Electromagnetic Non-Destructive Evaluation (XXI)
D. Lesselier and C. Reboud (Eds.)

doi:10.3233/978-1-61499-836-5-120

Product Uniformity Control – A Research Collaboration of European Steel Industries to Non-Destructive Evaluation of Microstructure and Mechanical Properties

F.D. VAN DEN BERG[a,1], P.J.J. KOK[a], H. YANG[a], M.P. AARNTS[a], P. MEILLAND[b], T. KEBE[c], M. STOLZENBERG[d], D. KRIX[d], W. ZHU[e], A.J. PEYTON[e], A. MARTINEZ-DE-GUERENU[f], I. GUTIERREZ[f], D. JORGE-BADIOLA[f], M. MALMSTRÖM[g], A. VOLKER[h], A. DUIJSTER[h], H. WIRDELIUS[i], A. BOSTRÖM[i], C. MOCCI[j], M. VANNUCCI[j], V. COLLA[j], C. DAVIS[k], L. ZHOU[k], R. SCHMIDT[l], S. LABBÉ[m], C. REBOUD[n], A. SKARLATOS[n], V. LECONTE[o], and P. LOMBARD[o]

[a] *Tata Steel, IJmuiden, The Netherlands*
[b] *ArcelorMittal Global Research, Maizières-lès-Metz, France*
[c] *thyssenkrupp Steel Europe, Duisburg, Germany*
[d] *Salzgitter Mannesmann Forschung GmbH, Salzgitter, Germany*
[e] *University of Manchester, Manchester, United Kingdom*
[f] *CEIT, San Sebastián, Spain*
[g] *Swerea-KIMAB, Kista, Sweden*
[h] *TNO, Den Haag, The Netherlands*
[i] *Chalmers University of Technology, Göteborg, Sweden*
[j] *Scuola Superiore Sant'Anna, Pisa, Italy*
[k] *University of Warwick, Coventry, United Kingdom*
[l] *ArcelorMittal Eisenhüttenstadt, Eisenhüttenstadt, Germany*
[m] *Université Grenoble Alpes, Grenoble, France*
[n] *CEA Commissariat à l'Energie Atomique, Gif-sur-Yvette, France*
[o] *ALTAIR Engineering France, Meylan, France*

Abstract. In steel manufacturing, the conventional method to determine the mechanical properties and microstructure is by offline, destructive (lab-)characterisation of sample material that is typically taken from the head or the tail of the coil. Since coils can be up to 7 km long, the samples are not always representative for the main coil body. Also, the time delay (typically a few days) between the actual production and the availability of the characterisation results implies that these results cannot be exploited for real-time adaptation of the process settings.

Information about the microstructure and material properties can also be obtained from electromagnetic (EM) and ultrasonic (US) parameters, which can be measured in real-time, non-destructively, and over the full length of the steel strip

[1] Corresponding Author, Tata Steel, Research & Development, PO BOX 10.000, 1970 CA IJmuiden, The Netherlands; E-mail: frenk.van-den-berg@tatasteel.com

product. With the aim to improve the consistency in product quality by use of inline EM and US measurements, a European project called "Product Uniformity Control" (PUC) has been set up as a broad collaboration between 4 major European Steel Manufacturers and 10 Universities / Research institutes.

Using both numerical simulations and experimental characterisations, we study the inline measured EM and US parameters in regard of the microstructural and mechanical properties. In this way, we aim to establish an improved understanding of their mutual relationships, and to apply this knowledge in existing and new non-destructive evaluation techniques.

In this paper, the concerted approach of modelling and experimental validation will be addressed, and results of this work will be shown in combination with inline measured data.

Keywords. steel, material characterisation, mechanical properties, non-destructive evaluation, inline monitoring

1. Introduction

The majority of steel used for automotive applications is produced as ‘infinitely’ long flat (strip) steel, with typical width of 1 – 2 m, and thickness ranging from 0.6 – 1.6 mm. For the ease of transport to the customer, the steel industry ships the strip steel as coils to the customers, see figure 1. Each coil typically measures several km of strip length and weights 10 – 20 tons. Next to the timely delivery of the product according to the required dimensional tolerances, the steel manufacturer strives for high quality standards, in particular regarding the surface condition, as well as the forming properties and the mechanical strength. This paper focuses on the latter quality aspects, i.e. the mechanical properties.

Figure 1. Steel coils ready for dispatch to customers.

