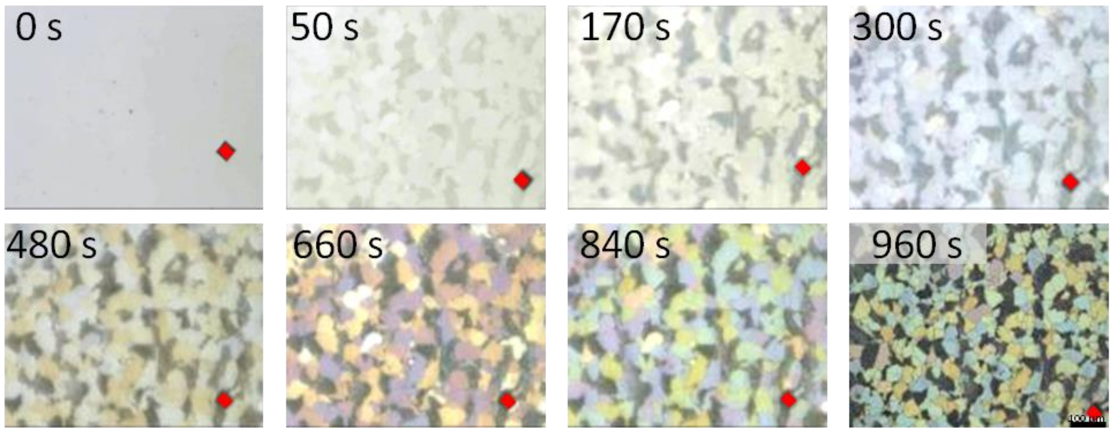

Figure 1. Time-resolved Beraha etching on steel. The microstructure is revealed step by step: second phase contrasting followed by matrix coloring. An indent (red rhombus in the right corner) was made for correlative microscopy,.

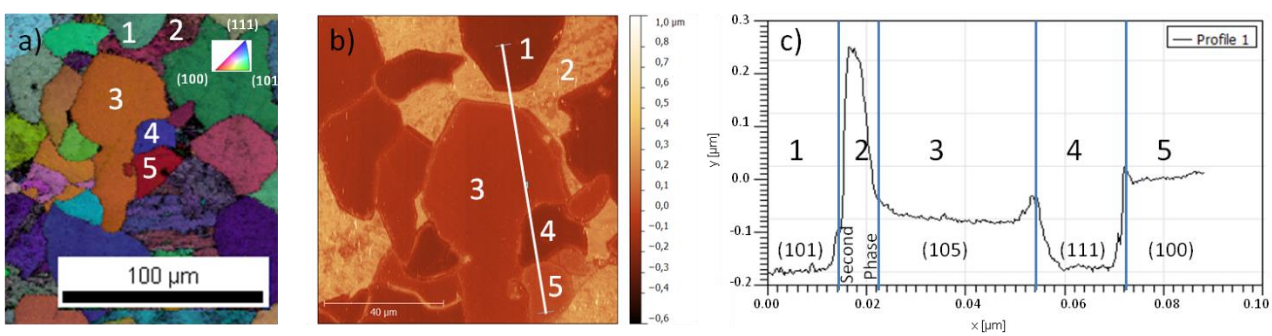

Figure 2. Correlative microscopy of Beraha-etched steel to investigate the orientation influence: a) EBSD map (IQ+IPF) before etching, b) AFM height map after etching, c) height profile along the white line in b).

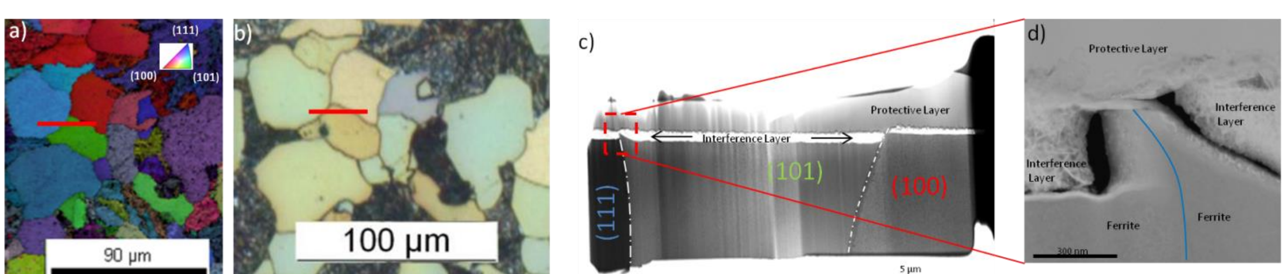

Figure 3. Correlative microscopy of Beraha-etched steel to investigate the layer formation in a certain area (red line): a) EBSD map (IQ+IPF) before etching with three main planes, b) optical micrograph after etching, c) STEM image of a TEM foil after FIB preparation with indexed details, d) STEM image of a grain boundary (blue line) at the interference layer.

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