Machine learning for microstructures classification in functional materials

A. K. Choudhary[1,2], A. Jansche[1], T. Grubesa[1,2], T. Bernthaler[1] and G. Schneider[1,2]

[1]Materials Research Institute, Aalen University, Germany
Beethoven Strasse 1, 73430, Aalen, Germany, amit.kumar.choudhary@hs-aalen.de
[2]Karlsruhe Institute of Technology, Karlsruhe, Germany

ABSTRACT

The search for novel magnetic phases requires efficient quantitative microstructure analysis to extract microstructural information and to correlate it with its intrinsic magnetic parameters. Kerr micrographs of magnets hold vital information for analysing the distribution of grains and magnetic domain structures in the sample and when compared to the Electron Backscatter Diffraction (EBSD) approach for grain analysis, Kerr microscopy (KM) requires less time for sample preparation, image acquisition and material analysis. However, due to the complex microstructural features, it is not feasible to use traditional approaches of image analysis for extracting this information. In this paper, we have developed a robust and time-efficient deep learning-based model for the extraction of microstructural information in the NdFeB sintered permanent magnets from Kerr microscopy images with high accuracy and compared its performance with EBSD output and with manually hand-labelled dataset prepared by a subject expert.

Keywords: deep learning, grain analysis, permanent magnets, Kerr microscopy, materials characterization

1 INTRODUCTION

Functional materials are the ones that have the ability to perform a certain 'function' under a determined stimulus. This function includes electric, magnetic and optical properties. Classification of functional materials based on their function can be grouped into magnetic materials, semiconductors, ionic conductors, superconductors, dielectrics, pyroelectrics, piezoelectric, ferroelectrics, electro-optics and ferroelectric relaxors. The knowledge of the relationships between composition, structure, processing and properties helps in the development of improved materials for known applications as well as for new uses. Materials scientists, chemists and physicists are in search of these relationships for many years by characterization of bulk properties of functional material properties and describing them with the theoretical models until recent past. Nowadays having knowledge about relationships between compositions, microstructure, processing and its macroscopic properties is the priority. [1]

One such functional material are NdFeB type magnets which are found in a wide range of applications such as power generation and transmission. The advancement of the automotive industry is highly dependent on permanent magnet motors for their hybrid and electric vehicles. Permanent magnet motors are highly efficient because of their lightweight construction and simple design. Other possible applications include wind turbine generators, e-bikes, computer hard disc drives. [2]

NdFeB magnets contain a large quantity of ferromagnetic iron and have the highest energy product among permanent magnetic materials. To get a better insight into the properties of magnetic materials, it is important to study the microstructural behaviour such as phase distribution, alignment of magnetic domain patterns and grain size distribution. During manufacturing the (Nd,RE)FeB-particles (RE = rare earth) are aligned with their magneto-crystalline anisotropy axis parallel to an external magnetic field which produces a strong preferential orientation distribution in this direction to reach the maximum possible remanence of the magnetic material. Ideally, the aligned grains are separated by a thin grain boundary phase, which forms upon liquid phase sintering and in solidified state minimizes the magnetic influence of one grain on another. During the sintering process, grain growth can occur in an undesirable manner and lead to abnormal grain growth. So, the monitoring of the grain size distribution is a part of quality assurance in magnet production. Sintered magnets can furthermore contain pores and rare earth oxide particles, which lead to the diminished volume fraction of the permanent magnetic phase and thus to lowered remanence. In the demagnetized state, the net magnetization of a grain is zero. This state is characterized by the formation of regions of uniform but opposite magnetization within the grain, which are called magnetic domains). [3]

Figure 1 shows an image acquired using a light optical microscope (LM), Kerr microscope and scanning electron microscope (SEM) with EBSD map. LM images gives better information of different phases in the alloy, KM images shows the distribution of grains and magnetic domain structures of each grain, and SEM-EBSD technique provides information on grain size distribution as well as texture information or orientation of each grain [4]. However, when compared to the LM or KM, acquiring SEM data for magnet alloys requires more effort and is time-consuming. As mentioned earlier that grain analysis is important for quality assurance and therefore an attempt has been made to obtain grain size distribution and magnetic domain information from the KM images using deep learning techniques as traditional image processing techniques fail to extract grains from such images and feature-based machine learning techniques are not robust enough to be used for different magnetic alloys. Further, KM has the advantage of being relatively inexpensive and able to handle a large range of magnetic alloys.

