

## **MATERIALS CHARACTERIZATION A CHALLENGE IN NDT FOR QUALITY MANAGEMENT**

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### **ABSTRACT**

NDT is mainly discussed as an engineering technology to detect, classify and size material imperfections, called material defects. Lattice defects and microstructure inhomogeneities like vacancies, dissolved atoms dislocations and precipitations are normally not discussed as defects but they are influence parameters which mainly predict the macroscopic properties, the physical properties as well as the mechanical-ones. In order to keep these properties constant in materials production more and more material characterization by NDT is integrated in the production processes. So far material ageing by thermal influences or by fatigue is a task for inspection ND-materials characterization – mainly by micromagnetic techniques - is also introduced in lifetime management strategies.

**Keywords:** ND-materials characterization, material property determination, ageing

### **INTRODUCTION<sup>1</sup>**

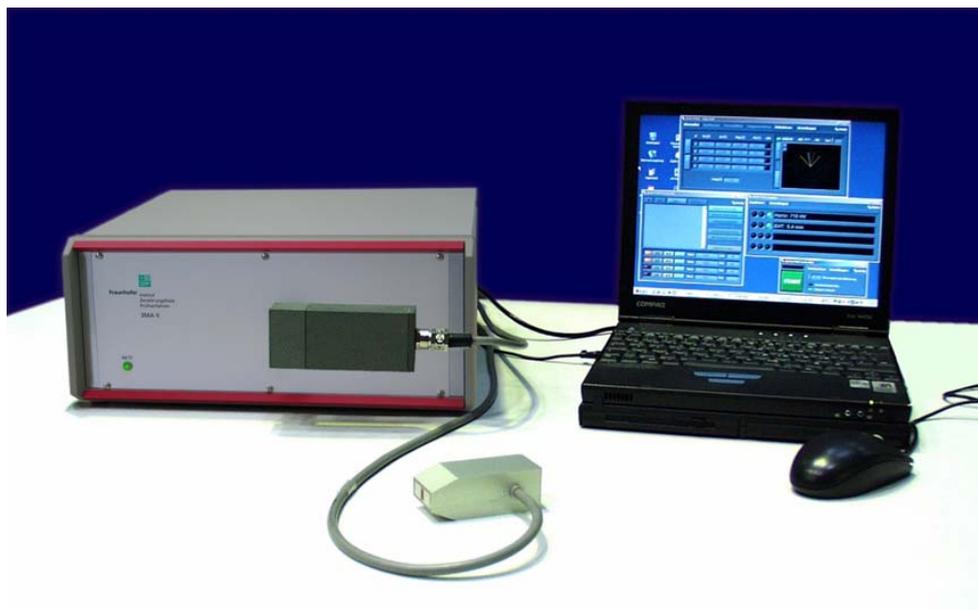
The reason to develop 3MA (Micromagnetic-, Multiparameter-, Microstructure-, and stress-Analysis), starting in the late seventies in the German nuclear safety program, was to find microstructure sensitive NDT techniques to characterize the quality of heat treatments, for instance the stress relieve of a weld. George Matzkanin [1] just has had published a NTIC report in the USA to the magnetic Barkhausen noise. The technique was sensitive to microstructure changes as well as to load-induced and residual stresses. Therefore a second direction of research started in programs of the European steel industry and the objective

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<sup>1</sup> The paper with a slightly different abstract was first presented at the ENDE 2007 Conference in Cardiff.

was to determine residual stresses in big forgings. Beside the magnetic Barkhausen effect also a magneto-acoustic-one became popular [2]. The technique has based on acoustic emission measurements during a hysteresis cycle and was – because of the high amplification – also sensitive to electric interference noise. Therefore the acoustic Barkhausen noise technique has never found a real industrial application. Later further micromagnetic techniques were developed: the incremental permeability measurement, the harmonic analysis of the magnetic tangential field and the measurement of the so-called dynamic or incremental magnetostriction by use of an EMAT [3, 4]. The methodology of the Micromagnetic-, Multiparameter-, Microstructure-, and stress- Analysis (3MA) in detail is described in [4]. On the basis of a multiple regression model, describing target quantities like hardness or yield strength as a function of measured micromagnetic quantities, the unknown model parameters are determined in a calibration step. By using a least square approach the unknown parameters are the solution of a system of linear equations. 3MA only can be applied at ferromagnetic materials. Here the techniques are especially sensitive to mechanical property determination as the relevant microstructure is governing the material behavior under mechanical loads (strength and toughness) in a similar way as the magnetic behavior under magnetic loads, i.e. during the magnetization in a hysteresis loop. Because of the complexity of microstructures and the superimposed stress sensitivity there was a need to develop the multiple parameter approach.

