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## Determination of Bulk Magnetic Volume Properties by Neutron Dark-Field Imaging

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### Abstract

For the production of high-class electrical steel grades a deeper understanding of the magnetic domain interaction with induced mechanical stresses is strongly required. This holds for non-oriented (NO) as well as grain-oriented (GO) steels. In the case of non-oriented steels the magnetic property degeneration after punching or laser cutting is essential for selecting correct obstructing material grades and designing efficient electrical machines. Until now these effects stay undiscovered due to the lack of adequate investigation methods that reveal local bulk information on processed laminations. Here we show how the use of a non-destructive testing method based on a neutron grating interferometry providing the dark-field image contrast delivers spatially-resolved transmission information about the local bulk domain arrangement and domain wall density. With the help of this technique it is possible to visualize magnetization processes within the NO laminations. Different representative manufacturing techniques are compared in terms of magnetic flux density deterioration such as punching, mechanically cutting by guillotine as well as laser fusion cutting using industrial high power laser beam sources. For GO steel laminations the method is applicable on the one hand to visualize the internal domain structure without being hindered by the coating layer. On the other hand, we can show the influence of the coating layer onto the underlying domain structure.

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## Introduction

Manufacturing of electrical machines, such as fans, pumps and industrial motors, tends to various machine types and design variants. Adjusting machine design, the choice of correct material grade as well as the introduction of innovative manufacturing technologies gives an important contribution to fulfill upcoming IEC standard regulations for efficiency classes for electric motors. Unfortunately mentioned manufacturing technologies are only rated in terms of monetary aspects due to lack of adequate investigation methods. The deterioration of magnetic sample properties during manufacturing is commonly being investigated using rectangular or toroidal samples in magnetic measurement units [1-3]. Some further research has been carried out through analyzing microstructural changes and resulting increase of micro hardness after mechanical punching and laser cutting. In conclusion, the existence of two magnetic deterioration mechanisms was pointed out [4]. Nevertheless, the relation between the magnitude of mechanical or thermal deterioration and the resulting macroscopic magnetic property modification, i.e. the change of permeability as a function of the distance from the cutting edge, stays undiscovered.

Nowadays the magnetic behavior of soft magnetic materials can be characterized by means of magneto-optical-Kerr-microscopy (MOKE) which is sensitive to surface domains [5]. The bulk domain arrangement, however, is responsible for the macroscopic magnetic properties. Here the surface sensitive MOKE technique finally fails. Neutrons have the advantage to easily penetrate centimeter thick metallic samples and its magnetic moment makes the neutron sensitive to magnetic materials and hence to investigate domain structures in bulk materials.

A new neutron imaging techniques based on a neutron grating interferometer (nGI) setup that delivers insight into bulk magnetic domain structures was recently developed at PSI. Detailed information can be found in the following references [6-8]. This setup delivers the so-called neutron dark-field image (DFI) and relies on diffraction gratings [9]. With this setup a field of view of 64 mm x 64 mm can be reached with a spatial resolution down to 50  $\mu\text{m}$ . The image contrast in the DFI for magnetic samples is based on scattering of unpolarized neutrons at magnetic domain walls inside the sample. The neutron beam undergoes multiple refractions at magnetic domain walls in the sample, which results in a local degradation of the coherence for neutrons exiting the sample. This local degradation decreases the ability of the neutrons to interfere with each other and leads to a local decrease of the signal. A detailed description of the dark-field image contrast can be found in [10].

So far the nGI technique was used on the one hand to investigate bulk magnetic domain structures [7], and on the other hand to study bulk magnetization processes [6]. The relaxed requirements on spatial and temporal coherence of the grating interferometer setup lead to exposure times that are comparable to other "non-neutron" domain observation techniques. The efficiency of our setup, with total exposure times of typically few minutes per DFI, allows us to study the dynamic response of the specimen under the influence of an externally applied magnetic field.