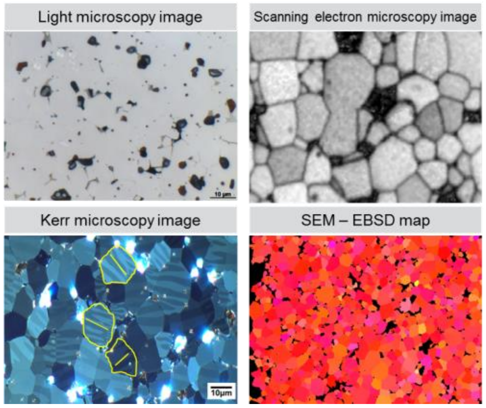

Figure 1 shows the microstructure of NdFeB sintered permanent magnet acquired using optical light microscopy showing different phases, Kerr microscopy image showing grains and orientation of magnetic domain structures highlighted in yellow, scanning electron microscope with electron backscatter diffraction map for texture or grain orientation information.

2 RELATED WORK

In the case of the KM images of NdFeB sintered permanent magnet alloys, the traditional machine learning approaches fail to classify the microstructures effectively due to the presence of magnetic domain structures with grey values in very close range. Pusch et al. [5] developed a semi-automatic approach to classify domain structures in magnet samples from correlative microscopy using Kerr micrograph and light optical micrograph. The correlative images were overlapped onto each other to find grain boundaries using threshold-based image segmentation. This approach also requires the user to prepare the sample with special attention so that grain boundaries are partially visible under optical light microscope and involves a human intervention to map partially visible grain boundaries.

In border context, grain boundary detection in materials microscopy is similar to the edge detection task in biomedical or natural images [6] [7]. There have been many state of the art DL models for such use cases and have proven to be effective on non-microscopy images of materials samples. Since these models are not trained on materials microscopy images, they fail to detect grain boundaries or edges in KM images of magnet samples.

The current approach for the grain boundary detection from KM images involves the user with subjective knowledge to manually trace the grain boundaries, apply traditional image processing techniques to post-process and quantify the grain size distribution. This approach generates results with high accuracy because of the involvement of the subject expert but consumes a lot of time and is highly dependent on human experts. This would mean that results may vary from one user to another depending on their subjective knowledge.

3 EXPERIMENTS

3.1 Materials and dataset

The dataset consists of a commercially available NdFeB sintered permanent magnet which is isotropic in nature. The sample used for experiments are acquired at 1000x magnification under a Zeiss AxioImager.M2 microscope with 10000ms exposure time and Kerr effect was visualized with a polarizer-analyser pair with 88º angle between the planes of polarization. The same sample was manually hand-labeled and a correlative EBSD map using a Zeiss Sigma 300 VP, EBSD was generated which is the reference or ground truth for comparing the performance of the developed models in this paper.

3.2 Machine learning and deep learning model for grain size analysis from Kerr microscopy images

A feature-based traditional machine learning approach was trained for the extraction of grain boundaries from Kerr images of magnet samples. This approach involved extraction of image features like HSV, RGB channels as colour features, bilateral filter, the difference of Gaussians and Sobel filters as edge features and median filter along with structure tensors as texture features. A random forest classifier with Gini function to measure split, maximum tree depth of 100 and out-of-bag score to generalize the model accuracy was tunned. This model achieved an f1 score of 0.93 but was highly sensitive to the change in the contrast of the KM images. This is because the model has been trained on hand crafted features and therefore is effective when applied on images that appear to have similar image features to that of images used for training the model.

To overcome this drawback, a U-net model with efficientnetb3 as the backbone, softmax activation function, Adam optimizer, binary cross-entropy (BCE) as loss function, Intersection over Union (IoU) and f1 score as performance metrics was trained for 250 epochs with a batch size of 4 and a learning rate of 0.0001. U-net has proven to be effective even on small datasets and we have used 17 images for training. The size of each image was 512x512 pixels and to prevent overfitting and increase robustness, the data augmentation technique was adopted. This includes transformations such as horizontal flips, affine transforms, perspective transforms, contrast manipulations, addition of Gaussian noise and image blurring. The pre-processing and post-processing steps for random forest classifier and U-net model involves the removal of artefacts and morphological operations to remove noise from predicted images respectively.

3.3 Magnetic domain structure analysis

Magnetic domain structures have a significant amount of information that affects the intrinsic properties of the magnet sample. Further, the appearance of the domain patterns is indicative of the orientation of the grains in two-dimensional space as seen in figure 2. The SEM-EBSD approach is one of the widely adopted tools for measuring the orientation of grains in the magnet samples. [4] The effective detection of grain boundaries using the developed model assists the user to have a closer insight into each grain and hence, it is feasible to analyse magnetic domain structures from KM images.

