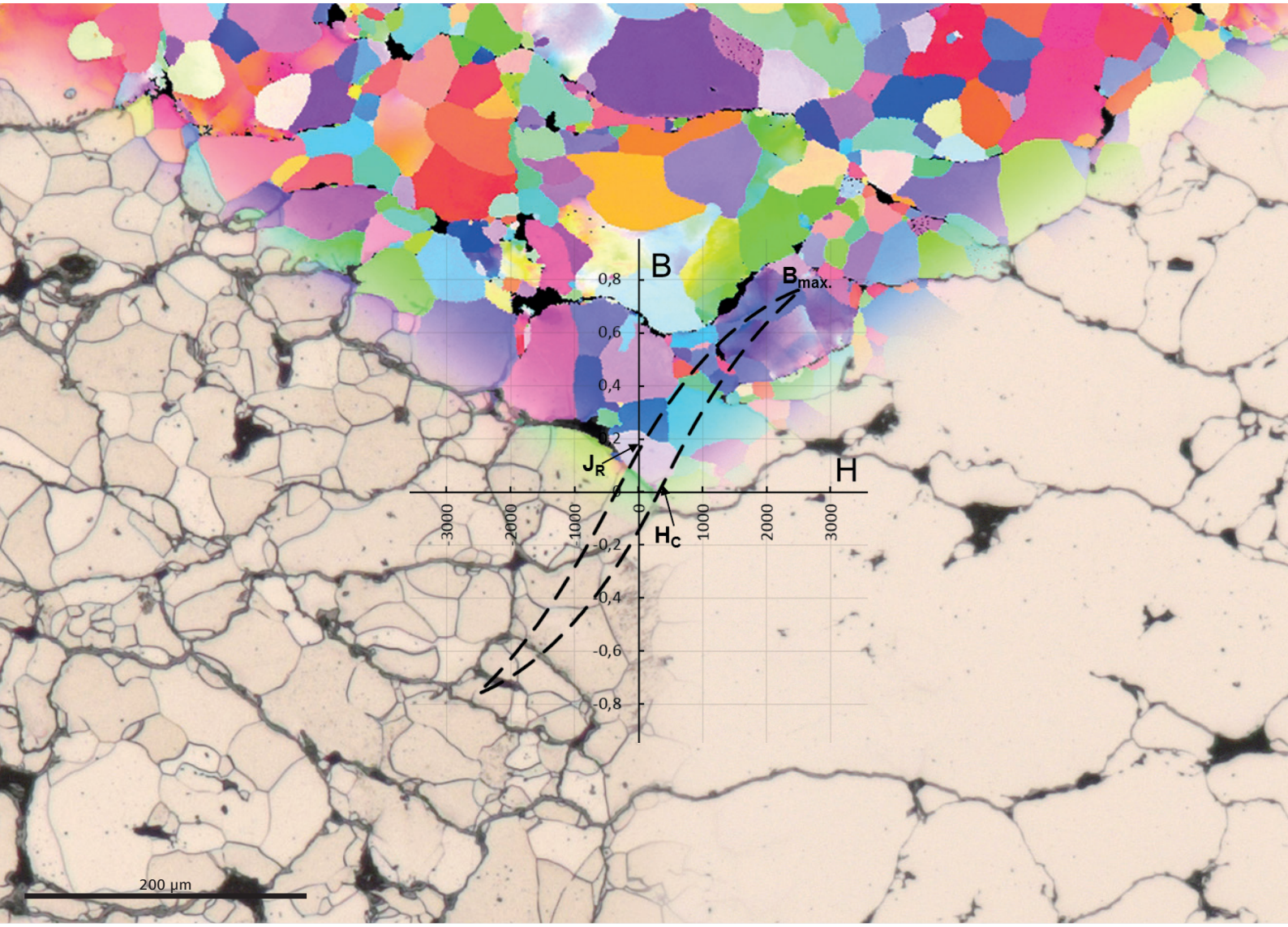

ZEISS Sigma 300

Quantitative EBSD Studies of Soft Magnetic Composites

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Authors: David Schuller, Dominic Hohs, Tvrtko Grubesa, Tim Schubert, Dr. Timo Bernthaler, Prof. Dr. Dagmar Goll, Prof. Dr. Gerhard Schneider
Aalen University, Materials Research Institute, Germany

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The trend in electric motor development is moving towards high magnetic induction and high-frequency designs. When selecting a core material, soft magnetic composite materials are a useful alternative to electrical steel sheets and offer significantly lower dynamic losses as a result of an electrical insulation coating on the iron particles that have been pressed in a powder metallurgy process. Electron backscatter diffraction (EBSD) makes it possible to determine all relevant microstructural information, such as grain size, grain orientation, and plastic deformation of the material. This makes EBSD using the ZEISS Sigma a powerful analytical method for the future development of the material.

