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ACCURATE MEASUREMENT OF COATING THICKNESS ON FULLY-FINISHED NON-ORIENTED ELECTRICAL STEELS USING FIB-SEM

NATANČNO MERJENJE DEBELINE LAKIRANE PLASTI NA GOTOVIH NEORIENTIRANIH ELEKTROPLOČEVINAH Z UPORABO FIB-SEM

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In this study we investigated the potential of using FIB-SEM microscopy to measure the thickness of coated layers on fully-finished, non-oriented electrical steel of NO grades. When using FIB-SEM with gallium ions (Ga^+) to prepare cross-sections of layers with a thickness of a few micrometers (μm), precise ablation of the material with good spatial resolution is essential. Ga^+ generated at an accelerating voltage of 30 kV enable the effective localized removal of material. The sample can be protected with a thin layer of platinum (Pt) deposited by Ga^+ to prevent damage to the top layer during the initial ablation process and to achieve a smoother final cut. The result of this procedure is a clean and sharp cross-section suitable for high-resolution SEM imaging. The presented microscopic method for measuring the thickness of the coatings on fully-finished, non-oriented, electrical steel samples using FIB-SEM has proven to be repeatable, robust, specific, and has an appropriate detection limit for the thickness range of typical coated layers in non-oriented electrical steel sheet.

Keywords: Non-oriented electrical steel, NO grades, coating layers, thickness measurement, FIB-SEM

Raziskali smo možnost uporabe mikroskopske tehnike FIB-SEM za merjenje debeline lakiranih plasti na gotovih neorientiranih elektropločevinah NO kvalitet. Uporabili smo tehniko FIB-SEM in z galijevimi ioni (Ga^+) uspešno pripravili prečne prereze lakiranih plasti debeline nekaj mikrometrov (μm). V tem postopku je bistvena natančna ablacija materiala z dobro prostorsko ločljivostjo. Ioni Ga^+, generirani pri pospeševalni napetosti 30 kV, omogočajo učinkovito lokalizirano odstranjevanje materiala. Vzorec je lahko zaščiten s tanko plastjo platine (Pt), nanešeno z Ga^+, da preprečimo poškodbe zgornje plasti med začetnim postopkom ablacije in da dosežemo bolj gladek končni rez. Rezultat tega postopka je čist in oster prečni prerez, primeren za visokoločljivo SEM slikanje. Predstavljena mikroskopska metoda merjenja debeline prevlek na gotovih vzorcih neorientiranih elektropločevin z uporabo FIB-SEM se je izkazala za ponovljivo, robustno, specifično in ima ustrezno mejo detekcije za območje značilnih debelin lakiranih plasti na gotovih neorientiranih elektropločevinah.

Ključne besede: neorientirane elektropločevine, NO kvalitete, lakirana plast, merjenje debeline, FIB-SEM

1 INTRODUCTION

Fully-processed, non-oriented electrical steel (**Figure 1)** is optimized for its magnetic properties and does not require further processing by the end user. All the properties are fully developed and controlled. The steel can be delivered uncoated or coated with insulation varnish and is ready to be stamped and stacked.[1]

During the characterization of coated layers on fully-finished, non-oriented electrical steels,[2] especially NO grades,[3] a precise determination of their thickness is crucial for understanding the material properties of the product and ensuring quality. NO grades[3] of non-oriented electrical steels with thicknesses under 0.35 mm are specialized for high-performance applications that demand low core losses and high efficiency, especially at medium and higher frequencies. (e.g., those intended for the traction motors of electric vehicles, which can have an RPM several times higher than industrial electric motors). NO grades provide the best-possible foundation for this technology.[1,4] Because of their good permeability (penetrability for magnetic fields) and low core losses (conversion of a portion of the energy into heat due to eddy currents), the NO grades are ideal for high-frequency applications. Normally, a thin insulating coating (varnish) is applied to the surface of the electrical steel to protect the material against environmental factors, and to prevent short circuits caused by low interlayer resistance and to reduce eddy-current losses between laminations.[5,6] Such coatings enhance the stamping performance, corrosion resistance, adhesion strength, and weldability of non-oriented electrical steels. In addition, coatings play a crucial role in reducing the power losses. By manipulating the surface roughness or varying the surface stress through coatings, overall power loss can be decreased. A typical surface of a NO25, fully-finished, non-oriented electrical