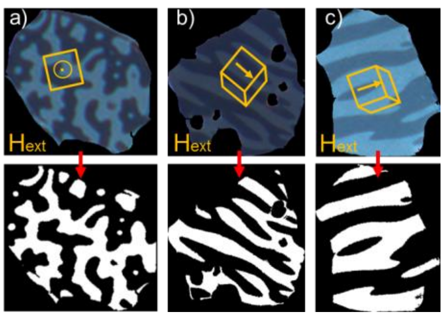

Figure 2 shows the extracted grains from the magnet sample and the magnetic domain structures as binary image. a) grain with closure domain structure and having preferential theta axis pointing out of the plane, (b) and (c) grains with stripe domain structure with theta axis pointing along the plane.

A supervised regression model was trained on features extracted from the grains and their magnetic domain structures to predict the orientation of grains within the sample. Haralick parameters [8] as texture features were extracted from each grain. Further, the domain structure from each grain was extracted using automatic threshold-based image segmentation followed by measuring the morphological parameters such as area fraction, aspect ratio and longest domain pattern within the grain. In addition to these morphological parameters, shape features such as Zernike moments [9] were calculated for patterns in each grain. The ground truth or labels for training the model were obtained from the correlative EBSD maps that give information on the orientation of each grain. The orientation of each grain from EBSD is represented as phi1 ($\phi 1$), phi (φ) and phi2 ($\phi 2$). For the experiments here, the new coordinate system which excludes phi2 was adopted because the information needed to learn phi2 from 2D Kerr images was not available. In the new coordinate system the range of phi1 has been reduced from 0º-360º to 0º-180º and it is referred to as theta (θ). Whereas, phi orientation ranges from 0º-90º and it has been transformed to rho (ρ) but has the same range as phi. The regression model is trained to predict theta and rho for each grain in the sample.

4 RESULTS AND DISCUSSION

To measure the effectiveness of the developed models statistically, the results from ML and DL approach was compared with reference data. The reference data includes hand labelled results by a subject expert and an EBSD map generated using correlative microscopy.

4.1 Grain size analysis

Figure 3 shows the area-weighted and number-weighted grain size distribution based on equivalent circle diameter (µm) for test sample NdFeB using a trained random forest classifier, a trained U-net model and the manual approach. Additionally, we have the grain size distribution from EBSD for the same sample to evaluate the accuracy of the manually generated results as it is the reference or ground truth. It is observed that the area under the area-weighted distribution and frequency-weighted curve between the manual and EBSD approach is 1.4 %, and 0.5 % respectively. The difference is less than 2 % and therefore having a manual approach as a reference for comparing the performance of trained models is productive because generating a correlative EBSD map is a challenging and time-consuming task.

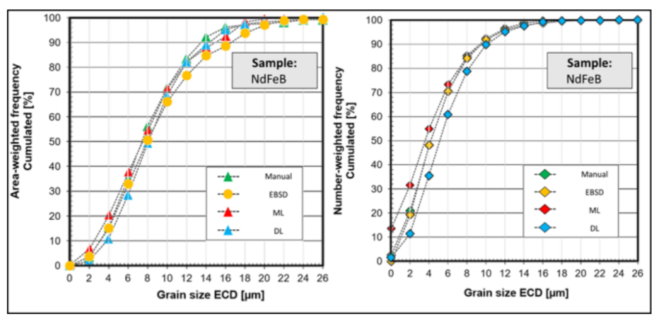

Figure 3 shows the number-weighted and area-weighted grain size distribution curve obtained from the manual approach, the EBSD approach, the trained random forest classifier and the trained U-net model for the NdFeB test sample acquired at 1000x magnification under Kerr microscope.

The random forest classifier model differs from the reference by 1.9 % and the U-net model by 1.5 % for the area-weighted grain distribution curve. Similarly, the deviation of the number-weighted grain distribution curve is 2.5 % for the random forest model and 1.9 % for the U-net. The performance of both models is close to the reference data with the U-net model having better performance than the random forest classifier. However, it has been observed that the trained feature based ML approach is highly sensitive to the contrast variation in the acquired image and also involves the user to frequently update the training dataset to make it work effectively on different magnetic alloys. In the case of the developed U-net model, the information required to train the model in the form of image features is performed automatically by the model and with the use of data augmentation, the robustness of the model to perform on contrast varying KM images is achieved.