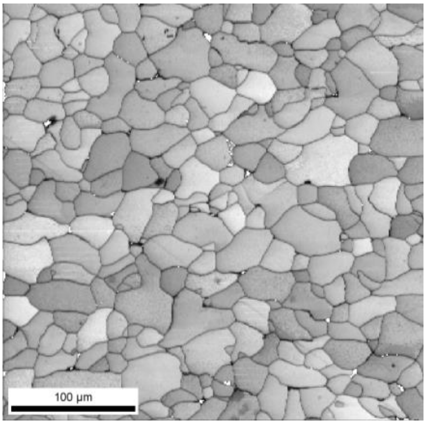

Figure 2. Example of granular microstructure of steel, here of a traditional low-carbon steel.

The mechanical properties are governed by the microstructure of the material. Traditionally, the microstructure and the mechanical properties of steel strip are determined from samples taken from the head or tail parts of a coil. From these samples, specimens are prepared for (automated) tensile testing and for microstructure analysis. Typical engineering parameters deduced from the stress-strain curve obtained by tensile testing are the yield strength (Rp) and tensile strength (Rm). Microstructural parameters that have a relation to the mechanical properties are grain size (distribution), crystal orientation (texture) (distribution) and phase (or phase constituent) volume fractions. These can be imaged by an electron backscatter diffraction (EBSD) measurement, as illustrated in figure 2, which shows the grain pattern of a low carbon steel.

In recent years, the strong drive to reduce the weight of cars has stimulated the development and manufacturing of new high-strength steel grades. These grades owe their higher strength from the finer dispersion of their microstructure. In particular, the microstructure of these 'advanced high-strength steels' (AHSS) is featured by reduction of the grain size and the introduction of (hard) secondary phases, small precipitates, and highly irregular grain boundaries, and combinations thereof, as illustrated in figure 3.

In comparison to the regular grades, the microstructure and hence the mechanical properties of these AHSS grades are more sensitive to the exact thermo-mechanical processing, i.e. the heating and cooling trajectories.

The partly batch-wise processing of the steel, in the form of slabs / coils, intrinsically results in the impossibility to have a fully identical thermal treatment for each part of the strip. For instance, the heating of slabs in furnaces is non-uniform, speed variations occur in the process (acceleration and deceleration of the strip), and the cooling down of the hot-rolled coil is non-uniform. As shown in figure 4, these phenomena may lead to a significant variation of the microstructure and hence in the mechanical properties. This typically occurs in the extremities (head and/or tail) of a strip. Figure 4 shows the variation of a magnetic measurement (here: IMPOC) over the length of an AHSS strip (Dual Phase grade (DP)). Clearly, the first 50 m of the strip deviates from the bulk part.

It is notably this type of inline measurement technology that can help to get insight into the occurrences of microstructure variations, i.e. to detect their occurrence, evaluate their severity and study the frequency of occurrence in relation to (given combinations of) processing parameters. Further along this road, microstructure monitoring can provide checks to keep up with the required tighter process window for AHSS and to ensure that the product meets the mechanical property specification over its entire length.

To investigate the topic of the uniformity of steel strip in more detail, and to develop instrumentation for the fast, inline characterisation thereof, a large European consortium, comprising 4 major European steel manufacturers, has been established in the project "Product Uniformity Control', started in 2013.

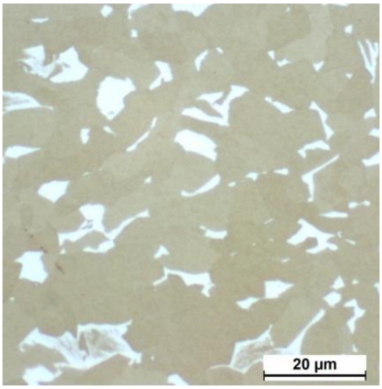

Figure 3. Example of AHSS microstructure, here with about 15% martensite (white area in picture), 12% bainite, and 73% ferrite.

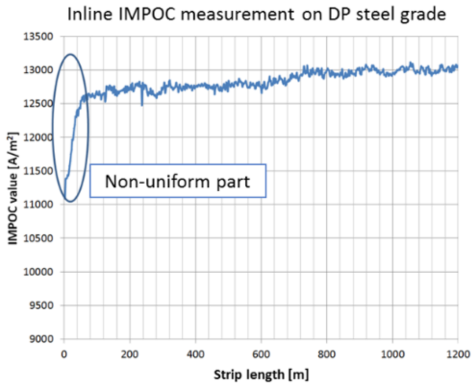

Figure 4. Example of inline magnetic measurement (IMPOC) on cold-rolled, galvanized AHSS steel (Dual Phase steel).