In the presented paper the nGI method was applied to visualize

- (1) the geometry dependent volume magnetization processes in a steel plate
- (2) the domain structures of GO laminations and coating influence
- (3) the influence of different manufacturing techniques for NO laminations in terms of magnetic property deterioration and its spatial spread [11]

## Experimental results

The DFI results of the nGI experiments were all carried out at the spallation neutron source SINQ at the Paul Scherrer Institut (PSI) using the beam port of the cold neutron imaging facility ICON [12]. Neutrons having a wavelength  $\lambda = 4.1 \text{ \AA}$  and a wavelength spread of  $\Delta\lambda/\lambda = 15\%$  provided by a velocity selector. The L/D ratio was 350. The nGI setup was combined with a state of the art neutron imaging detection system. The images were recorded using a 100  $\mu\text{m}$  thick  $^6\text{Li}/\text{ZnS}$  scintillator screen and a cooled charge coupled device (CCD) [Andor Neo sCMOS, 2560x2160 pixels, pixel size: 6.5  $\mu\text{m}$ ]. The effective spatial resolution of the camera setup of 100  $\mu\text{m}$  was determined by the intrinsic blurring of the scintillation screen.



## 2.1 Visualization of geometry dependent magnetization processes

The sample was an untreated poly-crystalline steel plate [steel grade: DC 01 (St 12.03)] with an edge length of 15 mm. The plate had a thickness of 750  $\mu\text{m}$  and was mounted with plastic screws on the front of the sample holder made of aluminum. The sample holder is built-in in the setup on a three axis positioning system to accurately place the sample centered between the cylindrical pole shoes of an electromagnet. The poles gap was 40 mm producing an almost uniform horizontal magnetic field in air up to 250 mT.

The sample can be rotated around the beam axis by an angle, to study the geometry-dependent magnetization processes. The magnetization process of the steel plate for the  $0^\circ$  orientation is shown in Fig.1 (top row). In zero field configuration,  $H = 0$  mT, the steel plate is clearly and fully visible in the DFI due to the rich multi-domain structure in the specimen. The bright color corresponds to domain wall rich areas, whereas red corresponds to domain wall free areas. When the magnetic field is increased to  $H = 88$  mT, contrast starts to vanish. At field values of  $H \geq 100$  mT, a vertically elongated region in the middle of the plate becomes visible.

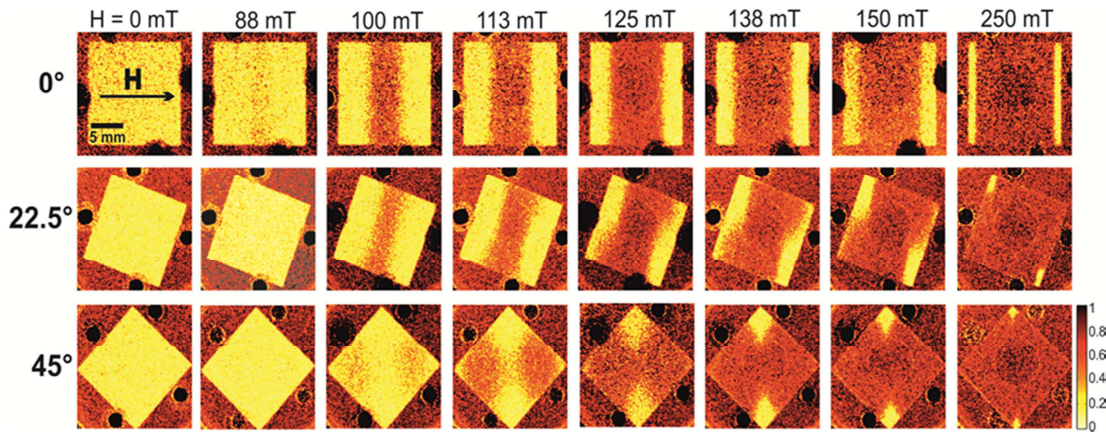


Fig. 1: Neutron dark-field images of the geometry-dependent magnetization process of the poly-crystalline steel plate for different orientations  $0^\circ$ ,  $22.5^\circ$  and  $45^\circ$ . Different magnetization behaviors and starting points of the propagation of the volume magnetization for each orientation are observed. The bright color in the DFIs indicates domain-wall-rich areas.