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Figure 1: Coils of the fully-finished, non-oriented electrical steel (SIWATT brand of SIJ Acroni d.o.o., Jesenice, Slovenia)[1]

steel sheet is represented in **Figure 2**. Studies have shown that improving the surface roughness can reduce power losses.[6] Insulating coatings can be organic, semi-organic, or inorganic. Several techniques are available for coating steel sheets, each with its own unique advantages. These methods include sol–gel processes, chemical vapor deposition (CVD), physical vapor deposition (PVD), plasma spraying, wet coating, printing, electroless plating, and electrochemical plating.[6] Different coatings have different specific properties, and the selection of coatings depends on subsequent application requirements, to provide continuous protection, good corrosion resistance, and the desired performance.[5] In recent years, the development of insulating coatings for electrical steels has increasingly focused on environmentally friendly solutions.[5,7]

Two conventional methods are commonly used for measuring coating thickness: the beta-backscatter method, which allows a non-destructive measurement of organic and inorganic layers on various substrates using radiation from radioactive atoms in either a contacting or non-contacting setup,[8] and the ASTM E376-19 standard practice, which specifies magnetic-field or eddy-current (electromagnetic) testing methods.[9]

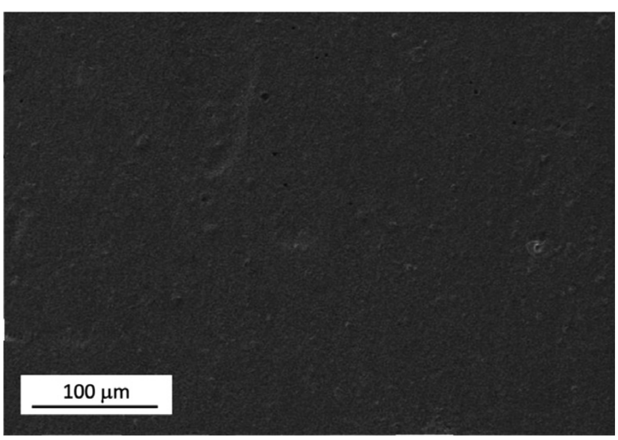

Figure 2: A surface of a NO25, fully-finished, non-oriented electrical steel sheet (SE Image)

Recently, the weigh–strip–weigh method has been demonstrated as a reliable approach to accurately determining the coating mass and thickness, addressing quality concerns that arise in post-production situations.[10] Additionally, a laser-induced fluorescence-based system provides highly accurate, real-time thickness measurements that surpass traditional methods and enhance process control and product quality.[11]

This study aims to complement the metallographic approach; we also employ focused-ion-beam (FIB) cross-sectioning to obtain direct measurements of the coating thickness. These high-resolution sections provide a reliable reference for evaluating the accuracy of the proposed method. Together, the two techniques allow a more precise assessment of thin insulating coatings on electrical steel sheets.

2 EXPERIMENTAL

The samples used in the study were 0.25- mm and 0.27-mm-thick, fully-finished, non-oriented electrical steel sheets made from Fe – 3.2 *w*/%Si – 1.0 *w*/%Al – 0.2 *w*/%Mn alloys (NO grades). The designation **NOxx-yy** generally indicates a thickness of **0.xx** mm, and a maximum core loss of **yy** in W/kg at a 1.0 T/400 Hz.[3]

For the preparation of cross-sections, focused-ion-beam scanning electron microscopy (FIB-SEM) using gallium (Ga) ions was employed.

Precise material ablation with high spatial resolution was required to obtain reliable cross-sectional profiles. Gallium ions generated at an accelerating voltage of 30 kV were applied to enable effective localized removal of material. Higher beam currents, ranging from 15 nA to 3 nA, were used for coarse milling of larger volumes during the initial milling stage. In contrast, lower beam currents (e.g., 700 pA) were used for the final polishing step, reducing material redeposition and producing smoother cross-sectional surfaces.