2. Background

2.1. Microstructure of steel

The microstructure of steel is formed during the manufacturing process of subsequently casting, hot-rolling, cold-rolling, (continuous or batch) annealing and finishing (temper rolling, coating and skin-pass rolling). Low-alloyed, low carbon steel has a ferritic grain like structure with average grain size in the 10 – 50 micron range. Higher alloying contents, often in combination with faster cooling trajectories, result in the creation of (additional, i.e. 'secondary') phases or phase constituents like pearlite, bainite and martensite, which are generally harder than ferrite.

In the study of the microstructure, different microstructure parameters are distinguished [2], i.e.:

- Grain size (distribution)
- Grain morphology, grain elongation (aspect ratio)
- Texture (crystal orientation)
- Secondary phase fractions (and connectivity of phases)
- Precipitate density and precipitate size
- Dislocations

Precipitates are small particles (1 nm – 1 um) of carbides and nitrides of (typically) Nb, Ti or V and dislocations are (tiny) lattice distortions due to straining of the material. All the microstructure parameters listed above play a role in the determination of the mechanical properties of steel, and hence they are of importance for the aim of materials characterisation. However, during the manufacturing of steel, these microstructural parameters will not vary one by one, but their variation of almost always coupled. This makes it difficult to attribute a change in a (macroscopic) property to its individual microstructural contributions in a quantitative way.

2.2. Non-destructive evaluation techniques used in steel manufacturing

Past research work [1-17] has investigated and developed electromagnetic (EM) and ultrasonic (US) techniques that are sensitive to individual features of the steel microstructure. Advantages of the EM and US techniques are their non-destructive nature and the potential to apply them in non-contact mode, and in harsh environments. These capabilities give them preference over X-ray diffraction (XRD) based methods: although providing more direct information on the microstructure in a lab environment, XRD measurements are (still) regarded to be too vulnerable and too expensive for use in inline monitoring applications in the steel processing industry, where water, dust and the fast movements of the product would spoil the signals. In the present scope of electromagnetic non-destructive evaluation (ENDE), this contribution will address only the EM related work that is carried out in the PUC project.

In the application of EM techniques for the NDE of the microstructure, an imminent problem is the fact that, generally spoken, magnetic properties are sensitive to all the microstructure parameters of steel listed in section 2.1, and hence are not selective. Accordingly, this poses a huge challenge in the interpretation of a variation in a measured

---

[2] The chemical composition of the steel is supposed to be known from the chemical analysis in the steelmaking plant.

EM signal in terms of a variation in microstructure. The next section highlights the approach which is followed by the consortium to investigate this problem in detail.

3. Approach

From the previous section, it is clear that the inverse problem in EM has too many solutions to give direct answers without further knowledge of the possible microstructure configurations that could occur within the scope of the process. Therefore, an approach has been defined consisting of both an experimental characterisation chain and a modelling chain, which has been depicted schematically in figure 5. In the experimental characterisation chain, samples from different stages in the steel production chain have been taken to investigate their (uniformity in) microstructure, EM properties and NDE instrument output parameters. In addition, a selection of samples are used for dedicated laboratory experiments to investigate influences from operating conditions, like the influence of lift-off, (transversal) speed and zinc coating thickness (since most steel suppliers galvanise the steel strip for automotive applications). Moreover, small reference samples have been prepared specifically to explore systematically the effect of variations in the microstructure parameter space on the fundamental (ultrasonic and) magnetic properties. The experimental data from these experimental studies partly serves as input, and partly as validation data for the simulation chain. In the simulation chain, models are built to investigate the direct problem: i.e. given a certain well-defined microstructure, what are its EM properties? And given these EM properties, what are the responses of the EM measurement equipment (in given operating conditions)?

