Accurate analysis of EBSD data for phase identification

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Abstract. This paper aims to investigate the reliability of software default settings in the analysis of EBSD results. To study the effect of software settings on the EBSD results, the presence of different phases in high Al steel has been investigated by EBSD. The results show the importance of appropriate automated analysis parameters for valid and reliable phase discrimination. Specifically, the importance of the minimum number of indexed bands and the maximum solution error have been investigated with values of 7-9 and 1.0-1.5° respectively, found to be needed for accurate analysis.

1. Introduction
Electron backscatter diffraction (EBSD) in the scanning electron microscope (SEM) is now a well established technique for examination of the crystallographic microstructure of single and multiphase materials [1]. The ability to rapidly analyse large areas of samples and the ease of specimen preparation when compared to transmission electron microscopy (TEM) is a major advantage promoting its use. However, in certain instances it can be difficult to detect and differentiate second phases which may be present in small amounts, especially when present as small or finely dispersed particles.

As part of on-going research to investigate the effect of aluminium on the microstructure and mechanical properties of low carbon, low nitrogen steels, EBSD has been employed as a technique to perform quantitative metallography. The results of this study have been discussed elsewhere [2, 3]. However, during the processing of the EBSD results, the authors have realized that by consideration of some practical points the EBSD results will be more reliable.

It should be noted that EBSD is becoming an increasingly popular characterisation technique with many researchers inexperienced in this area. It is well known that optimisation of hardware and the microscope set up such as accelerating voltage, specimen tilt and probe size together with good sample preparation are essential to obtain good quality results [1, 4]. Since EBSD data analysis is computer based to obtain reliable and accurate results we must pay particular attention to certain processing parameters such as the minimum number of indexed bands and the maximum solution error, questioning the given default values. The maximum solution error is a measure of the allowed angular error between the collected and predicted patterns i.e. the pixel will be unindexed if the angle between the pattern and solution is greater than the selected maximum solution error). In this paper the effect of these parameters on the EBSD results will be discussed.

2. Experimental Methods
EBSD investigations were carried out on a steel sample (0.02 wt% C, 1.40 wt% Mn, 0.48 wt% Al, 0.001 wt% N) which was prepared in a laboratory vacuum melt furnace as a 50kg cast and hot rolled to 13 mm thick plate, the finish rolling temperature being in the range of 1000-950°C.

The samples were prepared for EBSD by polishing on a Buehler Vibromat vibratory polisher using non-crystallising colloidal silica. EBSD was then carried out using a FEI Quanta FEGSEM from the centre of specimens with patterns detected using an Oxford Instruments camera. Patterns were analysed in real-time using the INCA software from Oxford.
Instruments. Maps were collected using different step sizes from approximately 2 µm down to 100 nm. To investigate the size of AlN particles, TEM has been employed. AlN particles were extracted by the carbon replica technique and examined using a Philips CM20 TEM operated at 200 kV.

3. Results and discussion

EBSD pattern quality and phase maps were obtained at different levels of magnification using step sizes of approximate 2, 0.5 and 0.1 µm, with analysis for the expected phases of ferrite, cementite and AlN; these are shown in figures 1a-c respectively. The default settings of the INCA crystal software were used with the minimum number of bands for indexing being 5 and the maximum solution error 2 degrees. The microstructure is indexed as being fully ferritic with some AlN and cementite particles. The corresponding pattern quality maps are shown to be of high quality. The AlN particle distributions look feasible since thermodynamic calculations performed using either the equation proposed by Seiverts [5] or the Thermocalc software suggest that the solvus temperature of aluminium nitride for this specific composition would not be lower than 1250 °C. This suggests that relatively large AlN particles should be expected within the bulk. In addition, TEM shows the AlN particle size to be approximately 500 nm (figure 2). Therefore, in theory, EBSD results obtained using a 100 nm step size can be expected to be reliable since at this step size there would be more than 4 pixels covering one particle which facilitate the phase identification process. The other issue which needs to be considered is spot size. However, since EBSD has been performed using a FEGSEM, the spot size and consequently special resolution is small.

