In-line Characterisation of Microstructure and Mechanical Properties in the Manufacturing of Steel Strip for the Purpose of Product Uniformity Control

Frenk VAN DEN BERG 1, Piet KOK 1, Haibing YANG 1, Maxim AARNTS 1, Jan-Jaap VINK 1, Willem BEUDELING 1, Philip MEILLAND 2, Thomas KEBE 3, Mathias STOLZENBERG 4, David KRIX 5, Anthony PEYTON 6, Wenqian ZHU 6, Ane MARTINEZ-DE-GUERENU 7, Isabel GUTIERREZ 7, Denis JORGE-BADIOLA 7, Kizkitza GURRUCHAGA 7, Peter LUNDIN 8, Arno VOLKER 9, Mariana MOTA 9, Johan MONSTER 9, Håkan WIRDELIUS 10, Claudio MOCCI 11, Gianluca NASTAS 11, Valentina COLLA 11, Claire DAVIS 12, Lei ZHOU 12, René SCHMIDT 13, Stéphane LABBE 14, Christophe REBOUD 15, Anastassios SKARLATOS 15, Tomas SVATON 15, Vincent LECONTE 16, Patrick LOMBARD 16
1 Tata Steel, IJmuiden, Netherlands
2 ArcelorMittal Global Research and Development Maizières Process, Maizières-lès-Metz Cedex, France
3 thyssenkrupp Steel Europe AG, Duisburg, Germany
4 Salzgitter Mannesmann Forschung GmbH, 38239 Salzgitter, Germany
5 Salzgitter Mannesmann Forschung GmbH, Duisburg, Germany
6 University of Manchester, Manchester, UK
7 CEIT, San Sebastián, Spain
8 Swerea KIMAB AB, Kista, Sweden
9 TNO, Delft, Netherlands
10 Chalmers University of Technology, Göteborg, Sweden
11 Scuola Superiore Sant’Anna, Ghezzano, Pisa, Italy
12 University of Warwick, Warwick Manufacturing Group (WMG), Coventry, UK
13 ArcelorMittal, Eisenhüttenstadt, Germany
14 Université Grenoble Alpes, Grenoble, France
15 CEA Commissariat à l’Energie Atomique, Gif-sur-Yvette, France
16 CEDRAT, Meylan, France

Contact e-mail: frenk.van-den-berg@tatasteel.com

Abstract. The uniformity of the microstructure of steel strip over the entire coil length and between different coils of the same grade is key to stable and consistent material behaviour in steel manufacturers’ proprietary processes, like rolling and levelling, and customers’ processes, like pressing and deep-drawing. In particular for high-strength steels, like dual phase and complex phase steels, the microstructure is very sensitive to processing variations resulting in a potentially larger spread in the mechanical properties of the product.

In July 2013, a large European consortium consisting of 15 institutes started an RCFS [1] – funded project called “Product Uniformity Control” (PUC) with the primary objective to achieve enhanced and sustained product uniformity of steel strip by improved interpretation of data from inline measurement methods that aim...
for real-time and non-destructive characterisation of microstructure and techno-
mechanical parameters. Commonly, these techniques are based on electromagnetic
(EM) or ultrasonic (US) measurement principles, which are favoured because of
their non-destructive and potentially contact-free nature.

To improve the techniques for inline materials characterisation, the PUC
consortium takes a systematic approach to investigate the interrelations between
mechanical properties -- microstructural parameters -- EM & US properties -- inline
measurement thereof. The studies involve dedicated laboratory experiments,
modelling of the EM and US properties of steel, modelling of inline measurement
setups and statistical analysis of data from inline measurement systems. The
synthesis of these activities should result in improved, model-based, calibrations and
finally in a broader deployment and integration of the inline material
characterisation techniques in steel manufacturing, adding value to the product and
enhancing the process efficiency throughout the production chain from hot-rolling to
finishing.