Introduction

Soft magnetic composites (SMCs) is the collective designation for iron-based soft magnetic composite materials that are created through a powder metallurgy process. The impressive nature of SMCs is characterized by significantly lower loss characteristics in the frequency range of > 2,000–10,000 Hz, which makes them an ideal alternative to the electrical steel sheets commonly used in automotive applications [1]. Their maximum permeability (μ_{max}) of around 500–1,000 and magnetic induction (B) of 1.6 T at 10,000 A/m, however, is lower than that of electrical steel sheets ($B_{10,000\ A/m}$ = 1.9 T, $\mu_{max} \geq$ 10,000). Even the hysteresis losses, which take on a dominant role in the frequency range of < 1,500 Hz, are around 5 times greater. As such, a thorough understanding of the microstructure of SMC materials is imperative to reduce hysteresis losses, increase both permeability and induction and will play a key role in their application in automotive electric motors of the future.

In 2014, the global market for soft magnetic materials amounted to around 45 billion US dollars. It is expected to grow by around 30 % to 65 billion euros by 2019.

Non-oriented electric sheets take up the largest share of the market at 81 %, while grain-oriented electric sheets come in at second place at 14.5 %. The remaining 4.5 % share is covered by other soft magnetic materials, which include amorphous soft magnets, ferrites, and highly silicated material variants with 6.5 % silicon as well as the SMCs.

Manufacturing process

SMC materials are manufactured using powder metallurgy processes and, as a core material of electric motors, offer three-dimensional properties and significantly lower eddy-current losses compared to the commonly used electrical sheets in high field/high-frequency applications [1]. The material used most in these applications is water-atomized pure iron, which has excellent compaction properties due to its round shape. To reduce dynamic losses at high frequency, the particles are also coated with an electrical insulation layer made of phosphorus oxide. Furthermore, the coated particles are made up of many individual grains as part of the water atomization process, as a result a distinction between particle size and grain size distribution must be clearly stated [2].