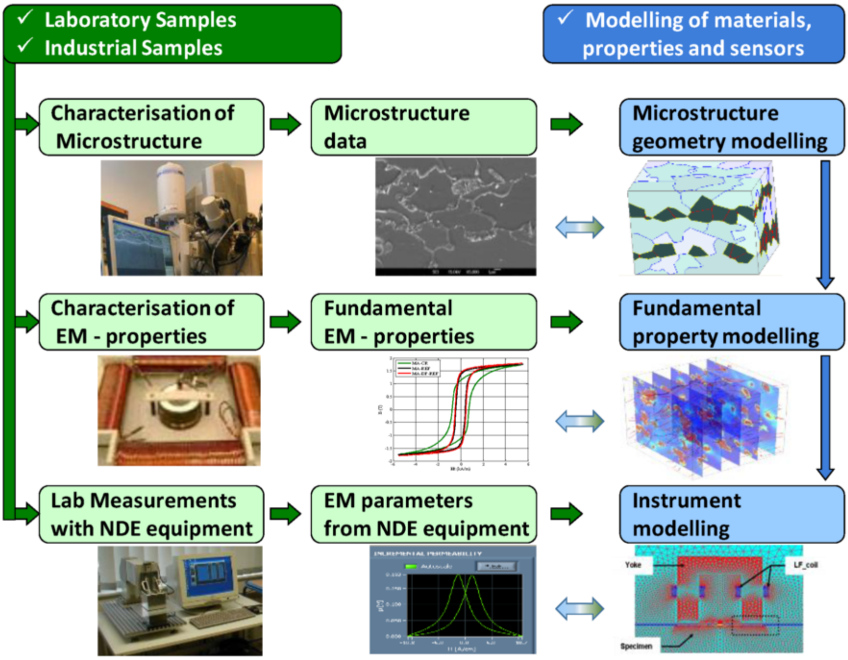

Figure 5. Experimental and modelling chains in the PUC project.

With the use of computer generated microstructures, it is straightforward to vary just one microstructure parameter at a time to study its individual effect on the magnetic properties (and on the NDE measurement), and rank the effects according to importance in a given position in the microstructure parameter space. Another possibility is to generate a multitude of different microstructures, even beyond physically realistic structures, to explore the full microstructure parameter space.

4. Description of models

Modelling of fundamental magnetic properties of polycrystalline microstructures like steel has to be considered as a 3D problem. Model reduction to 2D model descriptions is deemed to fail because even in the simpler case where isotropic behaviour is assumed, the reduction of the number of degrees of freedom limits the possible magnetic flux paths, resulting in solutions that falsely predict a harder magnetic behaviour. As a consequence, 2D microstructure descriptions that are readily obtained from micrographs or EBSD measurements cannot be used as direct input to magnetic property modelling, which underlines the need to rely on 3D microstructure geometry models for this purpose.

4.1. Microstructure geometry modelling

The 3D microstructure geometries are generated using multi-level Voronoi modelling [18-20], see figure 6 for illustrations. The microstructure generation tool allows to control grain size, grain morphology, texture, phase ratios and their distributions. The resulting geometries are described in text files with a format similar to EBSD output, providing a simple and efficient way to interface to fundamental property models.

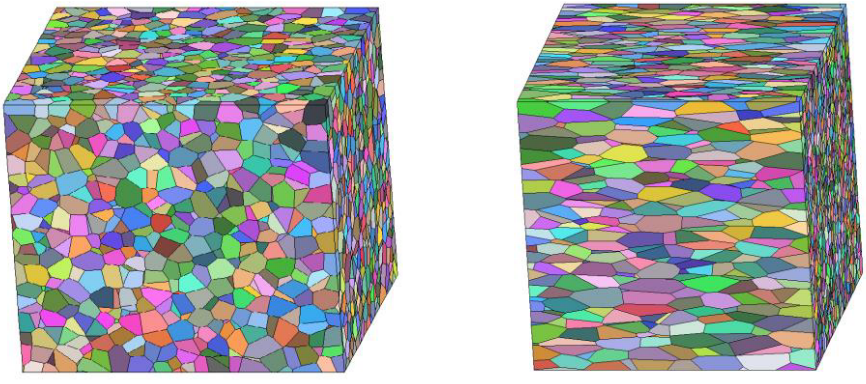

Figure 6. Example of computer generated 3D microstructures. Left: with equal grain aspect ratio in all 3 dimensions; Right: with 1:1:3 aspect ratio in grain size, allowing to study the effect of grain morphology.

4.2. Fundamental EM property modelling

For the modelling of the fundamental magnetic behaviour, two courses are taken: a micromagnetic approach to solve the magnetic problem on the (sub-)micron scale, and a finite element approach to deduce effective magnetic permeability on a mesoscale.