Whereas the first generation of 3MA equipments was basing on the magnetic Barkhausen noise and magnetic tangential field analysis only, 3MA equipment exist now in the forth generation also integrating incremental permeability and eddy current impedance measurements (see Figure 1). More than 100 installations are in use in different industrial areas. This mainly covers the steel and machinery building industries.



**Figure 1.** TCP-IP-based 3MA equipment and software in combination with a laptop

## 1. Applications in the steel industry

### Steel strip inspection

A lot of experiences with 3MA in the last 2 decades were to the continuous mechanical property determination at steel strips, designed to produce car bodies [5, 6], running with a speed of 300m/minute for instance in a continuous galvanizing and annealing line. Yield strength ( $R_{p0.2}$ ), tensile strength ( $R_m$ ), planar and vertical anisotropy parameters ( $r_m$ ,  $\Delta r$ ) are in the focus of quality assurance measures [4], all of them are defined by destructive test and cannot be measured continuously. Therefore 3MA correlations were calibrated.

Figure 2 shows a yield strength profile along a coil of 2.5 km length [5]. At the beginning and the end an unacceptable increasing of strength is detected higher than the upper acceptance level (blue line). The strength values are calculated by the 3MA approach from measured micromagnetic data. The red dots indicate the selection of specimens taken to destructive verification tests after performing NDT. The residual standard errors found by validation are in the range 4-7 % concerning the yield strength. Figure 3 shows a 3MA installation in the line of a strip producer; a robot is used to handle the transducer.

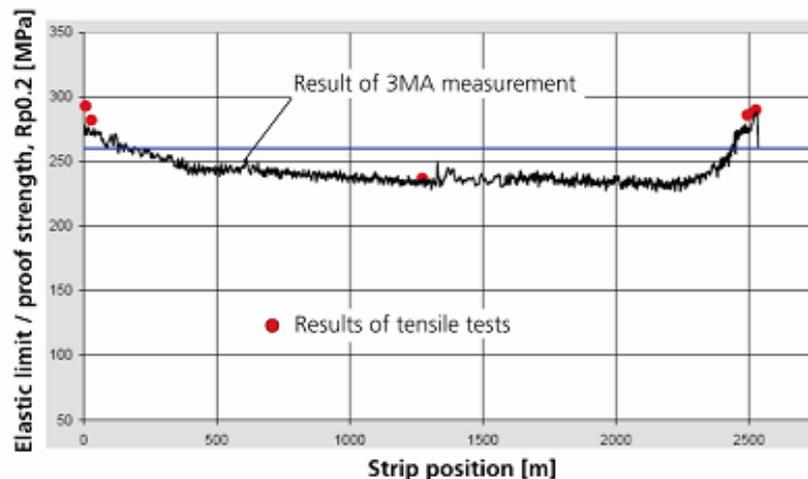


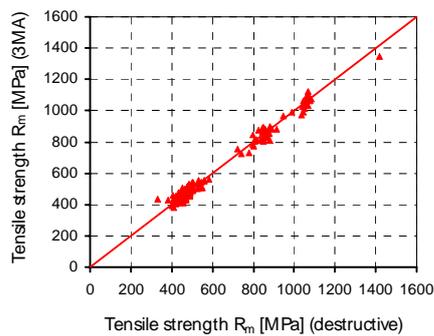
Figure 2. 3MA predicted yield strength [5]