This paper outlines the project approach, highlights interconnecting modelling
and experimental research work, and demonstrates first results. Various
contributions being presented at this WCNDT conference originate from the
 collaborative activities of this PUC project.

1. Introduction

In the steel industry, there is a continuous drive to cost-effective production of high quality
products. In particular the manufacturing of high-strength steels, having much more
complex microstructure than the “conventional” steels, demands a tighter process control
window. To dynamically adjust the target settings of the process parameters, inline
measurement instrumentation is required to characterise the microstructure state of the
(semi-)finished product. These measurement systems can aid in the further optimisation of
quality consistency and yield along the major part of the value chain of steel production.

Such equipment typically measures certain physical properties in a non-destructive
and contact-free manner over the entire length of a coil of steel strip, with a length
resolution in the order of metres. Commonly, electromagnetic (EM) or ultrasonic (US)
principles are applied in these measurement instruments, because of their demonstrated
sensitivity to microstructure (described in a later section) and their potential to operate in a
robust way in the harsh environment of steel processing lines.

The rationale to employ EM and US measurement instrumentation for inline
probing of the microstructure and mechanical properties is schematically depicted in Fig. 1.
The microstructure of steel governs the mechanical properties (like yield strength and
tensile strength), as well as the fundamental EM and US properties (for instance magnetic
coeercivity and ultrasonic damping). The dedicated inline equipment has been optimised for
fast data acquisition and for compliance with the conditions at the industrial process line,
therefore employing measurement techniques which usually probe associated or secondary
EM and US properties (for example harmonics of a hysteresis loop), from which the
fundamental properties may be inferred rather than directly determined. The measured
value is often also influenced by (varying) measurement conditions, like strip speed, strip
tension and lift-off deviations due to fluctuations of the pass line. For these reasons, the
measurement instrument produces an output parameter that is often related to, but not an
exact representation of, a given fundamental EM or US property. Figure 1 also shows the
potential to use the inline measured parameter for characterisation of the mechanical
properties, via their common link with the microstructure. Moreover, with the production
process determining the microstructure, the inline measurement can be used for process
control improvements and feed-back based process control.
Fig. 1. Graph depicting the link between: Mechanical properties - Microstructure – the
Measurand - online Measured ElectroMagnetic (EM) & UltraSound (US) parameters, as obtained by
online measurement systems. Linking the online measured product data with process data enables
identification of improvements in process execution and process control.

The relations represented by the arrows in Fig. 1 are however highly complicated
and have been the topic of various Research and Development projects in the European
context, like OMC [2], NANDACS [3], PLATEND [4], MicroControl [5] and NUSIMAG
[6]. Notably the OMC project [2] studied the methods IMPOC (measurement of residual
magnetic field), HACOM (harmonic analysis of magnetic loops) and LUS (Laser-based
UltraSound) for the purpose of online materials characterisation. OMC has shown that the
measurement results of magnetic systems depend not only on grade but also on variations
in measurement conditions like strip speed, tension and thickness. These and also many
other, often calibration related, issues could not be well explained and resolved, evidencing
the need for a more profound fundamental understanding of the underlying principles based
on (ab initio) modelling. The latter approach is addressed in the present European project
called Product Uniformity Control (‘PUC’), counting 15 different partnering institutes (see
author list). Amongst its partners are 4 major European steel producers (Tata Steel,
ArcelorMittal, thyssenkrupp Steel Europe, Salzgitter Mannesmann). The project started in
July 2013 and will last until December 2017.

2. Model and Validation Chain

In the PUC project, the objective is to detect deviations in microstructure based on inline
measurements. This goal concerns however an inverse problem, which is by definition
difficult to solve. Instead, we use a direct approach, attempting to solve the following
problem: Given a certain microstructure, what are its EM and US properties, and what is
the predicted output of instrumentation probing these properties? By model-based
simulation of instrument response for many cases in the microstructure parameter space,
databases can be built. These will feed surrogate models, which, in combination with
knowledge on the product and the process, can relate changes in instrument output to
changes in microstructure and mechanical properties.