After milling, the cross-sections were examined using a field-emission SEM operated at 5 kV. Secondary electron (SE) and backscattered electron (BSE) detectors were used depending on the required contrast, with working distances of 5–7 mm typically used to maintain high image resolution. The imaging conditions enabled detailed analysis of both the surface coating and the underlying microstructural features.

As a reference, the FIB-SEM method was calibrated and validated using the reference measurement system of a ZEISS Crossbeam 550/EDAX platform for FIB and FE-SEM/EDS analysis.

3 RESULTS AND DISCUSSION

The metallographic characteristics in terms of the texture and microstructure of the typical samples of the two selected NO grades[3] are shown in **Figure 3**.

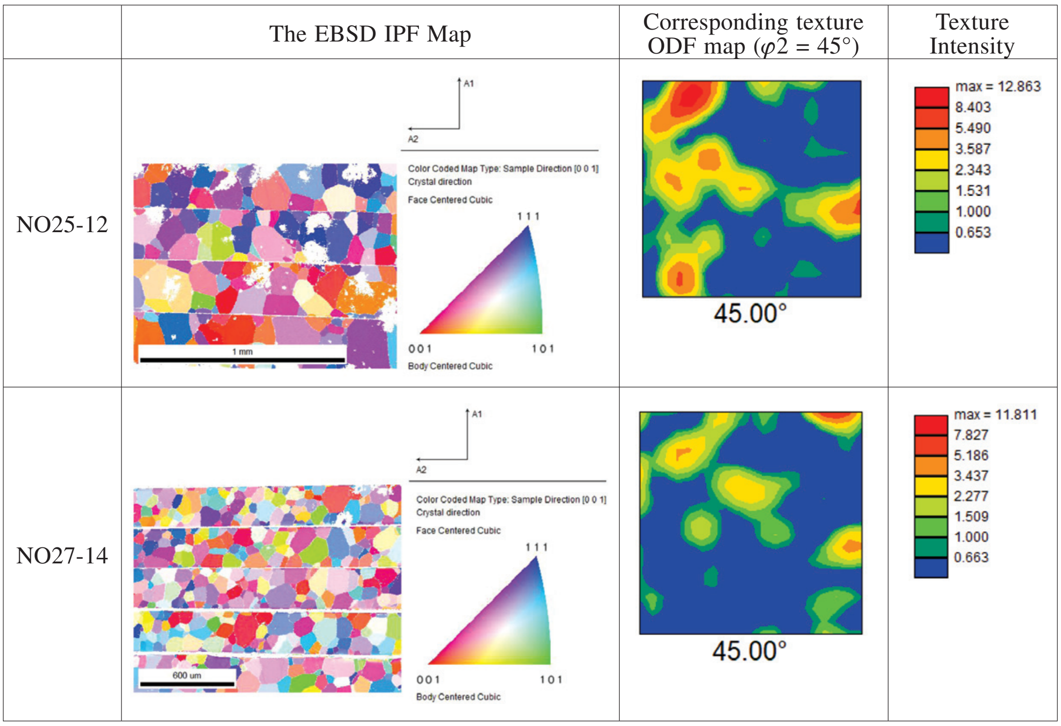

Figure 3: Microstructure and texture characteristics of the typical samples of the two selected NO grades[3] under investigation (SEM-EBSD)

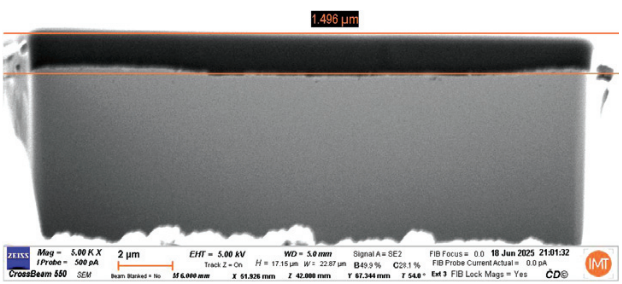

Figure 4: Coating thickness of a typical **NO25-12** sample (Cut 1; FIB-SEM)