4.2.1. Micromagnetic modelling

Micromagnetism, introduced by William Fuller Brown Jr in 1963 [21], treats the physics of magnetism at sub-micrometer scale. The small scale is required to resolve domain walls and their interaction with matter. Due to the fine spatial resolution that is required, the numerical computation of micromagnetic problems demands high computational power and the solutions are often limited to small volumes.

The solving technique applied here uses a finite difference like discretization scheme of the Landau and Lifshitz equations to find the energy minimum of the sum of these contributions at each step of a magnetisation cycle, resulting in the magnetic hysteresis loop [22,23]. It appears that a stable solution can be achieved when the configuration is set to saturation magnetization in the initial state, and relaxed to zero applied field.

4.2.2. Modelling of the effective relative magnetic permeability

For the particular purpose of phase ratio evaluation in dual phase and multiphase steels, a fast and effective solution to model the effective magnetic permeability is found by 3D finite element modelling in COMSOL of the granular structure, in which the grains have been assigned to different phases with different magnetic permeabilities. The same approach has been used to account for the grain size effect on the magnetic permeability by assigning a different (higher) permeability to a narrow zone around the grain boundary compared to the bulk.

4.3. EM-based NDE instrument modelling

EM NDE instruments that are used for inline measurement in steel manufacturing and that are studied within the PUC project, are IMPOC, HACOM, EMSPEC (MFIA) and 3MA. These instruments have been described in [24,25].

The modelling of these instruments is performed by different institutes using different software packages. For IMPOC [26], a combination of Finite Integration Techniques and a mesh-less modal approach is used to calculate the field gradient after a pulse excitation. HACOM [27] and EMSPEC [28] have been modelled in ANSYS-MAXWELL [29] and the 3MA [30] in ALTAIR-FLUX [31].

5. Results

Low carbon steel samples of identical chemistry have been heat treated to produce a range in grain size. These samples have been characterised on microstructure (grain size) and on magnetic properties, see figure 7. Their microstructures have been simulated in 3D geometry models, serving as input for the fundamental modelling of the magnetic behaviour. Figure 7 compares the initial magnetic permeability ($\mu_{r,i}$) and the coercive field Hc as function of the grain size. Details on samples, measurements and models are given in [32]. A good agreement is found between model prediction and experimental results, in particular for the $\mu_{r,i}$-model while the micromagnetic model may suffer from a too coarse mesh for a proper description of Hc in the lower grain size region.

A demonstration of the capabilities of the instrument modelling is given in figure 8, showing the modelled and measured incremental permeability (IP) as sensed by the 3MA instrument for different lift off settings. As can be observed, the IP curve shape is well reproduced. The effect of lift off on the signal drop is qualitatively well reproduced, albeit from a quantitative perspective further improvements could be made.

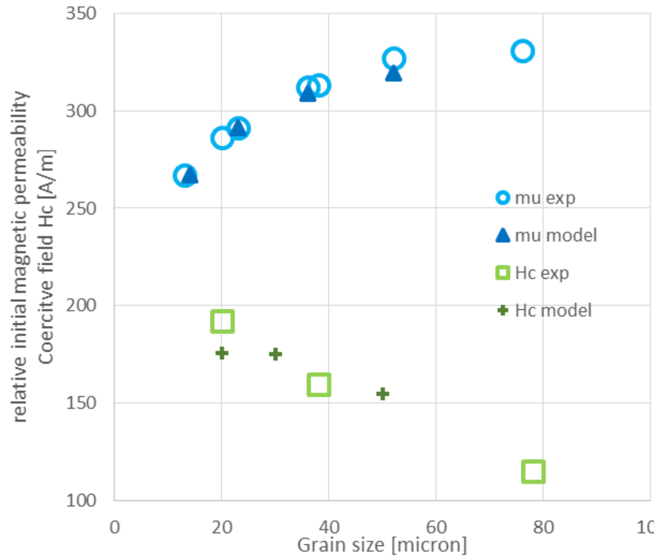

Figure 7. Model and experimental results for the initial magnetic permeability (mu) and coercive field Hc as a function of the grain size, for a fully recrystallised low carbon steel.