Fig. 2 depicts schematically the model and validation chain. In the model chain (left
in Fig. 2), the microstructures are generated with a dedicated simulation tool (described in
the next section). These microstructures are fed into models predicting the fundamental
electromagnetic (EM) and ultrasonic (US) properties. Again these fundamental EM and US
properties are fed into models that predict the output of measurement instruments, like
IMPOC, 3MA or LUS.
The right side of Fig. 2 depicts the experimental characterisation of respectively the microstructures, EM and US properties and NDE instrumentation. These characterisations are performed either on industrial samples (from the participating steel companies) or on reference samples produced in the laboratory. The results from the experimental work serve as input and validation data for the model predictions (central part of Fig. 2).

3. Modelling of Microstructures

For the prediction of the fundamental EM and US properties of a given microstructure, it is important to start from a well-defined 3D microstructure geometry. Experimental techniques to retrieve these data in statistical relevant quantities are however not readily available. Hence, within PUC, we rely on microstructure geometry simulation to generate

Fig. 3. Examples of numerically generated microstructures using multi-level Voronoi techniques. Top row, from left to right: IF, MA, DP; Bottom row, from left to right: DP (banded), Complex Phase, TRIP.
the required data using (experimental or theoretical) distributions of the microstructure parameters as input. Experimental distributions of microstructure parameters are generally retrieved from EBSD (Electron Back Scatter Diffraction) data or extracted from LOM (Light Optical Microscopy) or SEM (Scanning Electron Microscope) images.

The tool to generate 2D and 3D microstructure geometries has been developed at Tata Steel. It uses an advanced Voronoi based algorithm (Multi-Level Voronoi) which is capable of simulating realistic microstructure constituents (e.g. grains and particles) which have complex, non-convex, morphologies. In [7] the principle of the algorithm is explained and used for 3D crystal plasticity simulations in TRansformation Induced Plasticity (TRIP) steel grades. Examples of a wide range of applications can be found in [8] and [9]. As output the Multi-Level Voronoi programme produces geometric data files, which can be direct input for finite element mesh or a rectangular grid (pixel or voxel based). Properties can be assigned to phase and grain identifiers.

The microstructure simulation tool offers the advantages of generating many microstructures in a cheap and quick manner; Examples are given in Fig. 3 for Interstitial Free (IF), Micro-Alloyed (MA), Dual Phase (DP), Complex Phase (CP) and TRIP steel where grain boundaries and different phases (black or white for DP, CP and TRIP steels) can be seen. Additionally, extreme microstructures can be generated to enlarge the spectrum of microstructures that is yet practically accessible, and any microstructure parameter, like texture, secondary phases or dislocations, can be readily introduced, to investigate its individual contribution.

Models simulating the magnetic properties must rely on 3D simulations, as 2D simulations have reduced significance due to the inherent lower degree of freedom for the magnetic field lines. Certain US simulation cases also have to be carried out in 3D to properly account for scattering losses and anisotropic wave propagation, specifically in studies to the effect of grain size, morphology and texture. Fig. 4 shows three examples of 3D simulated DP steel microstructures, with an increasing volume fraction of second phase (martensite), i.e. 20, 40 and 80%. Here, for simplicity, the secondary martensite phase has the same (average) grain size as ferrite. In a more realistic case, the martensite grain size will be much smaller than the ferrite grain size, and distributed at the grain boundaries of the ferrite, like in the example DP microstructures that are displayed in Fig. 3.

For certain situations, the simulation of a large volume may be necessary. Normally, this would pose problems due to limits in computer memory and computation time. This situation may be solved by the introduction of periodic boundary conditions. A demonstration of the periodicity feature of the numerically generated microstructure is given in Fig. 5. A close view on the previous figure, Fig. 4, may convince the reader that also these 3D microstructures can be stacked to form larger periodic structures.