4.2 Magnetic domain structure analysis

Figure 4 shows the comparison between the orientation of grains obtained from the trained regression model and the EBSD approach. The theta and rho for each grain in the sample are obtained and plotted as cumulative and absolute frequency distribution curves with the calculation of the cumulative distribution function (CDF) from the probability distribution function (PDF). The difference in the interquartile distance (IQD) , Q25, Q50 and Q75 between measured EBSD and predicted values (theta and rho) suggests that the error is less than 10 %.

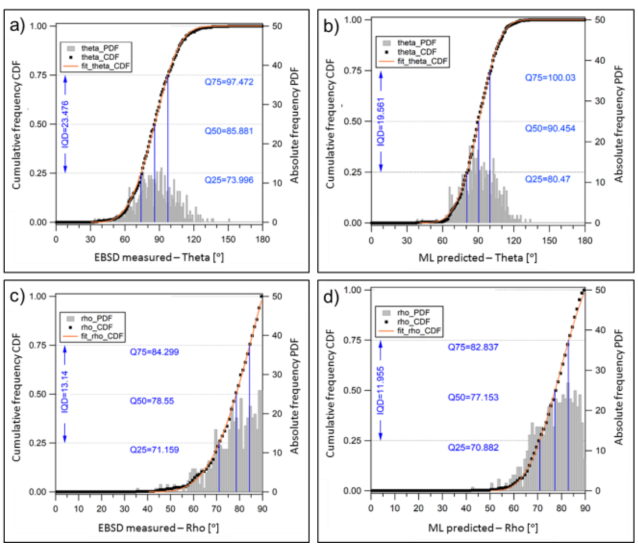

Figure 4 shows the comparison between the measured EBSD theta (θ) and rho (ρ) values against the predicted values for NdFeB sample. The IQD, Q25, Q50 and Q75 values are displayed for direct comparison for values.

Further, when compared to theta the predictions for the rho values are in very close range to the measured EBSD values. One of the factors which are responsible for the low precision for predicted theta values is the presence of closure domain structures in the sample. The regression model developed has limitations when it comes to the closure domain structures as it performs with less confidence on grains with closure domain structures. This is because the features used for training the model are biased towards stripe domain structures and therefore more information on closure domain structures is needed to create an unbiased training dataset for the orientation prediction model. However, the developed method can be adopted for analyzing the sample with stripe domain structures.

5 CONCLUSIONS

In this paper, we have explored the possibilities of using a traditional feature-based machine learning approach and a advanced deep learning approach for automated grain size analysis and predicting the orientation of detected grains from Kerr microscopy images. The trained U-net model has achieved good accuaracy in terms of IoU score of 0.94 and proved to be robust against the different NdFeB permanent magnetic alloys. The major advantage of such a model is that it requires less time for analysing the large samples when compared to EBSD and the manual approach.

Further, the regression model to predict the grain orientation with the help of domain structure information has shown that it is feasible to get first hand information of the orientation of garins in large samples with Kerr microscopy images. Improving the prediction accuracy of theta values by incorporating the more information to the regression model in the form of additional feature set has been identified as the part of further research.

REFERENCES

[1] P. M. Vilarinho, Scanning probe microscopy: Characterization, Nanofabrication and Device Application of Functional Materials, 3-33, 2005.
[2] S. M. Yusuf, Functional Materials, 111-154, 2012.
[3] Alex Hubert and Rudolf Schäfer, Magnetic Domains: The Analysis of Magnetic Microstructures, 11-19, 1998.
[4] D. Hohs, T. Grubesa, D. Schuller, D. Goll and G. Schneider, Microscopy and Microanalysis, 23, 596-597, 2017.
[5] V. Pusch, T. Bernthaler, F. Köppen, A. Wilde, A. Nagel and G. Schneider, Practical Metallography, 42, 171-176, 2010.
[6] S. Xie and Z. Tu, IEEE International Conference on Computer Vision, 2015.
[7] Gedas Bertasius, Jianbo Shi, Lorenzo Torresani, IEEE Conference on Computer Vision and Pattern Recognition, 2013.
[8] R.M. Haralick, K. Shanmugam and T. Dinstein, IEEE Transactions on Systems, Man and Cybernetics, 3, 610-621, 1973.
[9] Khotanzad and Y.H. Hong, IEEE Transactions on Pattern Analysis and Machine Intelligence, 12, 489-498, 1990.