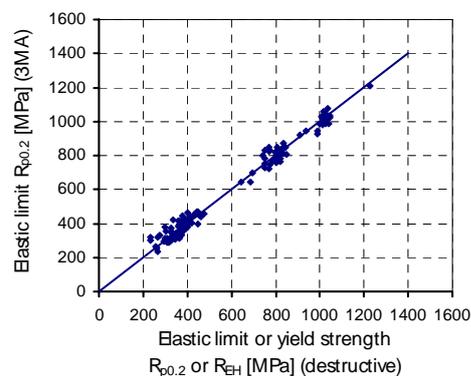
Figure 3. 3MA probe with robot at a strip line

## Heavy plate inspection

Ongoing research is to heavy plate inspection. The steel producer asks for the measurement of geometrical and mechanical properties, which have to be uniform along the product length and width, especially in the case of high-value grades used in off-shore application. Destructive tensile and toughness tests are performed by highly qualified and certified personnel according to codes and delivery conditions. The tests cannot be integrated into online closed loop control with direct feedback. To reliably test the mechanical hardness the surface must be carefully prepared by removing scale and decarburized surface layers and residual stresses are to relieve. The extraction of the test pieces and testing is very time and cost extensive. Costs in the range of several thousands Euro per year arise in a middle-sized heavy plate plant only by destruction of the test pieces.



**Figure 4.** Tensile strength predicted by 3MA [6]



**Figure 5.** Yield strength predicted by 3MA [6]

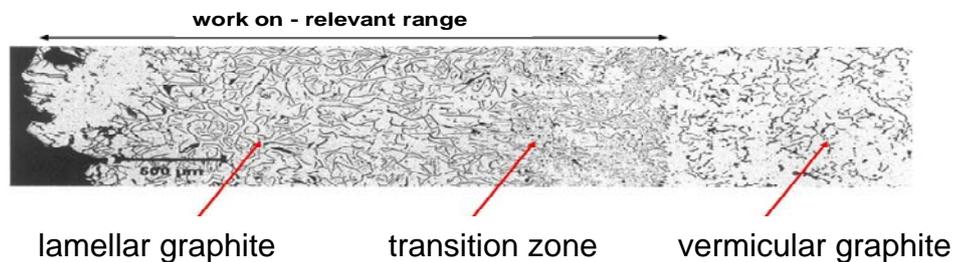
In case of a mother plate of several meters length the edges are usually subjected to other cooling conditions than the rest. Indeed, especially the plate ends are known to cool faster, generating an undesired increase in tensile strength  $R_m$  and yield strength  $R_{p0.2}$ . State-of-the-art is to cut-off the plate edges with non-conform properties based on empirical values concerning the cut-off length. As the destructive tensile test follows directly after the cut-off process of the edges, only the result of these tests can reveal the selection of a not appropriate cut-off length. This results in high costs due to reworking, pseudo-scrap and delayed shipment release; the European steel producers estimate their annual costs in the range of 11 million Euros. Knowing exactly the contour of the zone with unacceptable material properties would allow an open loop control of the cut-off process. Therefore heavy plate producers will replace the destructive quality inspection of test pieces by a NDT technology [6] applying 3MA (see Figure 4 and 5). By a manufacturer-specific calibration residual standard errors of 10 MPa ( $R_m$ ), 20 MPa ( $R_{p0.2}$ ), and 4HB in the Brinell hardness can be obtained. It should mention here that in the 3MA calibration also other measuring quantities can be integrated so far they provide other independent information, for instance elastic properties. By using ultrasonic waves propagating in thickness direction, i.e. a compressive wave excited by a piezoelectric transducer (index L) and two linearly polarized shear waves (polarized in, index SHR, and transverse, index SHT, to the rolling

direction) excited by a EMAT, normalized time-of-flight quantities can be derived describing crystallographic texture effects. Taking into account these quantities ( $t_{SHR}/t_L$ ,  $t_{SHT}/t_L$ ,  $(t_{SHR}-t_{SHT})/t_L$ ) together with the micromagnetic parameters then a regression result is obtained again reducing the residual standard error.

