ELECTROMAGNETIC NDT FOR LIFETIME MANAGEMENT
BY MONITORING OF AGEING PHENOMENA

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ABSTRACT

Starting in 1976, the 3MA-methodology (Micromagnetic, Multiparameter, Microstructure and Stress Analysis) was developed. 3MA has its basis in micromagnetic NDT techniques which are the measurement of magnetic Barkhausen noise, of the magnetic incremental permeability, of the eddy current impedance, the harmonic analysis of the magnetic tangential field strength, and the measurement of the dynamic or also called incremental magnetostriction. All of these techniques ask for a local magnetization in a hysteresis loop and therefore 3MA can only be applied at ferromagnetic materials.

3MA was used to characterize ageing phenomena in pressure vessel and pipeline steels as thermal ageing and neutron degradation as well as material states when thermal ageing and low cycle fatigue were superimposed. In the case of austenitic stainless steels when exposed to mechanical static or cyclic loads – the material reacts localized with phase transformation to bcc α’ martensite - 3MA techniques can be applied. In all other cases UT is utilized and - for instance a time-of-flight-measurement – can characterize cyclic deformation. When the mechanical loading is performed at elevated temperatures (300°C) EMAT are applied.

Keywords: electromagnetic NDT, lifetime management, monitoring, ageing of materials

1. Introduction

In Germany in charge of the minister of economy and technology R&D are performed to enhance nuclear safety. Non-destructive testing, optimization of standardized and development of enhanced techniques is an essential part of the program. Besides the development of NDT for detecting and sizing of macroscopic irregularities (defects), material characterization became an important topic as more as the plants come near the end of the design lifetime and ageing phenomena play a role. The materials are exposed to elevated temperatures (up to 340°C) and to low cycle thermo-mechanical cyclic loads. High cycle mechanical loads can be superimposed, for instance by pumping vibrations. The nuclear pressure vessel in addition is exposed to neutron irradiation initiating embrittlement. Besides the standard ISI in Germany ageing management of the components is code-accepted and therefore technology is also developed on the basis of NDT to on-line monitor ageing phenomena. Fraunhofer-IZFP has in the last decade developed different approaches which will be discussed here in more detail. These techniques are especially important when lifetime enhancement and prolongation is the policy.
2. Thermal ageing of a Cu-rich martensitic steel

2.1 Thermal ageing

Beginning in 1998 Fraunhofer-IZFP in co-operation with the Materials Testing Institute at the University Stuttgart [1] has investigated the low-alloy, heat-resistant steel 15 NiCuMoNb 5 (WB 36, material number 1.6368) which is used as piping and vessel material in boiling water reactor (BWR) and pressurized water reactor (PWR) nuclear power plants in Germany. In all damage situations, the operating temperature was between 320° and 350°C. Even though different factors played a role in causing the damage, an operation-induced hardening associated with a decrease in toughness (-20%) was seen in all cases. The material degradation can be visualized by using micromagnetic NDT [2]. The micromagnetic approach applied by the Fraunhofer-IZFP is introduced in the literature with the acronym 3MA (Micromagnetic, Multiparameter, Microstructure and stress Analysis) which indicates on one hand the multiple dependency of microstructure parameter influences and on the other hand the sensitivity of the selected inspection quantities to applied and residual stresses. The microstructure parameter influences are due to lattice defects as vacancies, dissolved atoms (candidates are C, H, N, Cu), dislocations, precipitates, grain- and phase-boundaries which acts as pinning points to temporarily pin the Bloch-walls of magnetic domains in ferromagnetic materials when magnetized in a hysteresis loop. This fact clearly indicates the application of micromagnetic NDT to ferromagnetic materials or to materials with ferromagnetic phases only. In [2] the 3MA-approach is detailed described as a solution of an inverse problem where micromagnetic parameters are measured and target quantities as mechanical hardness, hardness depth, yield strength, tensile strength, Charpy energy, ductile to brittle transition temperature (DBTT) and others are predicted by regression or pattern recognition algorithms. The micromagnetic parameters measured are basing on magnetic Barkhausen noise, the incremental permeability, the Fourier analysis of the magnetic tangential field strength and a multiple frequency eddy current impedance measurement. The correlation to mechanical properties is evident because the above mentioned microstructure parameter influences, as well as stress, are also the pinning points of dislocation movement under mechanical loads.