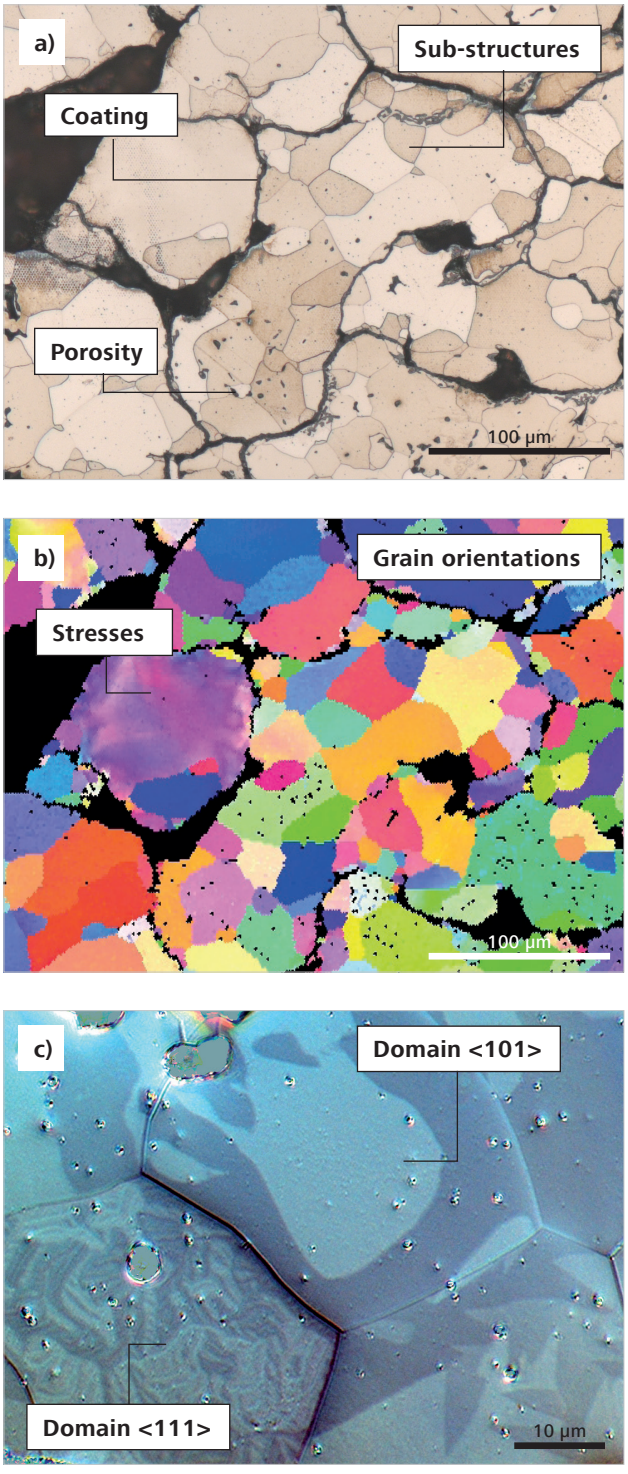

Figure 1 *Investigations into a heat-treated SMC: a) light microscope (brightfield) after etching, b) EBSD (inverse pole figure) before etching, and c) Kerr microscopy before etching: 200× and 1000× magnification respectively.*

During the pressing process (between 600 and 800 MPa as standard) for forming SMC components, the particles or grains are deformed and stresses are introduced. The microstructure state is recrystallized and annealed, hysteresis losses are

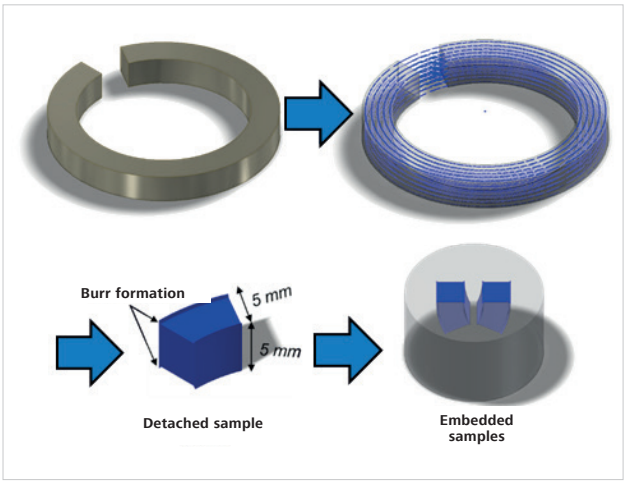

Figure 2 *Schematic representation of the sample size and orientation of the segments.*

minimized, and the level of permeability increases depending on the press force and heat treatment [3].

Figure 1a illustrates the differences between the particle and grain size distribution in the brightfield on an etched sample. With the use of EBSD, the sub-structure within the iron particles can be detected without etching, and additional statements can be made regarding the orientation and the deformation and stress states of the grains (see Figure 1b). Investigation of the domain structure using Kerr microscopy techniques makes it possible to carry out investigations into the structure and the motion behavior of domain walls under field (see Figure 1c). EBSD measurements allow for the quantitative collection of crucial microstructure information, which has an impact on magnetic properties. In addition to the particle size distribution, grain orientation, and evaluation of misorientations, these measurements represent an evaluation of the deformations [4].

Sample Selection

This investigation involved commercially available SMC materials. At a pressure of 800 MPa, all samples were pressed into ring components with an outer and inner diameter of 55 and 45 mm respectively and a height of 5 mm. The samples were then measured magnetically. To analyze the microstructure, segments of the rings were separated into sample cross sections and prepared using metallographic methods (Struers RotoPol-31). It was essential not to cause any damage to the surface at this stage. EBSD is a highly surface sensitive process and, roughly, only the top 40 nm are measured [4]. It is for this reason that the final preparation phase involves vibration polishing, electropolishing, or ion polishing. The samples were

post-infiltrated to avoid breakouts and the resulting scratches. All materialographically evaluated ring samples have the same pressing direction orientation as determined by the production process (see Figure 2).