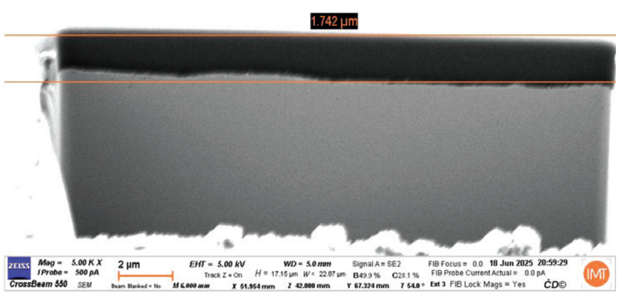

Figure 5: Coating thickness of a typical **NO25-12** sample (Cut 2; FIB-SEM)

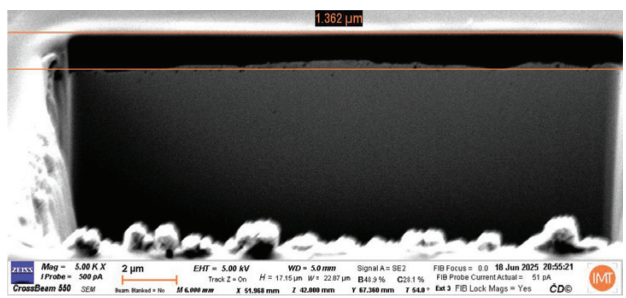

Figure 6: Coating thickness of a typical **NO25-12** sample (Cut 3; FIB-SEM)

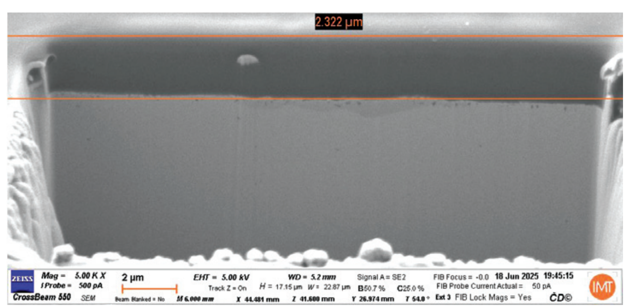

Figure 7: Coating thickness of a typical **NO27-14** sample (Cut 1; FIB-SEM)

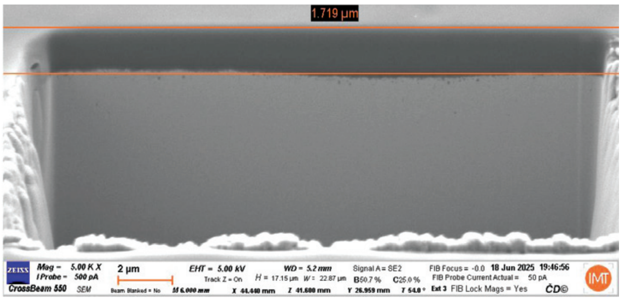

Figure 8: Coating thickness of a typical **NO27-14** sample (Cut 2; FIB-SEM)

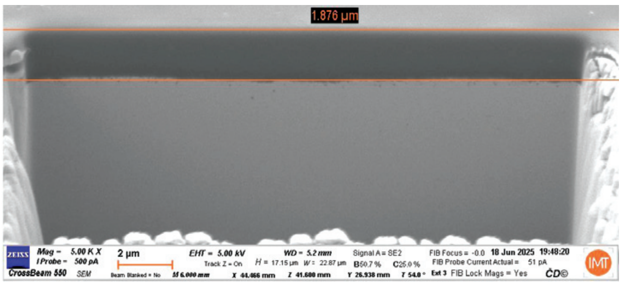

Figure 9: Coating thickness of a typical **NO27-14** sample (Cut 3; FIB-SEM)

Figures 4 to 9 show SEM images of FIB-prepared cross-sections used to determine the coating thickness of the two fully finished, non-oriented electrical steel grades. The thicknesses of the measured coatings were 1.5±0.2 µm in the case of the selected **NO25-12** grade, and 1.9±0.3 µm in the case of the selected **NO27-14** grade.