## 2. Application in automotive and machinery building industry

### Car engine casting

To reduce the weight of the power supply unit the car combustion engines cylinder crankcases can be made of cast iron with vermicular graphite (GJV), because this material in a Diesel engine allows a higher loading pressure even by reduced wall thickness. However, the service life of machining tools is during processing an engine block made from GJV substantially smaller compared with a block from cast iron with lamellar (flake) graphite (GJL).

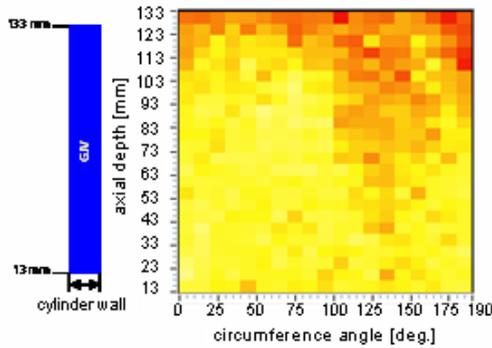


**Figure 6.** Microstructure gradient obtained in a cylinder region of a cast engine [7]

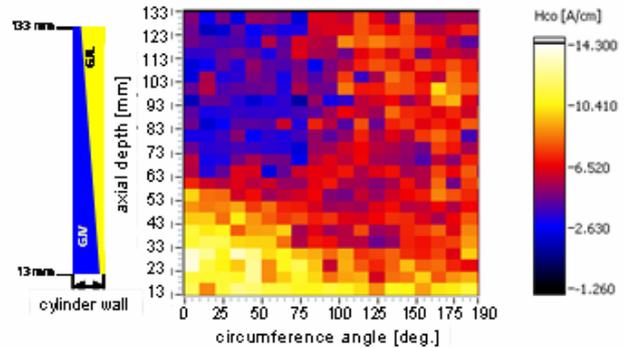
This disadvantage can be eliminated by an innovative casting technology that produces a continuous microstructure gradient in the cast iron from lamellar graphite at the inner surface of the cylinders to vermicular graphite in radial direction. By implementing some chemical additives into the core of the mould which can diffuse in the cast iron during the solidification process in the mould the gradient with a continuous transition from lamellar graphite and finally vermicular graphite is obtained. However, the technology can only be used by the casters so far the gradient quality can be characterized and monitored by NDT. Figure 6 documents in a micrograph such a gradient beginning at the left side with cast iron (inner cylinder surface) and lamellar graphite followed by a transition region and vermicular graphite on the right side.

3MA techniques always cover a certain analysing depth depending on the magnetising frequency and geometrical parameters of the magnetisation yoke, etc. So far the gradient has different graphite compositions within the analysing depth, 3MA quantities should be influenced. Based on measurements at an especially designed calibration test specimen set 3MA quantities were selected to image the gradient with optimal contrast. As reference quantity to calibrate 3MA the local thickness of the GJV-layer was evaluated by using micrographs and optimized pattern recognition algorithms in the microscope. A special designed transducer head was developed to scan the cylinder surface by line scans in hoop direction and rotating the head, then shifting the head in axial

direction to perform the next line scan. Figure 7 and Figure 8 show as example the coercivity images derived from the tangential field strength evaluation ( $H_{C0}$  in A/cm) and line scans covering an angle range of  $190^\circ$ .



**Figure 7.** Coercivity image of a reference block made from GJV

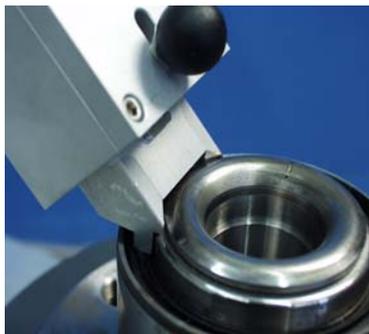


**Figure 8.** Coercivity image of a block with GJV/GJL gradient

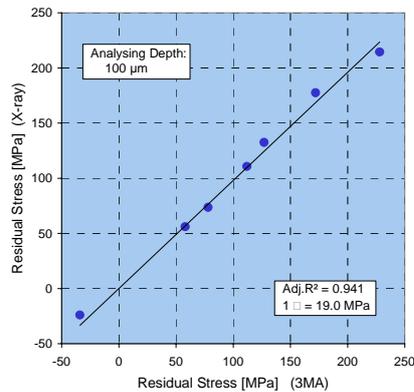
Combining different 3MA quantities in a multiple regression the thickness of the GJL layer was predicted. A regression coefficient of  $R^2 = 0.93$  and a residual standard error of  $\sigma = 0.06$  mm was obtained [7].