Fig.4. Examples of numerically generated 3D microstructures with increasing volume fraction of martensite (black grains). Ferrite grains are given different colour to enhance visibility.
As a final example, Fig. 6 demonstrates that also phenomena that happen at a completely different dimensional scale, like precipitates, can be successfully introduced in this microstructure representation. Not only the way in which the precipitates are distributed, but also their concentration and their size (or size distribution) can be tuned to the needs of the user.

The preceding paragraphs clearly illustrate that the approach using Multi-Level Voronoi simulation offers a versatile tool to include many microstructure parameters, like:

- Grain size (mean)
- Grain elongation (aspect ratio)
- Texture
- Secondary phase fraction and distribution
- Precipitate density and precipitate size
- Dislocation pile-up at grain boundaries

![Fig. 5. Example of a periodic microstructure. The base blocks have been presented with a gap between them for better visibility. The periodicity features allows the construction of larger microstructures using the basic microstructure block. Periodicity can be introduced for both the 2D and 3D structures.](image)

![Precipitates randomly distributed](image)

![Precipitates concentrated at grain boundaries](image)

**Fig. 6.** Representation of precipitates, in a random distribution, and distributed at the grain boundaries.

### 4. Electromagnetic and Ultrasonic Properties in Relation to Steel Microstructure

The sensitivity of EM and US properties to steel microstructure has been established in many scientific papers. For example, the relation of the magnetic coercivity as function of microstructural parameters is scattered over many studies: as a function of (a) grain size, in [10,11], (b) dislocation density in [12,13], (c) precipitation density in [14], (d) degree of recovery and recrystallisation in [15-17], and (e) carbon content [13,18-21].

With regard to ultrasonic properties, theoretical foundations by [22, 23] have been experimentally tested by [24-27] (and, together with improvements in laser-based ultrasonic measurement technology, in [28,29]), proving the relative correlations with microstructure, and simultaneously showing the need to include the effects from precipitations, residual stress, texture and complicated microstructure into wave scattering models. Studies on magnetic anisotropy [30-35] and ultrasonic anisotropy [36-44] to evaluate texture, underline the challenge to disentangle the contributions from texture, stress and strip tension from the signal.

The observation of the strong coupling of different microstructure phenomena on the macroscopic physical behaviour of steel has motivated the consortium to develop multi-scale models to enhance the understanding of the relation of the EM and US properties with the steel microstructure.
5. Simulation and Validation of Fundamental Properties

5.1 Electromagnetic Properties

Ab initio modelling of the magnetic behaviour of steel is carried at Université Grenoble Alpes by numerically solving the micro-magnetic energy functional, which includes the 5 terms: Spin exchange energy, Magnetocrystalline anisotropy, Zeeman Energy, Magnetomechanic energy and Demagnetizing energy. Due to the fine spatial resolution that is required to resolve the domain walls, the numerical computation of micromagnetic problems demands high computational power and the solutions are often limited to small volumes. For the computation, a dedicated software environment EMicroM has been developed, which can be used to visualise the evolution of the magnetic domain structure during a change of the applied magnetic field. This type of modelling includes pinning effects arising from grain boundaries and irregularities in the lattice. More details can be found in a separate contribution to this conference [45]. Experimental input and validation data on magnetisation curves on samples are collected using a strip tester at CEIT and an Epstein frame at thyssenkrupp Steel.

In a special focus to evaluate the phase volume ratios of multi-phase steels, the University of Warwick focuses on the modelling of the effective initial magnetic permeability of multi-phase steels. This modelling is performed by importing the 3D microstructure models into the software package COMSOL [46], and using values for the magnetic permeability for the individual metallurgical phases that are either obtained from literature or from experimental characterisation by one of the PUC project partners. Further details are given in another conference contribution [47].