Beneath the measured coatings, some (internal) oxidation layers are observed, typical of Fe-Si-Al alloys. The presence of silicon and aluminium (elements with a high affinity for oxygen) leads to the formation of a thin oxide network within the material. Such oxidation can influence both the mechanical and magnetic properties of the steel. In general, surface oxides in non-oriented electrical steels increase the core losses. The oxidation behaviour is strongly affected by the alloying elements, surface conditions during processing, and the presence of segregants, all of which can influence the oxidation rate, the mechanisms involved, and the morphology of the oxide scale.[12]

In NO grades, measuring the coating thickness is particularly challenging because the electrical steel sheets themselves are very thin, typically 0.2–0.3 mm. One of the major difficulties arises when the sample is not perfectly aligned during the measurement, i.e., when it is slightly tilted relative to the reference plane of the measurement system. Even a slight inclination increases the effective measurement path, leading to an apparent coating thickness greater than the actual value. This effect is especially pronounced in very thin coatings, where such errors can become comparable to the actual layer thickness. If the tilt is not detected or adequately corrected, it may lead to the misinterpretation of data, inaccurate results, and, ultimately, incorrect conclusions in research or industrial processes. This need becomes even more pronounced when considering the stringent performance requirements placed on electrical steels used in advanced industrial applications.

In the highly competitive automotive industry, compliance with relevant standards, along with continuous improvement and employee training, is essential for delivering high-quality products.[13] Consequently, novel material characterization methods are crucial for ensuring the quality and reliability of components, including for the characterization of NO grades of the non-oriented electrical steel sheets commonly used in automotive applications.[14–22]

In our study, the measurements were performed using the calibrated reference measurement system of the ZEISS Crossbeam 550 FIB SEM, equipped with EDS and EBSD capabilities. By employing high-precision sample mounting and plane-parallel cutting, we successfully overcame the methodological challenges described above. The presented microscopic approach for determining the coating thickness has proven to be repeatable, robust, and highly specific, with a suitable detection limit for the thickness range characteristic of typical coated layers.

The FIB-SEM method, when applied to fully finished, non-oriented electrical steels of NO grades, has proven highly effective for measuring coating thickness. Based on our experience, this metallographic approach, performed under vacuum conditions, is also well-suited for the analysis of multilayer materials, nanostructures, semiconductor components, and thin films thicker than 100 nm. It is particularly advantageous when high spatial precision and minimal material damage are required, or when the sample is sensitive to the surrounding atmosphere (e.g., oxygen).

4 CONCLUSIONS

The FIB-SEM cross-section metallographic method for a coating-thickness measurement used in this study on fully-finished, non-oriented electrical steels of NO grades proved to be highly effective and well-suited for this type of analysis. The procedure produces a clean, sharp cross-section suitable for high-resolution SEM imaging. The FIB-SEM method was calibrated and validated using the reference measurement system of the ZEISS FIB-SEM Crossbeam 550 electron microscope, equipped with EDS and EBSD. The presented microscopic method for measuring coating thickness has proven to be repeatable, robust, specific, and has an appropriate detection limit for the thickness range of typical coated layers in fully-finished, non-oriented electrical steel sheet of various NO grades.

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5 REFERENCES

[1] SIWATT, Non-oriented electrical steel, https://www.acroni.si/en/brands/siwatt (accessed October 25, 2025)

[2] SIST EN 10106:2016, Cold rolled non-oriented electrical steel sheet and strip delivered in the fully processed state, Slovenian Institute for Standardization, Ljubljana, Slovenia, 2016

[3] SIST EN 10303:2016, Thin magnetic steel sheet and strip for use at medium frequencies, Slovenian Institute for Standardization, Ljubljana, Slovenia, 2016

[4] NO grades: Non-grain-oriented electrical steel strip compliant with DIN EN 10303, https://www.waelzholz.com/en/steel-materials/electrical-steel-strip/no-grades (accessed October 25, 2025)

[5] X. Hu, X. Zhang, C. Pi et al., Study on formation mechanism and corrosion resistance of an environmentally protective insulating coating on non-oriented electrical steel, Materials Chemistry and Physics, 304 (**2023**) 127819, doi:10.1016/j.matchemphys.2023.127819

[6] E. Elgamli, F. Anayi, Advancements in Electrical Steels: A Comprehensive Review of Microstructure, Loss Analysis, Magnetic Properties, Alloying Elements, and the Influence of Coatings, Applied Sciences, 13 (**2023**) 10283, doi:10.3390/app131810283