Fig. 1. Magnetic Barkhausen noise of the service exposed and recovery annealed material in the mechanical load-induced stress-free state (no loading)
The Cu-rich WB36 steel, according its specification, has in between 0.45 and 0.85 mass% Cu (in average 0.65%) in its composition. The half part of the Cu is in precipitation because of annealing and stress relieve heat treatment during production, the other half still is in solid solution and can precipitate when the material is exposed at service temperatures. The material can obviously be recovery annealed when after the service exposures again is heated-up at the stress relieve heat treatment temperature and hold some time. The precipitates are dissolved again in solid solution obtaining a microstructure state comparable but not identical to the ‘as delivered’ state. Micromagnetic investigations at first were performed at ‘service exposed’ (57,000h at 350°C) and ‘recovery annealed’ (service exposed + 3h 550°C) material using cylindrical (diameter 8mm) test specimens. Whereas the hysteresis curves of the two microstructure states are nearly identical, differences were observed when the magnetic Barkhausen noise was registered (Fig.1) and when the lengthwise magnetostriction was measured (Fig. 2). The specimens were measured in the stress-free state as well under variable tensile load in order to reveal the stress sensitivity of the microstructures.

Fig. 2. Lengthwise magnetostriction measured by strain gages under hysteresis magnetization in case of service exposed and recovery annealed material which is in the load-induced stress-free state (no loading)

The service exposed microstructure has higher Barkhausen noise maximum (Fig. 1) and lower magnetostriction values (Fig. 2). Both effects indicate the influence of tensile residual stresses induced by the Cu-rich precipitates in the iron matrix [1, 2, 3]. In TEM and SANS investigations the precipitation state was studied. The particle size is in between 2nm – 20nm distributed. Particles < 6nm diameter have body centred cubic crystallographic structure like the iron matrix (coherent precipitates). As the atomic radius of Cu is larger compared with iron the Cu precipitate acts with compressive stresses which are balanced by tensile residual stress in the matrix. Particles with diameter > 20nm are face centred cubic and in between these two states a transition crystallographic structure exist. About 50% of the precipitates have this transition structure and especially contribute to micro residual stresses in the tensile stress regime in the matrix.

Further investigations in order to statistically confirm the results were performed at 400°C in order to speed-up the precipitation process. Comparing the coercivity (Fig. 3) derived from the harmonic analysis of the tangential magnetic field strength with the measured Vickers hardness 10 as reference to characterise the thermally aged microstructure both quantities are correlated and meet a typical hardness maximum which is the critical material state for possible failure of a component if the design has not taken into account the strengthening ageing effect. When the exposure times are further enlarged hardness is decreasing by precipitation coarsening. In order
to obtain the good correlation in the 3MA-approach (Fig. 4) beside micromagnetic characteristics eddy current impedances were implemented. These are especially suitable as the Cu precipitates contribute to an enhanced electrical conductivity.

Fig. 3. Coercivity $H_{C0}$ derived from Fourier analysis of the magnetic tangential field strength and Vickers hardness $HV_{10}$

Fig. 4. 3MA prediction of Vickers hardness $HV_{5}$

It is typical in the 3MA approach that a calibration is performed at a set of well-defined calibration pieces. However, the approach is verified by using additionally independently selected test pieces.

2.2 Thermal Ageing superimposed by Low Cycle Fatigue (LCF)

When in addition to the thermal ageing LCF is superimposed the degradation is enhanced. Fig. 5 visualizes this fact by discussing the result of Charpy impact tests. The heat E2A is WB36 material in the ‘as delivered’ state. The largest shift in the DBTT (measured at 41J) is observed in case of 300°C exposure temperature and 2400 s cycling period.

Micromagnetic properties allow the characterizing of this degradation. A clear correlation can be found to the hardness enhancement, Fig. 6 documents the result. Besides E2A two other heats were tested. The E59 material came from a used vessel which at 350°C for 57,000 h was in service. The material was investigated in the state ‘recovery annealed’ (600°C, 3h) named E59 EG. Furthermore, some material of E59 was especially heat treated, ‘stepwise stabilised
annealed in order to stabilise the Cu precipitation distribution in coarse particles, named E59 S4. Compared with E59 EG, E59 S4 is less prone for further precipitation development under service conditions.

![Notch impact energy of specimens E2A fatigued at different temperatures and exposure times](image1)

**Fig. 5.** Notch impact energy of specimens E2A fatigued at different temperatures and exposure times

![Correlation to Vickers hardness 10](image2)

**Fig. 6.** Correlation to Vickers hardness 10

3. **Neutron degradation**

In the case of power plant components, such as pressure vessels and pipes, the fitness for use under mechanical loads is characterised in terms of the determination of mechanical properties such as mechanical hardness, yield and tensile strength, toughness, shift of DBTT compared with not degraded material, fatigue strength, etc.
Table 1: Irradiated material, Charpy specimens

With the exception of hardness tests which are weakly invasive, all of these parameters can be determined within surveillance programs by using destructive tests only on special standardized samples (Charpy V samples and standard tensile test specimens). The specimens are exposed in special radiation chambers near the core of the Nuclear Power Plant (NPP) to a higher neutron flow than at the inner surface of the pressure vessel wall in order to generate a worst case.