In this region the domain wall density is strongly reduced. For increasing field values between  $113 \text{ mT} \leq H \leq 150 \text{ mT}$ , this area expands toward the sample edges, gaining rapidly in width. For the maximum external field value of  $H = 250 \text{ mT}$ , the larger part of the plate is magnetized beside two small vertical domain-wall-rich strips at the edges.

For the  $22.5^\circ$  orientation, the steel plate is rotated clockwise. When the magnetic field is increased to  $H = 100 \text{ mT}$ , the contrast recovers, forming a rectangular, vertically inclined stripe in the middle of the plate. The starting position in the middle of the sample is similar to that of the  $0^\circ$  case. However, the starting point of the contrast development is delayed. At field values between  $113 \text{ mT} \leq H \leq 150 \text{ mT}$ , the rectangular area in the middle expands again toward the sample edges and gains rapidly in width. However, at  $H = 125 \text{ mT}$ , the rectangular area collapses and for field values between  $125 \text{ mT} \leq H \leq 150 \text{ mT}$ , the area expands towards the sample corners, which are closer to the pole shoes (top right, bottom left), until these corners are fully magnetized. At maximum field, two small domain wall-rich rectangular areas are remaining on the top left and bottom right corners.

For the  $45^\circ$  orientation, the steel plate is oriented with one diagonal along the magnetic field axis. Up to field values of  $H \leq 88 \text{ mT}$ , the steel plate is completely visible in the DFI. For the following magnet field step at  $H = 100 \text{ mT}$  the contrast starts to vanish again, in the form of two mirror-symmetrical vertically elongated areas. At  $H = 113 \text{ mT}$ , two oval grainy areas are visible, although the horizontal edges close to the pole shoes are still not magnetized. For field values between  $113 \text{ mT} \leq H \leq 150 \text{ mT}$ , these two oval areas expand towards the middle part of the plate as well as to the edges close to the pole shoes. The area of the rhombus decreases for increasing



external field values. For field  $H = 250$  mT, the remaining rhombohedral domain wall-rich areas are found at the top and bottom corners.

## 2.2 Domain structures of GO laminations and coating influence

Grain oriented (GO) electrical steel is a soft magnetic material for applications that require low core losses and high permeability like distribution and power transformers. This silicon steel is manufactured in very complicated processes to achieve special alignment of the grains during the rolling process, the so-called Goss-texture with  $\{110\} \langle 100 \rangle$  texture preferred orientation. Especially, the high permeability grades with misorientation of less the  $3^\circ$  have huge grains and therewith large magnetic domains. For further improvement of the magnetic properties different techniques can be used, like thinner gauge material, improvement of the orientation and magnetic domain refinement through additional surface treatment or compressive stress of the isolation layer [13 -14].

The effect of the domain refinement is known from surface sensitive MOKE-microscopy and global magnetic measurements, but the spatially resolved, bulk behavior of the magnetic domains and their relation to the losses of the GO electrical steel can only indirectly be derived from surface observations. The nGI technique offers the possibility to observe bulk domains and the bulk magnetic domain structure through the isolation layer without the preparation of the GO steel under different magnetic fields [15].

For the experiment we used a high permeability GO steel sheet with the dimension of 300mm x 30mm x 0.27mm (comparable to M103-37P) with and without isolation layer. The sample without the coating layer was additionally polished for MOKE observation. The sample was mounted in a sample holder and build-in the nGI setup with parallel orientation of the gratings to the magnetic flux, similar to  $0^\circ$  orientation.

The DFI image of the GO steel with and without coating is shown in Fig.2 in zero magnetic field. The GO lamination with coating shows domain walls or domain wall rich areas in black color almost parallel to the direction of rolling. The polishing and removal of the coating changes the domain structure dramatically resulting in a reduction of domain walls, as shown in Fig.2.