Hardware and image acquisition

The EBSD measurements for determining grain orientation, texture, and size were recorded using a ZEISS scanning electron microscope (ZEISS Sigma 300 VP). The EBSD analysis involved the use of an EDAX camera and software (EDAX Hikari, OIM v7.2.1 (orientation imaging microscopy), while the EBSD measurements were taken with an acceleration voltage of 20 kV, a step size of 1 µm, and a magnification of 100× (screen size approx. 0.96 mm²). Furthermore, only grains with over 10 measuring points were taken into consideration (corresponding to an area of 40 µm²), which are also entirely visible within the measuring surface. The influence of settings such as step size, exposure time, and magnification has a significant impact on the EBSD analysis and therefore must be adjusted to suit the issue and material in question. The EBSD analysis provides information on the image quality (IQ), which evaluates the signal quality of every measuring point, and on the inverse pole figure (IPF) (see Figure 3). While the image quality makes it possible to quantify the elastic component of lattice stresses by means of evaluating the diffraction pattern, evaluations of the misorientations are based

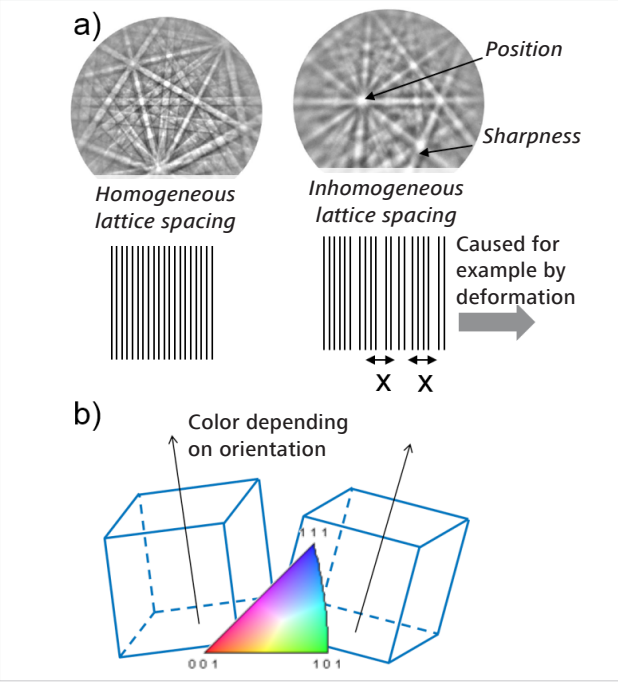

Figure 3 *EBSD measurement results: a) image quality (evaluation of signal quality), b) determination of orientation (evaluation of the measuring point orientation).*

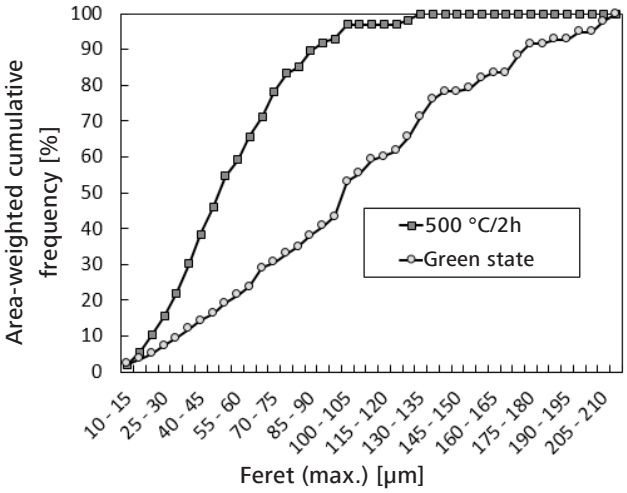

Figure 4 *Cumulative area-weighted frequency of the grain size distribution in the green state and in a heat-treated sample (500 °C / 2 h).*

solely on plastic deformation components. The orientation distribution makes it possible to establish structural information on the size and shape of a grain.

Determining the Grain Size Distribution via EBSD

The grain size distribution of SMC samples in relation to their heat treatment temperature was investigated by means of EBSD (see Figure 5). This allows recrystallization effects to be investigated within the strongly deformed particles, and even very small particles of > 10 µm can be detected with ease. By comparing the green state and the states following heat treatment at 600 °C and 800 °C respectively, Figure 5 clearly illustrates that the number of grains increases as a result of recrystallization and the new formation of grains. Furthermore, at a temperature of 800 °C, there is an increasing disintegration of the insulation coating. This is illustrated by the iron particle boundaries becoming difficult to detect and the increasing sintering of the particles. Figure 4 shows an example particle size distribution for a green-state SMC material and the occurrence of recrystallization and grain refinement caused by subsequent heat treatment at 500 °C. The heavily deformed grains, which are therefore subject to a high dislocation density, highlight that the first recrystallization processes start to occur at 500 °C. The optimal range for minimizing hysteresis losses during recovery and recrystallization, while simultaneously minimizing the weakening of the insulation coating, can be determined by means of EBSD analysis on the basis of the grain structure.

