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The Reliability of EBSD-based Microstructure Analysis

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The electron back-scatter diffraction (EBSD technique is an ideal tool to describe the multiphase nature of materials in a quantitative manner provided that the diffraction patterns of different phases can be distinguished clearly. EBSD has been widely used in past years and it still increases its popularity which is reflected as an progressive increase in the number of publications per year.

The statistical reliability of EBSD based results, despite its popularity and widely usage, remains an open question since typical observation volumes for EBSD are usually small compared to other techniques such as XRD. In particular, the tendency to use smaller step sizes for materials with a finer microstructure limits the investigation to relatively small areas in order to complete the measurements within the available (or a reasonable) time. Therefore, generalized conclusions are frequently drawn based on information obtained from a relatively small volume of the material. Particularly for heterogenous materials, scans of smaller areas quite possibly give rise to inaccuracies.

The aim of this study is to investigate the EBSD measurement parameters (i.e. step size, size of the probed and scanned areas) on the validity of phase volume fraction determination and of texture analysis. For that purpose the low-alloyed TRIP steel (described in the previous section), that contains about 10% metastable austenite in a matrix of ferrite and bainite, is used. The austenite is very heterogeneously distributed in the form of bands, mainly due to Mn segregation. This particular arrangement of austenite deteriorates the optimum mechanical properties and also makes the characterization of phase composition particularly important and difficult. In addition, the accompanying martensitic transformation in TRIP steels is highly crystallographic and makes the texture analysis critical. Therefore, the present TRIP steel is an ideal material to investigate the representativeness and reliability of EBSD datasets.

EBSD scans of various area and step sizes were systematically analyzed in order to determine the optimum sampling conditions. Moreover, Cochran's formula was used to determine a theoretical optimum sample size for predefined levels of precision and confidence. A significant deviation of theoretical optimum sample size from the experimentally determined one was found. This deviation, for the most part, is a result of the heterogeneous distribution of the austenite phase. Errors that may arise from insufficient sampling and also from false indexing of EBSD patterns at phase boundaries were also discussed. In addition, a new mapping technique, that helps to cover larger areas and probe more grains while keeping the particular advantages of a small step size, is presented. This new technique can keep the measurement time short and datasets small with minor modifications in the commercial data-acquisition software. This technique can be fully automated and it keeps the electron beam in focus even on extremely large surfaces, which is crucial for correct indexing and hence for improving the reliability of texture analysis of hardly indexed austenite in the present TRIP steel. Lastly, the comparison of results obtained by EBSD and XRD revealed that the EBSD technique is reliable and representative

provided that sampling and sample preparation are adequately done.

References:

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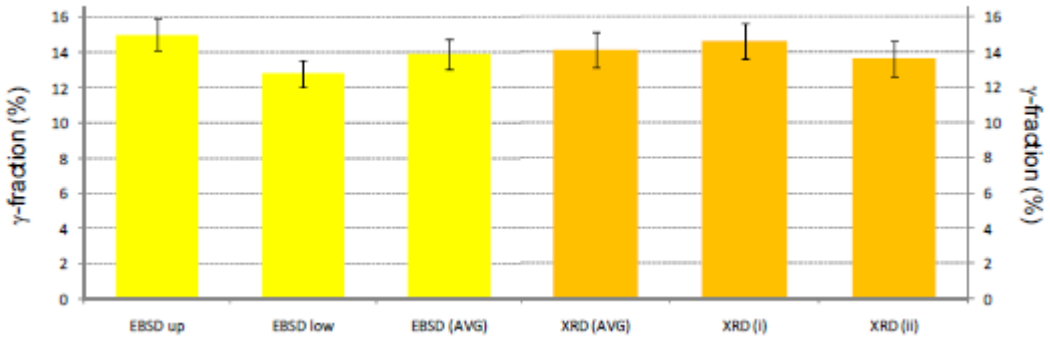

Figure 1. The austenite volume fraction of the TRIP steel obtained by EBSD and XRD techniques. The EBSD side error bars indicate the error due to possible mis-indexing. The XRD side error bars are estimated to be 1%, coming from selection of fitting parameters for Rietfeld refinement as well as texture effects.

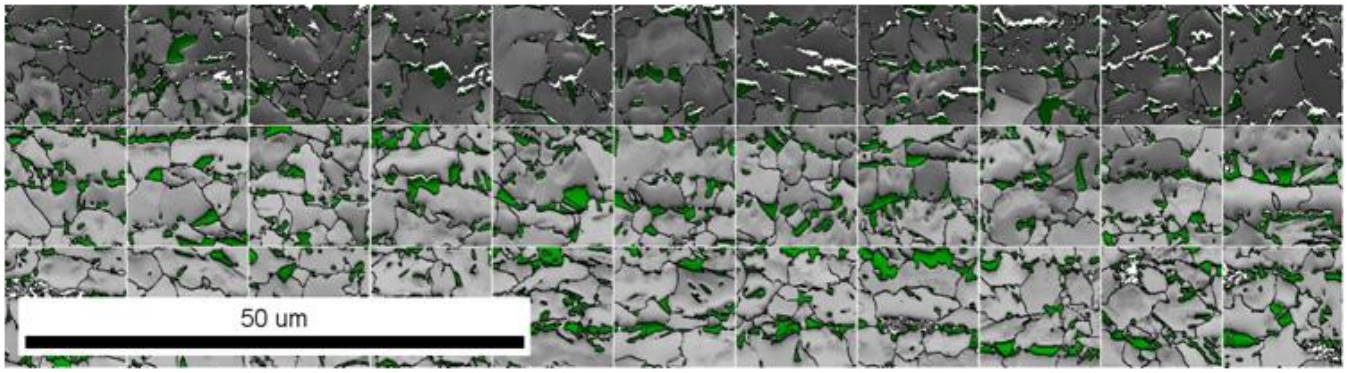

Figure 2. The pattern quality maps obtained using the new mapping technique. Note that the small maps were taken 200 μm apart from each other. For ease of post-processing and visualization these smaller high resolution maps are combined into a single larger one.

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