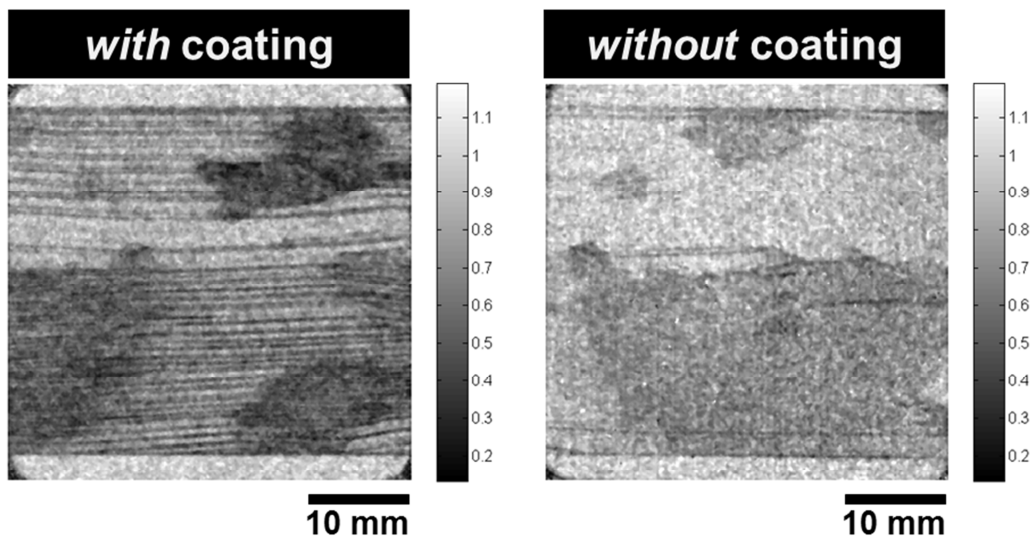


Fig. 2: Neutron dark-field image (DFI) of a 270  $\mu\text{m}$  thick GO lamination with coating. Black color corresponds to domain walls or domain wall rich areas. (left) lamination with coating which is transparent for neutrons and the DFI shows individual domain walls. (right) same lamination polished, without coating and the domain structure has changed dramatically.



### 2.3 Influence of different cutting techniques on NO laminations

Several non-oriented fully processed electrical steel laminations were retrieved parallel (RD) to the rolling direction from the sheet (0.35 mm thick) and cut into Epstein strips with a size of 30x250 mm<sup>2</sup> by laser cutting. Afterwards, the samples were annealed at constant temperature (800°C) for 2 h and cooled down slowly for approximately 18 h. Subsequently, smaller strips with a width of 5 mm and 10 mm were cut off using different techniques: punching, guillotine, solid-state laser and CO<sub>2</sub> laser cutting. Both laser beam sources were integrated into the same type of cutting machine (TruLaser 7025) to guarantee stable process conditions and comparable kinetic states. For each laser setup the chosen process parameters represent the typical industrial application for inert-gas fusion cutting of electrical steels. The punching machine equipped with a blank holder system (TruPunch 3000) provided a maximum stroke rate of 1000 per minute at 1810 kN maximum punching force. The guillotine was used with no blank holder on one side to investigate the effect of asymmetric deformation on resulting magnetic parameters.

The average grain size of  $100 \pm 10 \mu\text{m}$  was analyzed near the cutting edge by scanning electron microscopy (SEM) after certain metallographic preparation. This sample was retrieved from the sheet by laser cutting to prevent any mechanical grain deformation. In order to measure the required magnetic field strength  $H$  (peak value) to achieve a certain magnetic flux density  $B$  within the material ( $B$ - $H$  curve) a Brockhaus magnetic measurement unit (SST) that comprises the magnetizing and secondary windings and a U-shaped flux return path was used. The samples were measured at 50 Hz applying magnetic field control according to the fields of the neutron grating interferometry investigation. That field frequency choice results from the measurement unit configuration in order to gain a good form factor ( $< 1.12$ ) which describes the ratio of the root mean square value to the average rectified value of alternating current signals.

In order to magnetize the sample for the neutron grating interferometer measurement a special holder was used, which consists of primary and secondary winding with two flux return paths that are constructed as double yoke. Additionally, an opening was cut, which is necessary to guarantee nonrestrictive neutron beam passing. Thus, samples with a maximum width of 30 mm can be analyzed. These samples were magnetized applying static magnetic fields with DC demagnetization after every single measurement.