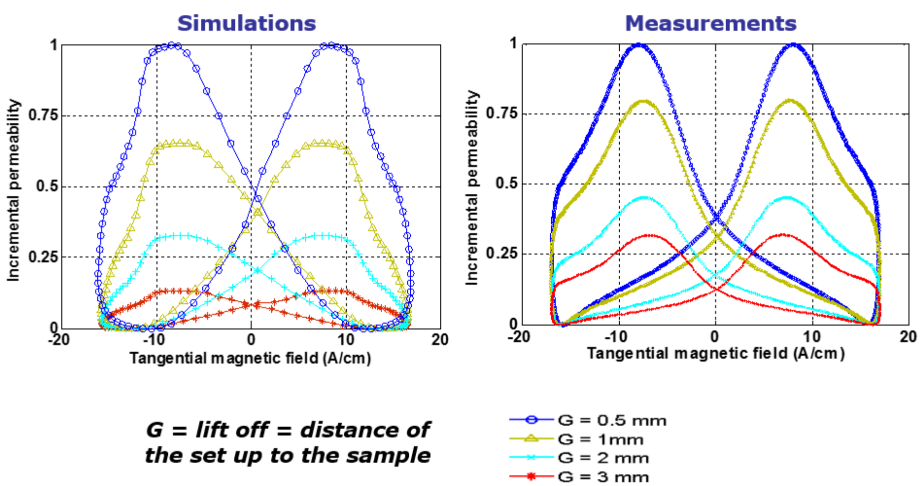

Figure 8. Simulated and measured incremental permeability of the 3MA, for different lift off settings.

An illustration how the inline magnetic data can be converted to microstructure information is given in Figure 9. Here, the data from the inline IMPOC system, situated in the pickling line of one of the steel manufacturers in the consortium, has been analysed on its deviation from the baseline of a given coil of an interstitial-free steel grade. The

deviations stem from the positioning of the steel slabs on support bars in the reheating pusher-furnace prior to hot rolling. At the support bar positions, the slab temperature remains lower than in the unsupported length sections, resulting in slight differences in grain size for this grade.
From measurements on samples, phenomenological relations have been deduced between IMPOC values and coercive field Hc, and between Hc and grain size (see Figure 7), which form the basis for the calculation of the grain size evolution over strip length as depicted in Figure 9. In the calculation, a baseline grain size of 28 microns has been assumed based on microstructure characterisations on multiple samples from this grade.

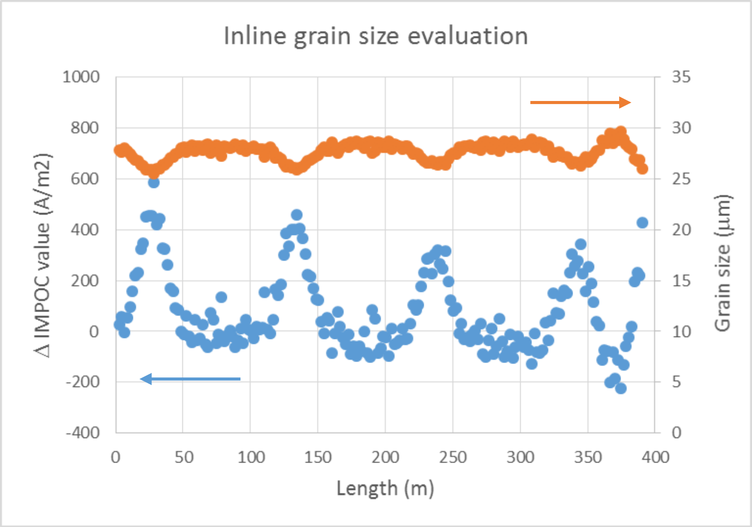

Figure 9. Microstructure evaluation over length using the electromagnetic IMPOC instrument. The blue markers (bottom, left axis) represent the deviation of the IMPOC data from the baseline. The orange markers (top, right axis) shows the grain size deduced from the measurement.

6. Conclusions & Outlook

Within the European collaboration project PUC, we have been able to develop direct models to predict magnetic behaviour and sensor response. Microstructure variations at grain size scale could be measured and modelled and appear to have significant effect on the magnetic properties, with good correspondence between models and experimental observations. We have shown that the 3D microstructure geometry modelling is a powerful tool to generate a multitude of well-defined microstructure variations, A next step is to broaden the microstructure model space to study the inversion problem, e.g. by the use of reduced models or surrogate modelling, in order to interpret the sensor measurements back to microstructure variances. From the relations between instrument data, fundamental electromagnetic properties and microstructure, we managed for certain steel grades to deduce the grain size over length from inline magnetic measurements.