[7] C. Pi, X. Zhang, J. Liu et al., Study on the passivation effect and corrosion mechanism of an environmentally protective insulating coating on non-oriented electrical steel, Materials Today Communications, 38 (**2024**) 107978, doi:10.1016/j.mtcomm.2023.107978

[8] ASTM E376-19, Standard Practice for Measuring Coating Thickness by Magnetic-Field or Eddy Current (Electromagnetic) Testing Methods, ASTM International, West Conshohocken, PA, USA, 2019

[9] Beta-backscatter method, https://www.helmut-fischer.com/applications/solutions/beta-backscatter-method?utm_source=chatgpt.com (accessed October 25, 2025)

[10] C. Grimmett, R. A. Fleming, Accurate Measurements of Electrical Steel Coating Thickness Using the Weigh-Strip-Weigh Method, ASEE Midwest Section Conference, Fayetteville, AR, USA, (**2025**) 1–5, doi:10.18260/1-2-1139.1153-57840

[11] J. Bublitz, M. Kreso, A. Jordan et al., Advanced Coating Thickness Measurement in Electrical Steel, 7th European Steel Technology and Application Days - ESTAD 2025, Verona, Italy (**2025**) Contribution No. 197

[12] D. Steiner Petrovič, Influence of Selenium on the Internal Oxidation of Fe–Si–Al–C Alloy, ISIJ International, 51 (**2011**) 9, 1539–1544, doi:10.2355/isijinternational.51.1539

[13] Z. Tanasić, A. Jokić, G. Janjić et al., Business process improvement in the automotive industry - quality methods and tools, Acta technica corviniensis, 14 (**2021**) 4, 49–54

[14] L. M. Duarte, J. D. de Alencar Santos, F. N. Costa Freitas et al., A novel approach based on pattern recognition techniques to evaluate magnetic properties of a non-grain oriented electrical steel in the secondary recrystallization process, Measurement, 167 (**2021**) 108135, doi:10.1016/j.measurement.2020

[15] S. Tumanski, Modern methods of electrical steel testing – a review, Przeglad Elektrotechniczny, 3 (**2021**), 162–167, doi:10.15199/48.2021.03.31

[16] M. Heller, A. Stöcker, R. Kawalla et al., Characterization Methods along the Process Chain of Electrical Steel Sheet – From Best Practices to Advanced Characterization. Materials,15 (**2022**) 32, doi:10.3390/ma15010032

[17] N. Leuning, M. Heller, M. Jaeger, et al., A new approach to measure fundamental microstructural influences on the magnetic properties of electrical steel using a miniaturized single sheet tester, Journal of Magnetism and Magnetic Materials, 583 (**2023**) 171000, doi:10.1016/j.jmmm.2023.171000

[18] C. Song, J. Cao, J. Xiao et al., High-temperature constitutive relationship involving phase transformation for non-oriented electrical steel based on PSO-DNN approach, Materials Today Communications, 34 (**2023**) 105210, doi:10.1016/j.mtcomm.2022.105210

[19] P. Rękas, M. Nowicki, P. Gazda et al., A measuring setup for testing the mechanical stress dependence of magnetic properties of electrical steels, Journal of Magnetism and Magnetic Materials, 577 (**2023**) 170791, doi:10.1016/j.jmmm.2023.170791

[20] G. Paltanea, V. Manescu, A. Antoniac et al., Mechanical and magnetic properties variation in non-oriented electrical steels with different cutting technology: A review. Materials, 17 (**2024**) 1345, doi:10.3390/ma17061345

[21] H. Mamiya, N. Terada, K. Hiroi et al., Role of neutron Bragg-edge spectroscopy in development of practical magnetic materials, Journal of Applied Physics, 138 (**2025**) 143904, doi:10.1063/5.0285904

[22] M. Lo Giudice, A. Salvini, M. Stella et al., Efficient hysteresis characterization and prediction in 3D-printed magnetic materials using deep learning, IET Science, Measurement & Technology, 19 (**2025**) e12233, doi:10.1049/smt2.12233