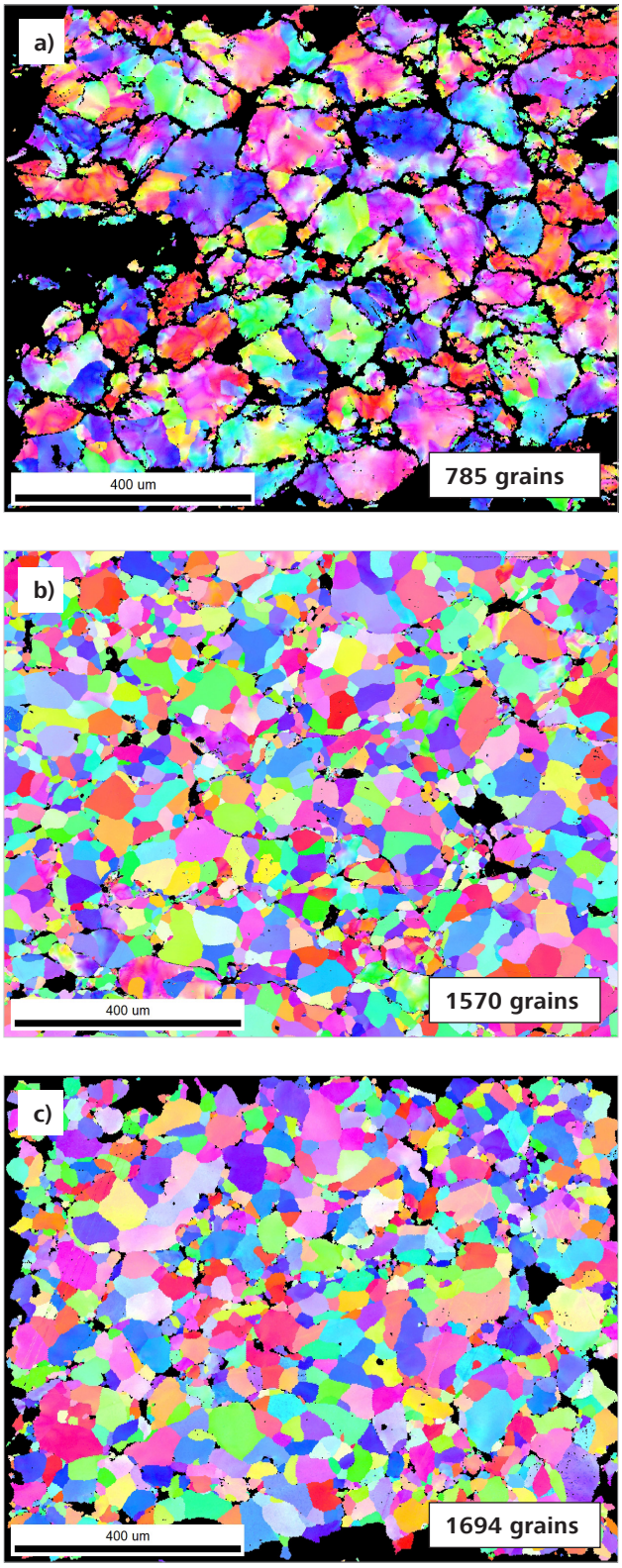

Figure 5 *Determination of the grain size distribution in SMC materials: a) green state; b) 500 °C, and c) 600 °C.*