### Wheel bearing inspection

The fixation of the inner ring of wheel bearings is performed by a wobble riveting process. As a consequence a residual stress is built up in the ring which may not exceed a limit value of about 300 MPa to get a perfect quality.



**Figure 9.** 3MA-Probe at test location



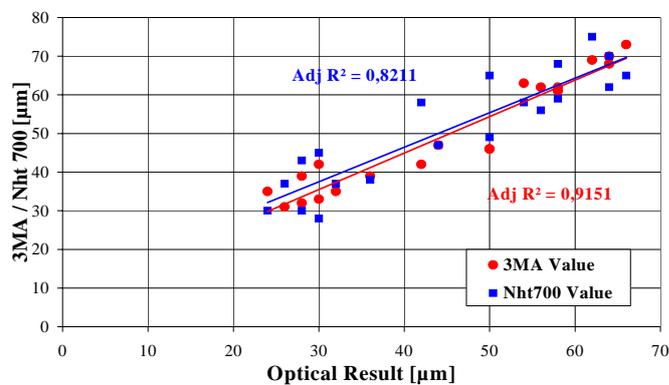
**Figure 10.** Residual stress calibration

The usual technique to inspect the residual stress state is x-ray diffraction which is destructive in nature because it requires a preparation of the test location. Furthermore it can only be performed statistically. The 3MA technique allows a fast non-destructive estimation of the residual stress level (Figures 9, 10). After a calibration step by using x-ray reference values a 100% quality inspection of these parts is possible. The calibration procedure requires a coincidence of the

3MA and x-ray calibration positions because residual stress varies along the circumference. That means the 3MA data have to be recorded in a first step before the x-ray test location is prepared by etching. According to Figure 10 the residual standard error in the calibration is in the 20 MPa range. Besides the residual stress additionally the surface hardness can be measured.

### Evaluation of microstructure and stress gradients

Machined parts in most of the cases have more or less steep gradients in their properties near the surfaces. To improve the lifetime of mechanical highly stressed machinery components the bearing areas are surface-hardened from the  $\mu\text{m}$ - up to the millimetre range depending on the requirements and on the hardening technology.



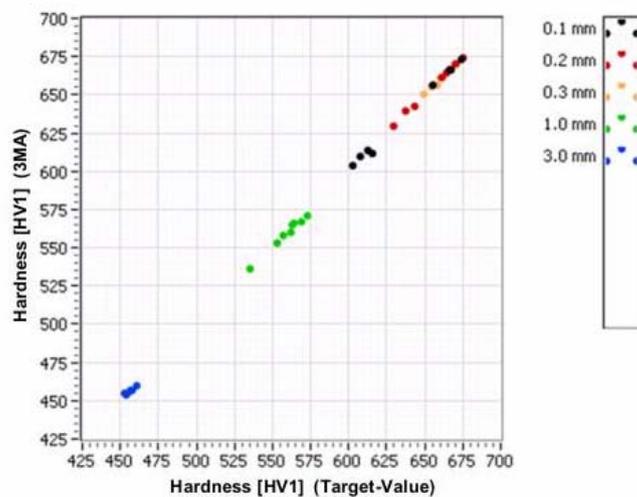
**Fig.11.** Comparison of nitrating hardening depth measured by 3MA and Nht 700 (Vickers) versus optical result

Additional surface finishing by grinding can superimpose surface near defects of microstructure and residual stresses which can result in a part breakdown. To inspect the production quality in many cases not only the properties immediately at the surface but also information of the properties below the surface are desired. 3MA is an effective tool to investigate the properties near the surface as well as the range below the surface up to several millimetres in depth.

