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# Phase discrimination by pattern brightness in EBSD mappings

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ABSTRACT

A new method that allows identifying different phases with a similar crystal structure without using information from the EDS detector was developed. The method is based on the thermal diffuse scattering (TDS). Experimental tests were conducted on Mg-2Y-Zn and AA3105 alloys to validate its applicability to discriminate different phases in different alloys. The results showed clearly that the new method is more accurate and faster than other conventional methods.

## 1. Introduction

In the last decades, the electron backscatter diffraction (EBSD) technique has been used widely as a tool for process development, quality control, investigation of the orientations of crystals, the microstructure, grain sizes and distribution of lattice defects and local strains in bulk crystal of materials in the scanning electron microscope (SEM) [1–9]. Nishikawa and Kikuchi reported the observation of a diffraction pattern in backscattering mode for the first time in 1928 [10–12]. An automated pattern analysis was carried out using a Hough transform to locate band positions and band widths in the pattern and hence the interplanar spacing [4]. At the end of 20 century, fully automated EBSD systems were developed by Adams [5]. Later on, Wang explained the basic principle of formation of the Kikuchi bands [13,14] which is based on the thermal diffuse scattering (TDS) as the responsible phenomenon for the formation of the bands in the high angle scattering. TDS strongly depends on the atomic weight [15,16]. The intensity of TDS is given by:

$$I_{TDS} = (f^e)^2[1 - exp(-2W)] \quad (1)$$

where $f$ is the scattering factor that yields for a large scattering vector $s = \sin\theta/\lambda$:

$$f_k^e(s) = \frac{e}{16\,\pi^2\,\varepsilon_0}\frac{[Z - f_k^x(s)]}{s^2} \approx \frac{e}{16\,\pi^2\,\varepsilon_0}\frac{[Z]}{s^2} \quad (2)$$

Here, $f^x$ is the X-ray scattering factor and Z the atomic number. Thermal diffuse scattering becomes dominant for $s > 1.25$, which occurs at high scattering angles.

The EBSD technique has been also used to discriminate different phases in the microstructure of multiphase materials. In 1996, Goehner introduced a phase discrimination method based on the crystallographic indexing of EBSD patterns [17]. In general, phase discrimination methods are either based on the crystallographic indexing of the patterns [17–20] or simultaneously using energy-dispersive X-ray spectroscopy (EDS) data on their compositional distinction [21]. The crystallographic indexing of the patterns uses the position and intensity of the Kikuchi bands, which are unique for a particular crystal lattice and orientation [8]. In this method, the possible phases are ordered according to a user-defined weighting of indexing votes, confidence index, angular fit and bandwidth ratios. The phase with the highest ranking factor is assigned to the measured point [19,21]. The crystallographic indexing of the patterns is limited to discriminate phases with dissimilar crystal structures and close lattice constants [18].

A second method was suggested by Nowell et al. [21] and is called the Chi-method. In this, X-ray spectra of different phases are collected with an additional EDS detector.

Compared to the crystallographic indexing method, the Chi-method allows distinguishing between phases with similar crystal structures and lattice constants. However, it is imprecise due to a bigger size of the interaction volume compared to the excitation volume [21]. In 2006 Wright et al. [22] extended the ideas for different IQ measures proposed by Tao et al. [23] and reported that the contrast observed in maps constructed using the EBSD image quality mapping (IQ) arises from strain, grain boundaries, phase, surface topology and the IQ metric which describes the average height of these peaks could show the phase discrimination. However, the grain boundaries are also appeared simultaneously. In 2013, Payton et al. [24] developed a method to visualize the microstructure with installing extra forward scattering detectors (FSD) as a complimentary tool to EDS to assist phase segmentation and identification in EBSD. Recently, the latest improvement was reported by Wright et al. [2]. They reported that EBSD detectors could be used as an imaging device which allows for a great

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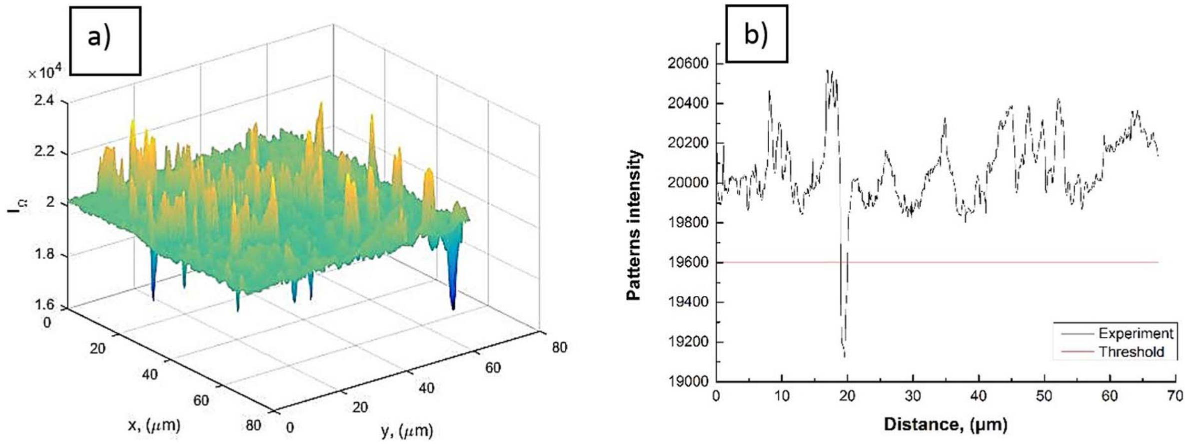


**Fig. 1.** a) A 3D map of the microstructure of an AA3105 constructed by POIB method, b) the patterns' brightness of a row of patterns during EBSD mapping with the distance. The black line is the calculated intensities of the patterns which shows the intensities of the patterns fluctuation in a row which is related to the presence of different phases in the matrix. The red line defines threshold value for the phase identification. Values higher than the threshold are considered as matrix whereas lower to the $Mg_2Si$.