Following the EBSD analysis of the steel which indicated the presence of AlN, both EDX mapping and backscattered imaging within the SEM were employed to add further confirmation. However, no contrast indicative of the presence of different phases within the ferrite matrix was found by BS-SEM and also no aluminium was detected using EDX. This could be attributed to the poor spatial resolution for both backscattered imaging and EDX analysis but since the attempt to show the presence of AlN by back scatter imaging and EDX
technique did not succeed it was thought that more investigation was required to confirm the presence of AlN.

To examine the reliability of the EBSD results, reprocessing of the data was performed with analysis solely for the phases, ferrite and cementite. However, the pixels previously indexed as AlN do not now appear as unindexed points but are indexed as ferrite. This could be the consequences of overlapping two patterns. On the other hand, it could be an indication of using inappropriate tolerances when processing the EBSD data. To examine the suitability of the default tolerances, the raw data were reprocessed with the expected phases being Al\textsubscript{3}Ni\textsubscript{2}, Al\textsubscript{3}Zr and the silicate mineral, Augite; these “nonsense” phases possess a range of different crystal structures and are most definitely not present in the sample. Figure 3a shows the subsequent phase map. The majority of the sampled area has been indexed as one of these three phases. Therefore, it was believed that the applied tolerances used to process the EBSD data were not tight enough to avoid mis-indexing of the data.

![Figure 2. TEM micrograph of AlN particles](image)

We believe that the two key parameters which can affect the accuracy of the pattern indexing are the minimum number of indexed bands and the maximum solution error. Figure 3b shows the effect of varying these parameters on pattern indexing. As can be seen, with the minimum number of bands set to 7 and a maximum allowable error of 2°, a significant number of data points are still mis-indexed, implying that the values of these parameters are insufficient to give reliable results. Increasing the number of matching bands to 9 reduces the number of mis-indexed pixels, and a further reduction is made by lowering the allowed angular error to 1.5°; the number of indexed points with these inappropriate phases now falls to almost zero. However, some pixels (0.3%) are still being indexed as one of these clearly incorrect phases.

Figures 4a and 4b show the examination of the same parameters to reprocess the initial sample. Based on given results the optimum minimum indexed bands can be a range between 7 and 9 and the optimum value for maximum error solution could be around 1.5. It should be noted that after reprocessing the initial data with new obtained criteria (9 minimum indexed band and 1.5 maximum solution error) negligible number of pixels indexed as AlN particles and also the indexed ratio fall to 95 %.
Figure 3. EBSD data reprocessed with the expected phases being Al₃Ni, Al₃Zr and Augite. Fig. 3a shows reprocessed EBSD data with 5 minimum indexed bands and a maximum solution error of 2° together with the relative assignments (% of the indexed pixels). Fig. 3b shows the effect of the minimum indexed bands and maximum error solution on the percentage of indexed pixels.

![Figure 3](image)

Figure 4. The effect of the minimum number of indexed bands (a) and the maximum solution error (b) on the percentage of indexed and unindexed pixels respectively as either ferrite, AlN and cementite as the expected phases.

![Figure 4](image)

4. Conclusions
The results obtained from this study show that although the importance of hardware set up can not be ignored, the used data processing criteria plays a remarkable role in making the EBSD results reliable and reproducible. In particular, we have shown the importance of the minimum indexed band and maximum error solution. Thus, treating automated EBSD as a standard “package” which will provide quick and easy data is not on its own adequate to produce good quality results. However, by employing the modified criteria the authors still failed to observe a reliable and reproducible AlN distribution by EBSD.

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References
[1] Gourgues-Lorenzon A F, Int. Mater. Rev. 52 65
[2] Palizdar Y, Scott A J., Cochrane R C and Brydson R, Mater. Sci. Tech.25 1243
[3] Palizdar Y, Cochrane R C, Brydson R, Crowther D, San Martin D and Scott A Mater. Charact. (In press)
[4] Gladman T 1997, The Physical Metallurgy of Microalloyed Steels (London: Institute of Materials)