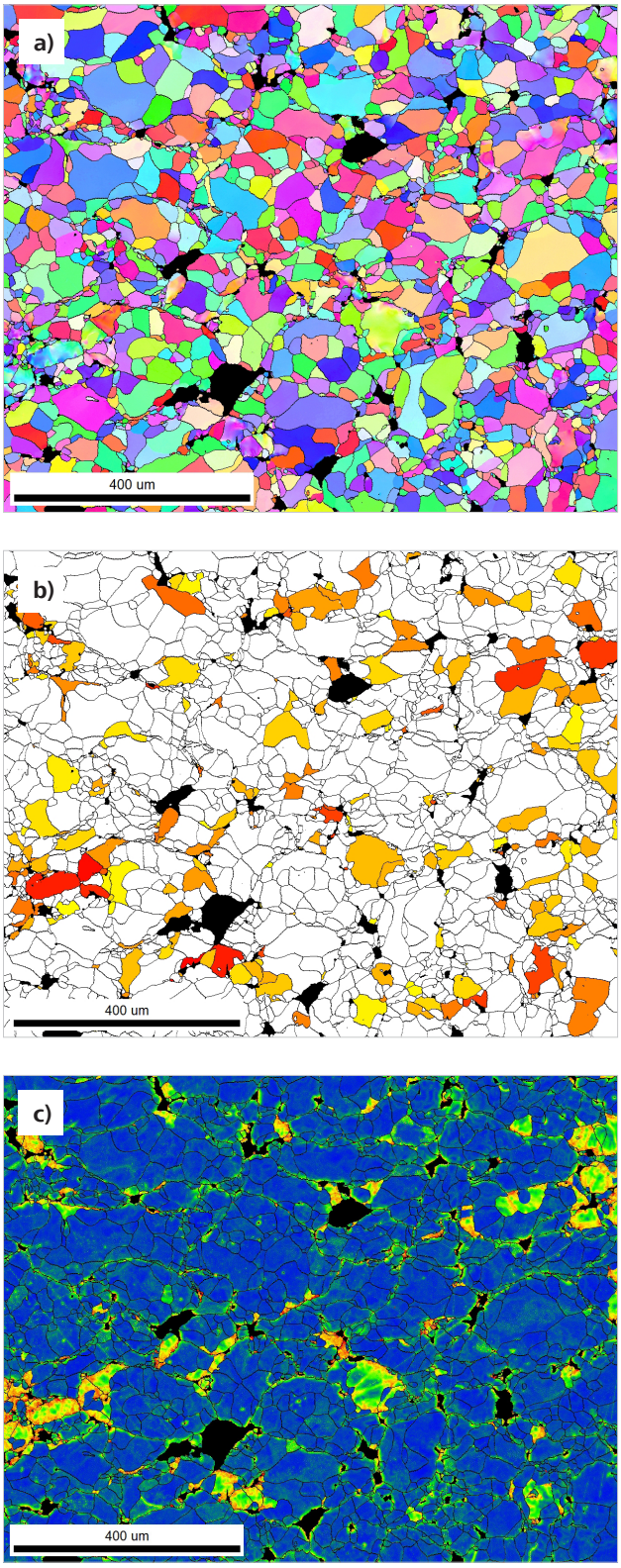

Figure 6 *Comparison of various analysis tools for evaluating misorientations within a grain in SMC materials: a) inverse pole figure (IPF), b) grain spread misorientation (GSM), and c) kernel average misorientation (KAM).*

Determining the Deformation Status via EBSD

To quantitatively determine the misorientations within the individual grains in SMC materials, it is crucial to select a suitable range in the first step in order to assess the orientation from which a deformation influence exists. The misorientations could also be due to factors such as a natural fluctuation effect within the grains, differences in orientation between adjacent grains or grain boundaries, or the impact of preparatory work, thus distorting the measurements. It is therefore crucial to define a suitable measurement reference with known properties [4]. In this case, this may be a fully annealed or heat-treated specimen, or even the starting powder before compression. While there is a scattering of the orientation within a grain in diffuse areas, indicating misorientations, in the unprocessed inverse pole figure (IPF) (Figure 6a), it can still be clearly quantified using special analysis tools. The grain spread misorientation (GSM) tool in 6b and the kernel average misorientation (KAM) tool in 7c are two such examples. These tools make it possible to carry out various types of assessment and quantification of misorientations. While GSM involves every grain with misorientations in the defined area being completely colored, the KAM tool evaluates a defined number of pixels in the vicinity of a misorientation, thus allowing far more detailed interpretation of deformations within the particles.

Outlook

The investigation into the microstructure of SMC materials using EBSD, depending on the manufacturing process and its impact on the grain shape, size, deformation, or annealing, and thus magnetic performance, provides essential information for the in-depth understanding and further development of these materials of the future. The combination of conventional microscopy, Kerr microscopic analysis, and EBSD allows all relevant influences to be analyzed at a microstructural level and systematically applied to the custom tailoring of soft magnetic composite materials.

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Carl Zeiss Microscopy GmbH
07745 Jena, Germany
microscopy@zeiss.com
www.zeiss.com/sigma