The DFI in Fig. 3 shows the bulk domain density at field value of 1500 A/m for the 4 different machined (mechanical or laser treated) samples. The DFI profile of the sample that was cut with the mechanical shear (guillotine) on top is caused by the unsymmetrical material deformation when the top edge was cut without blank holder. The DFI profile for the punch press causes a symmetric profile with slightly higher DFI values in the middle of the strip and thus a higher magnetic activity or less domain wall density.

The DFI profiles clearly show that both mechanical techniques deteriorate the magnetic properties mostly in the regions of the cutting edge with decreasing extend towards the middle of the sample. The DFI provides information about the deterioration depth of the cutting processes.

In contrast to the mechanical cutting, the DFI profiles for both the laser fusion cutting with the disc and the CO<sub>2</sub> laser shows nearly no deterioration effects at the edges. Although, the intensity peak values regardless of their location are reduced slightly at high magnetic fields in comparison to mechanical processing.

Presented DFI findings promote the scientific interpretation of magnetic deterioration mechanisms interacting with applied magnetic fields, especially for laser cutting. However, the sole DFI contrast distribution cannot be used to display directly magnetic induction values  $B$  within the material, whose understanding is necessary to promote selecting the correct obstructing material grades and designing efficient electrical machines.

Because the local dark-field intensity represents the macroscopic flux density, the integral of the profile correlates with the magnetic flux density in the whole sample. Further work is foreseen to directly relate the local DFI value with the local macroscopic flux density.



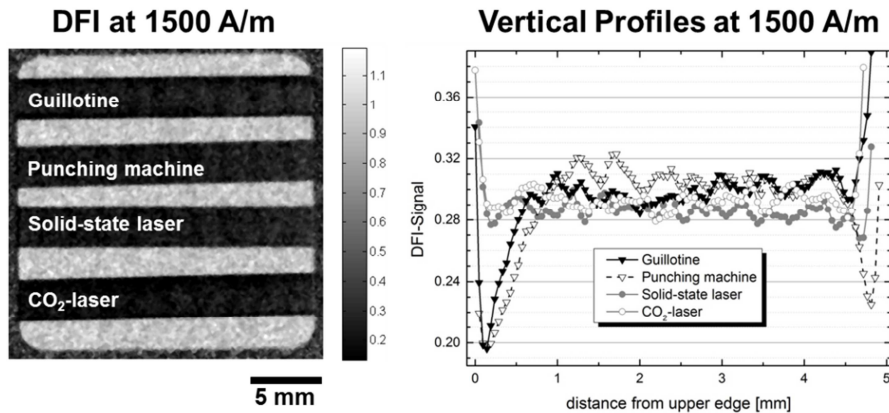


Fig. 3: (left) Dark-field image (DFI) for four Epstein strips with 5 mm width applying four different manufacturing techniques. Dark color represents domain wall rich areas. (right) Vertical profiles through each strip from the top to the bottom edge. Clearly visible is the deterioration effect at the edges for the mechanical cutting techniques.

## Conclusion

For the first time a unique measuring technique is presented that gives access to the domain bulk structure of electrical steels by detecting qualitatively the amount of domain walls in a specific material volume. With the help of the neutron grating interferometer geometry dependent volume magnetization processes can be visualized.

In order to produce customized grain-oriented electrical steel grades with low core losses, high permeability and low noise at operating state this technique promotes a better understanding of manufacturing processes e.g. rolling, coating deposition or domain refinement and their influence on the magnetic domain structure by analyzing domain wall motion at a given magnetic field.

Furthermore, by measuring the domain wall density distribution in non-oriented electrical steel strips, the resulting deterioration of magnetic properties, i.e. magnetization behavior can be analyzed spatially-resolved for a given material grade and cutting technique as a function of the external applied magnetic field. Herewith, the realization of a modeling approach for machine designers to forecast manufacturing related permeability drop is supported.

Further work will concern the visualization of magnetization processes in sample laminations with complex stator, rotor and transformer contours. In addition this measuring technique might be suitable to promote the evaluation of various steel production processes due to the fact that for the first time new material parameters can be determined such as a domain wall density and possibly domain wall velocity.

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