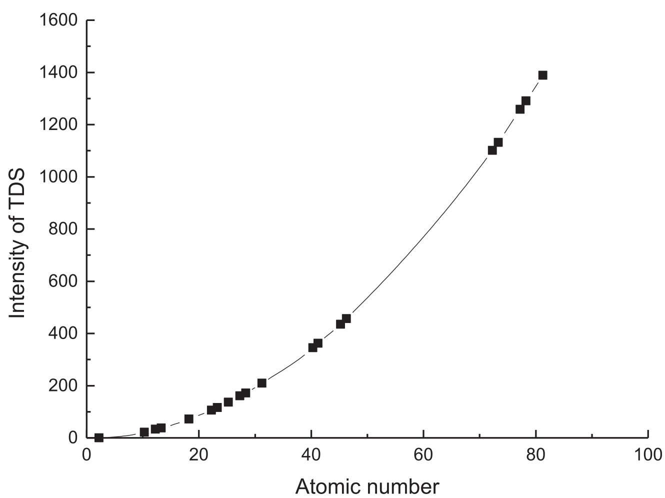


**Fig. 2.** The intensity of the TDS with the atomic number. Increasing the atomic number increases the TDS strongly.

amount of flexibility in image formation. The phase discrimination methods previously reported are limited to dissimilar crystal structures or IQ map usage or required an extra EDS or FSD. For example, in Mg-2Y-Zn alloys containing a magnesium rich solid solution and LPSO precipitates, the phases cannot be discriminated based on the crystallographic indexing as the phases have similar crystal structure and the Chi Method requires an extra EDS detector.

In this study, a new method was developed to overcome the limitation distinguishing different phases with similar crystal structures without using EDS or FSD detectors. The method is based on the evaluation of the intensities of Kikuchi patterns and will be called in the rest of the paper as a Pattern Overall Intensity Based phase discrimination method (POIB-method).

## 2. Experimental Procedure

### 2.1. Material Preparation and Experimental Procedure

The materials used in this study were a Mg-2Y-Zn and an AA3105 alloys. The magnesium samples were studied after homogenization and hot-rolling. Thus, the samples were homogenized for 16 h at 450 °C. Subsequently, rolling was conducted at 450 °C with a speed of 1500 cm/min and 50% area reduction. By contrast, the aluminum alloy was characterized only after casting. All samples were ground with 400–4000 sand paper and then polished with 3 μm and 1 μm diamond paste. The final step of preparation of the magnesium samples was done by ion beam polishing (Ar ions accelerated at 2 kV, 70° of sample tilting angle and 15 rpm of sample rotation) using a Gatan-PECS system for removal of the oxide layer. The final stage of the aluminum sample was polishing with 3 and 1 μm diamond paste. The magnesium alloy contains a long period stacking ordered phase (LPSO)·The aluminum alloy is a multiphase material, which is composed of mainly an aluminum matrix and primary precipitates, namely, $Mg_2Si$ and β-Al(Fe,Mn)Si.

The samples were examined by utilizing a cross beam XB 1540 FIB instrument (Carl Zeiss SMT AG, Germany) with EBSD-EDS (EDAX Inc., Draper, Utah, U.S.A.) detectors to collect EBSD patterns [25]. The SEM was operated at 20 KV accelerating voltage and beam current is 2.7 nA. In the experiments, exposure times were 0.1 and 0.02 s with pixel binning 470 × 470 and 96 × 96 pixels respectively. Measurements were conducted with two step-sizes, 0.250 μm for Fig. 4 and 0.040 μm for Fig. 6.

## 3. Phase Identification Method

As aforementioned, the thermal diffuse scattering depends strongly on atomic number Z. Therefore, the increase of the atomic number of the elements under a constant acceleration voltage and electron density increases the density of the diffracted electrons, which will lead to the higher brightness of the patterns. The different brightness of the patterns was used to discriminate different phases in the new method. The patterns are raw and no background subtraction were applied. The background subtraction will make a correct phase discrimination impossible, because the background subtraction changes the overall intensity of patterns. The image post processing procedure of the POIB-method can be briefly described as follows:

1. An EBSD map composed of a set of Kikuchi patterns $\Omega_{xy}$ containing single pixels $P_{ij}$ is defined. The total intensity $I_\Omega$ of a pattern is calculated as:

$$I_\Omega = \sum_i^n \sum_j^n P_{ij}$$

2. $I_\Omega$ is considered as the pattern brightness. The intensity $I_\Omega$ is mapped to the respective coordinates (x,y) of the EBSD patterns. The