Albeit significant progress have been made, there are still phenomena that need to be studied in a quantitative way, like the effects on the magnetic behaviour caused by microstructure details at the submicron scale, i.e. precipitates and dislocations.

Furthermore, texture, stress and depth gradients are potentially present in the material due to the rolling processes to which the steel strip is subjected. The incorporation of these effects is therefore also important for a good understanding of the measurement data. The large number of microstructure parameters that have influence on the magnetic properties underline that a deep knowledge of the production process and its resulting metallurgical variations is a prerequisite in the interpretation which microstructure variation(s) may cause the measured (magnetic) variations.

7. Acknowledgements

The research leading to these results has received funding from the European Union's Research Fund for Coal and Steel (RFCS) research programme under grant agreement nr. RFSR-CT-2013-00031.

References

[1] R.M. Bozorth: Ferromagnetism, 1951, Van Nostrand, New York; Reissued IEEE Press, 1993.
[2] S. Chikazumi: Physics of Magnetism, Wiley, New York, p 182, 1964.
[3] B.D. Cullity: Introduction to magnetic materials, Addison-Wesley, MA,USA, pp236-237, 1972
[4] J. Degauque, B. Astie, J.L. Porteseil, R. Vergne, J. Magn. Magn. Mater., 26, 261, 1982.
[5] J. Degauque, Solid state phenom., 35-36, 335, 1994.
[6] R. Ranjan, D.C. Jiles, P.K. Rastogi, IEEE Trans. Magn., MAG-23, No. 3, 1869-1876, 1987.
[7] D.C. Jiles, J. Phys. D: Appl. Phys. 21, 1186-1195, 1988.
[8] E. Arzt, Acta mater., Vol 46., No. 16, pp. 5611-5626, 1998.
[9] D. H. L. Ng et al., IEEE Transactions on Magnetics, Vol. 37, No. 4, 2734-2736, 2001
[10] J.W. Byeon, S.I. Kwun, Mat. Trans. 44, 10, 2184-2190, 2003.
[11] M.J. Sablik, F.J.G. Landgraf, IEEE Trans. Magn., 39, 2528, 2003.
[12] A. Martínez-de-Guerenu, et al., Acta Mater, V52, no. 12, pp. 3657-3664, 2004.
[13] K. Gurruchaga, et al., Metallurgical & Materials Trans. A, V 41A, Issue 4, pp. 985-993, 2010.
[14] B.A. Auld, Acoustic Fields and Waves in Solids, John Wiley, NY, 1973
[15] F. Stanke, G.S. Kino, The Journal of the Acoustical Society of America, Vol 75, 665, 1984.
[16] M. Dubois, A. Moreau, M. Militzer, J.F. Bussiere, Scripta Mater. V39, pp. 735-741, 1998.
[17] Y. Guo, R. B. Thompson, and F. J. Margetan, AIP Conf. Proc. V657, pp1347-1354, 2003.
[18] S Yadegari et al., Computational Materials Science 84 (2014) 339-349
[19] SMT Mousavi et al., Acta Materialia 67 (2014) 239-251
[20] G Anand et al., Materials Science and Technology 30 (2014) issue 9, 1086-1093
[21] W.F. Brown, Micromagnetics, Interscience Publishers, New York, 1963.
[22] S. Labbé, P.Y. Bertin, Journal of Magnetism and Magnetic Materials 206 (1999) 93-105.
[23] S. Labbé, SIAM Journal on Scientific Computing 26 (2005) 2160-2175.
[24] M. Stolzenberg et al., Stahl und Eisen, Vol. 7, pp 80-90. 2012.
[25] M. Stolzenberg et al., Technical Steel Research, 2013, EC Publication, ISBN 978-92-79-28961-3.
[26] A. Skarlatos et al., WCNDT 2016, http://www.ndt.net/article/wcndt2016/papers/we2h3.pdf
[27] J. Kroos et al., Technical Steel Research 2005, EC Publication ISBN 92-894-9631-2.
[28] W.Zhu et al., WCNDT 2016, http://www.ndt.net/article/wcndt2016/papers/mo1g3.pdf
[29] http://www.ansys.com/en-GB/products/electronics/ansys-maxwell
[30] P. Meilland and P. Lombard, WCNDT 2016, http://www.ndt.net/article/wcndt2016/papers/we1h2.pdf
[31] http://www.altairhyperworks.com/product/flux
[32] L.Zhou et al., WCNDT 2016, www.ndt.net/article/wcndt2016/papers/tu2h2.pdf