NDE OF MATERIAL DEGRADATION BY EMBRITTLEMENT AND FATIGUE

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ABSTRACT. This paper presents an overview on current activities and results concerning NDE of material degradation by embrittlement and fatigue. In general we differentiate between NDT methods for the characterization of embrittlement and fatigue which are either microstructure or crack sensitive. At the present time there is no reliable NDE technique known which can be used on technical components to detect the onset of failure during the early stages of embrittlement and fatigue. Therefore, microstructure sensitive methods are required which have potential for detecting degradation during the early stages and for infield capability. Such methods are micro-magnetic methods, eddy current methods and thermal methods. These techniques, however, have only been tested under laboratory conditions to date. The micro-magnetic method will be discussed concerning embrittlement. Results on eddy current testing by using GMR and thermal methods are documented concerning fatigue characterization.

INTRODUCTION

Service induced degradation of reactor pressure vessels according to licensing regulations is the main limiting factor of the operating life time of nuclear power plants. Degradation of reactor pressure vessel material properties includes a decrease in fracture toughness, an increase in strength and an increase in the fracture appearance transition temperature (FATT). The vessel operating factors, primary neutron irradiation and thermal ageing, have an effect on intrinsic material properties (microstructure) that affect both mechanical and electromagnetic properties. The magnetic and electrical properties influence electromagnetic NDT quantities that may be used to monitor the condition of reactor pressure vessel materials in so far as NDT results reliably correlate with mechanical properties and relevant components are accessible.

In German power plants the copper-alloy ferritic steel WB 36 is used for piping material below an operating temperature of 300°C and for vessel material up to an operating temperature of 340°C. Over the last 10 to 15 years failures to components made of WB 36 have arisen in conventional power plants after long-term operation. Smallest operating-induced copper precipitates with a particle diameter of 1.5 to 3.0 nm can cause material degradation which results in decrease in toughness and an increase in the fracture appearance transition temperature [1]. An example for such damage is a feed water pipe of WB 36 which exploded after 130,000 operating hours at 350° C in the conventional power plant Kardia 1 in Greece in 1998 [2].

The evaluation of early fatigue damage and thus the prediction of the remaining lifetime is a task of practical relevance for example in the chemical, nuclear, as well as in the
aircraft industry. High performance materials e.g. titanium are used in aircraft structures. Especially under high-cycle fatigue conditions more than 90% of lifetime is spent before cracks are usually detectable. The reason for this are small and subsurface cracks generated from fatigue induced compressive stresses within the surface region. Under HCF conditions very small non-detectable cracks can become unstable and result in catastrophic failure.

In the chemical industry, for example, austenitic stainless steels are in widespread use, mainly because of their high toughness and resistance to corrosion attack. However, under static as well as fatigue load the material has the tendency to respond with localized phase transformation from the non-magnetic γ to the martensitic and ferromagnetic α’ phase. Magnetic permeability changes are observed in the martensitic structure development and in residual stress effects where the electrical conductivity is influenced by the changing dislocation density and arrangement. Bearing in mind the tasks faced by industry with regard to titanium and austenitic stainless steels, new approaches to on-line monitoring of fatigue characterization are essential.

MEASURING RESULTS

NDE of Material Degradation caused by Embrittlement

In the framework of a research project [1], a very extensive microscopic characterization of the WB 36 material was performed by Karl-Heinz Katerbau and his group at the University of Stuttgart. The microstructure was investigated with the aid of SANS (Small Angle Neutron Scattering), appropriate for the detection of smallest Cu-precipitates as well as with TEM (Transmission-Electron-Microscopy) suitable for the detection of larger Cu-precipitates. SANS investigations have proved that globular Cu-particles in the range of 1-3 nm are responsible for the decrease in toughness. Larger coherent particles (> 3 nm) are characterized in the TEM by the coffee-bean–like shape as shown in Fig. 1.

At the IZFP an approach has been developed for the determination of residual stresses of the third order, i.e. residual stresses of coherent precipitates [3]. The procedure is based on the load dependent Barkhausen analysis and allows a determination of residual stresses with an accuracy of ± 1.5 MPa. On the basis of this procedure measurements were carried out on service exposed and recovery-annealed specimens originating from a vessel drum of WB 36 [4].

FIGURE 1. WB-36-Cu-precipitations after long-term service.
Between both microstructure states a residual stress difference of about 20 MPa was determined. This value is an integral value. The local residual stress state in the vicinity of a copper precipitate is much higher and can be in the yield strength range.