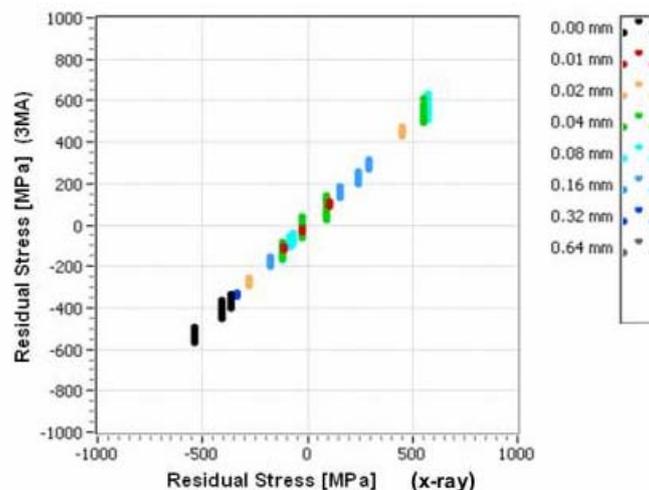
One example of a 3MA application in industry is the determination of nitrating hardening depth NHD of piston rings on the flank side and on the tread surface. Typical values of nitrating hardening depth are between 60 and about 100  $\mu\text{m}$ . It is found by the customer that the reproducibility of the non-destructive values of hardness and hardening depth in piston rings is better than the conventional testing by a metallographic Vickers hardness test (Nht700), as can be seen in Figure 11 [8]. The reason of that behaviour seems to be the difference in the lateral resolution of the conventional and the non-destructive testing method. Due to a diameter of the 3MA receiver coil of about 2 mm the 3MA values are covering a much larger inspection area. Fast data evaluation by 3MA allows a complete production feedback control.

The occurrence of grinding defects, e. g. in gear wheels, is a main problem since many years which is caused by too much heat input during the grinding process. Modern grinding tools allow much higher grinding speed compared to

former machines but on the other side this can result in more defects. To get information on the quality of grinded microstructure states the common method in industry is the nital etching technique. Grinding defects are indicated by the discoloration of the surface. This technique is effective as long as the surface information is sufficient to estimate the quality. But it fails if in a preceding production step defects are produced below the surface which are covered in the next production step by a perfect finishing. Several examples of defective gear wheels investigated by hole drilling method and x-ray diffraction have shown that in a depth of 100  $\mu\text{m}$  high tensile stresses up to several 100 MPa can be present whereas at the surface a perfect compressive state of several hundred MPA has been found. These hidden defects cannot be detected by nital etching. As a consequence after some time small cracks are covering the surface due to stress-relieve even without a mechanical load.



**Figure 12.** Hardness calibration at various depths; hardness values determined by 3MA versus target values



**Figure 13.** Residual Stress calibration at various depths; RS values determined by 3MA versus X-ray reference values

Since several years IZFP has gained experience in the non-destructive detection and quantitative evaluation of such grinding defect gradients by 3MA in cooperation with industrial partners and in different research and

development projects [9, 10]. After a calibration step 3MA can be used to evaluate different target values simultaneously, especially the hardness and the residual stress at the surface and in several depths below the surface (Figures 12 and 13). To get unambiguous results calibration must be done carefully. Calibration is mainly determined by well defined calibration specimens and only valid to the target ranges available by calibration. In most cases calibration is restricted e. g. to the material, to the actual machining parameters and even to the 3MA probe in use. If any variation occurs, its influence on the validation of the existing calibration has to be checked and if necessary a recalibration or extension of the existing calibration has to be performed to include any disturbances. These limitations and the calibration effort may be seen as a disadvantage of 3MA. But if an optimal calibration is developed the fast non-destructive determination of various quality parameters which is desired concerning expensive security related parts justifies this effort.

### 3. Conclusion

3MA is a matured technology and a wide field of applications is given. However, besides the success story we also can find critical remarks from industrial users. These are mainly to the calibration efforts and problems of recalibration if a sensor has to be changed because of damage by wear. Therefore actual emphasis of R&D is to generalize calibration procedures.

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