![Graph](attachment:image.png)

Fig. 7. Prediction of the change of DBTT ($\Delta T_{41}$) in the case of the pressure vessel base materials, $R$ is the correlation coefficient; RMSE is the residual main standard error
From time to time these specimens are removed from the chambers and used for destructive tests. The number of the samples is limited and in the future it will be very important that reliable non-destructive methods are available to determine the mechanical material parameters on these samples without destruction of the specimens.

Furthermore an in situ characterisation of the reactor pressure vessel inner wall through the cladding is of interest for inservice inspection, additionally to the measurements on samples. To solve this task a combination testing technique based on 3MA and the dynamic magnetostriction measurement by using an EMAT (Electromagnetic Acoustic Transducers) was developed [3]. Algorithms based on pattern recognition are suitable to characterize pressure vessel material (base material and welds) in terms of changes in the DBTT evaluated at the Charpy energy of 41J ($\Delta T_{41}$). Fig. 7 presents the result of the approach in the case of the base materials. It should be mentioned here that a similar result is obtained at weld material taking into account also material of Russian WWER pressure vessels.

4. Fatigue of austenitic stainless steel material

An online monitoring measuring technique was enhanced in co-operation with the Technical University Kaiserslautern and an EMAT sensor was integrated by IZFP [4] into the servo-hydraulic machine. Because fatigue experiments should be monitored at service temperature of 300°C the idea was to integrate ultrasonic transducers in the clamping device of the fatigue specimen and to monitor the ultrasonic time-of-flight (tof) of a pulse propagating from the transmitter to the receiver transmitting the fatigue specimen (Fig. 8).

![Fig. 8. Schematic diagram of wave propagation: wave propagation direction ‘z’ and particle displacement ‘r’ (a), fatigue specimen and EMAT probes with radial polarized wave type (b), clamped fatigue specimens (1) in grips, which enclose the transmitter at the one end and received at the other as well as Ferritescope (2) and an extensometer (3) (c)](image)

Because of the high temperature exposition coupling-free electromagnetic acoustic transducers (EMAT) were used based on a pan cake eddy current coil superimposing a normal magnetic field produced by a permanent magnet. By exciting Lorentz forces radially polarized shear waves are excited [5].
Figure 8 shows the integration of the EMAT into the clamping of fatigue specimens at the servo hydraulic machine. The mean tof-value (Fig. 9 and Fig. 10) measured online shows a distinct behaviour as function of the fatiguing, i.e. the number of load cycles, and is different in the case of ambient temperature fatiguing (Fig. 11) and at 300°C (Fig. 12).

Fig. 9. Time-of-flight (tof) measurement procedure

Fig. 10. Determination of the averaged tof as function of the cycling number N
Whereas the mean tof-values in the case of fatiguing at ambient temperature continuously increase (Figure 11) which is, according to SEM and X-ray diffraction investigations, due to α' cold-forming martensite development this is not to observe when fatiguing at 300°C (Figure 12). In this last case the mean tof-values show after an increase due to strain hardening a decreasing or a saturation effect before the dominant increase to the specimen break due to cracking can be documented. There is no martensite development.

### 5. Conclusions

By use of micromagnetic non-destructive techniques the ability to characterise materials ageing was demonstrated:

- by non-destructive hardness prediction when hardness is enhanced due to Cu precipitation and thermal degradation
- by supplying non-destructively an early warning before fatigue life is elapsed due to Low Cycle Fatigue measuring the time-of-flight of an ultrasonic pulse propagating between transmitter and receiver
- by non-destructively predicting the DBTT 41J shift when degradation of pressure vessel material is due to neutron embrittlement; the world-wide new micromagnetic approach
contains correlations to characterize the neutron degradation of pressure vessel material of western design as well as of Russian design.

The demonstration was at well-defined laboratory-type specimens but a high sensitivity and confidence level of the results was obtained. However, the next development step to perform is the demonstration of the techniques at real life components and the integration in inservice testing respectively in ageing management procedures of real plants. This will in addition include UT by using EMAT. The special emphasis of a next project is the integration of the new NDT-approaches into an existing thermo-mechanical fatigue characterizing system which has its basis on surface temperature measurements [6].

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7. References