Contributions [45] and [48] respectively address the change of electromagnetic properties with grain size and increasing dislocation density at the surface due to an increase in skin pass level. The poster session [49] compares different parameterisations of (experimental) BH curves to be used in modelling of the instrument response of EM-based NDE systems like IMPOC and 3MA.

5.2 Ultrasonic Properties

The ultrasonic properties of steel are simulated using two complementary modeling techniques, i.e. an analytical and a numerical approach, which are progressed by Chalmers University and TNO respectively.

The analytical approach (by Chalmers) models the interaction between the ultrasonic energy and the grain as a scatterer and damper based on its material properties together with prescription of the material properties of assumed homogeneous bulk material. This means that each specific representative grain can be specified as an individual or be prescribed with material properties based on a statistical distribution or some other predefined functionality. The mentioned interactions have been built into the ultrasonic simulation environment (simSUNDT) used at Chalmers.

TNO uses the 2D or 3D microstructures from the Voronoi simulations by Tata Steel as input and next solves the ultrasonic wave equations using a finite difference method. Snapshots of US wave amplitudes can be visualized in the microstructure as well as the transient of the US signal ‘recorded’ at the surface.

Validation of the models is covered by the institutes Salzgitter Mannesmann and Swerea KIMAB. Details, model results and validation of these methods are given in this conference [50,51]. These papers address effects from grain size and texture.
6. Simulation of NDE equipment

In this conference, a number of contributions are attributed to the simulation and the model validation of the NDE techniques of Laser-based UltraSound (LUS, paper [50]), EMspec [52], IMPOC [53] and 3MA equipment [54], which are modelled by Chalmers, University of Manchester, CEA and CEDRAT respectively. For ultrasonic techniques, both Salzgitter Mannesmann and Swerea KIMAB provide validation data, while for the electromagnetic instruments, the steel companies Tata Steel, thyssenkrupp Steel and ArcelorMittal provide validation data with laboratory versions of the NDE equipment.

7. Characterisation of Product Uniformity

Fig. 7 shows an example from Salzgitter Mannesmann how inline measurements can be used to monitor the uniformity of the mechanical properties over the length of a coil. Here predictions for the yield strength (Rp) and tensile strength (Rm) over coil length are produced based on inline data from the HACOM system, after extensive calibration and compensation of the signal for variations in the measurement conditions. Fig. 7 shows the Rp (red) and Rm (blue) curves for a dual phase (DP) steel after passage through the continuous anneal line, in conjunction with the required tolerance windows (the light-gray bands).

Instantaneous presentation of such data to operators and quality engineers aids in quality assurance, yield and logistics by customer-oriented routing of material. Another example of inline monitoring is given in contribution [55], where the partner Scuola Superiore Sant’Anna has analysed data from the IMPOC system recorded at production lines at Tata Steel and at thyssenkrupp Steel Europe on the traces from skid bars in the furnaces at the reheating stage prior to hot rolling.
8. Conclusions & Outlook

The research work within the PUC consortium is destined to fill gaps in knowledge and equipment concerning the relationships that have been illustrated in Fig. 1. Since these relationships are complicated, having many influencing factors, which are often coupled, the project strongly relies on (multi-scale) modelling to study the dependency on single parameters and to enhance the understanding of the coupled effects.

A particular useful tool for this work is the numerical microstructure generator based on multi-level Voronoi techniques, which is capable of generating virtual microstructures with realistic appearance and distributions. This tool allows investigation of the effect of a single microstructure parameter at a time and hence in combination with models that predict the fundamental EM and US properties for these virtual microstructures, a ranking can be made of the influence level of the individual microstructure parameters on the physical properties. In combination with models for the inline NDE sensing equipment and microstructure-based prediction models for the mechanical properties, we can prospect robust continuous monitoring of mechanical properties. Results of modelling and experimental work on specific topics are highlighted in eleven further contributions to this conference [45, 47-56].

Acknowledgement

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] RFCS = Research Fund for Coal and Steel, see http://ec.europa.eu/research/industrial_technologies/rfc_en.html