Investigations by MPA Stuttgart have shown that Vickers hardness is suitable for the quantification of embrittlement though it has the disadvantage that it is not repetitively and area-wide applicable and because spot tests require information about critical test areas. Non-destructive early-detecting of the hardness increase is therefore a most favorable solution for this problem. Based on the analogy between dislocation and Bloch-wall movement, electromagnetic measuring techniques are suitable for the determination of mechanical material properties. The suitability of micromagnetic NDE techniques for the characterization of the Vickers hardness was investigated. A measuring system was successfully calibrated for the prediction of HV 10 by Barkhausen noise and upper harmonics analysis of the magnetic field. Electromagnetic measurements and Vickers hardness measurements were carried out on 12 cylindrical samples which were service-simulated at a temperature of 400° C. All investigated samples reveal a hardness maximum after 1,000 hrs. length of service simulation. The mean increase in hardness is about 40 HV 10. The decrease in the hardness after passing through a maximum is caused by the growth of copper precipitates, the so-called “Ostwald-Ripening” [5].

Fig. 2 shows the magnetic hardness, i.e. a coercivity $H_{\text{co}}$, in comparison with the mechanical hardness, HV 10, as function of length of service simulation. Fig. 3 demonstrates the sufficiently good correlation between non-destructively determined hardness values and mechanical hardness values. The correlation coefficient was better than 0.97 and the error band was smaller than 5 HV 10. In order to prove the practicability of this approach, it was also tested under superimposed tensile loads in order to simulate residual stress influences. The electromagnetic hardness determination succeeds even under such unfavorable outside influences.

Parallel to the activities in Germany in 1999 within the framework of a research program from the Nuclear Regulatory Commission, Donna Hurley at the National Institute of Standards and Technology has carried out non-linear ultrasonic measurements on A710.
Electromagnetic non-destructive hardness determination of WB36 by using upper harmonics and Barkhausen noise analysis

FIGURE 3. Correlation between non-destructively predicted hardness values (HV 10_{NDT}) and Vickers hardness values (HV 10 mech), correlation coefficient $r^2 > 0.97$, and error bandwidth < 5 HV 10.

steels with copper-rich precipitates [6]. The behavior of this steel is similar to that of WB36, although there are minor differences in their respective chemical compositions [6]. The measuring quantity derived from non-linear ultrasonic measurements is $B$ [6]. The non-linearity parameter $B$ was compared with Vickers hardness values. According to Donna Hurley the reason for the deviation is the influence of internal coherency strains surrounding the precipitates on the measuring quantity $B$.

Reactor pressure vessel steels contain only a third of the copper content of WB36. However, also in this case, the embrittlement is caused by copper rich precipitates as a consequence of neutron irradiation. At the IZFP, electromagnetic measurements were carried out on different neutron irradiated samples by using the micromagnetic 3MA device [7].

In a hot cell laboratory with a manipulation system, electromagnetic measurements were carried out on irradiated Charpy-V samples from the Chinon nuclear power plant in France. 5 pairs of samples with different exposure times (0, 4, 7, 9, 14 years) were investigated. Calibration was performed on the base of only 5 samples, one measurement of each exposure time was made, using the fluence value as reference value. Good correlation ($R=0.98$) was obtained between fluence predicted by 3MA and fluence reference values by testing the approach with the remaining 5 specimen.

George Alers at The National Institute of Standards and Technology in Boulder, USA, carried out dynamic magnetostriction measurements by using EMATs on a CT-sample set which was neutron irradiated inside a hot cell. A correlation was found between the shear wave velocity and the neutron irradiation. By the development of a NDE technique for determination of embrittlement in an installed reactor pressure vessel the stainless steel coating on the inside surface of the vessel has to be taken into consideration. This layer is about 10 mm thick and can complicate the penetration of the cladding by using magnetic fields and ultrasonic waves.

The first successful measurement on a pressure vessel section with a 10 mm thick cladding was performed by George Alers. A pair of permanent magnets and an electromagnet were used for exciting an ultrasonic shear wave by magnetostrictive coupling in the ferromagnetic base material. The frequency of this shear wave was the same as that used to drive the electromagnet, about 10 kHz. This low frequency was used for an adequate penetration of the cladding and producing a thickness resonance with respect to the wall thickness.
NDE of Material Degradation caused by Fatigue

Cyclic loading leads to heat dissipation caused by internal friction and microplasticity. Excitation of this thermal effect within the specimen was achieved in a servo hydraulic fatigue machine and temperature measurements were made using an infrared camera.

At low cycling frequencies, three stages of the temperature evolution can be observed. Measured after an approximately linear increase of the temperature at the beginning (stage 1), the slope of temperature development with time, \( \frac{dT}{dt} \), decreases gradually (stage 2) due to heat losses (heat conduction, radiation and convection). Finally, an equilibrium temperature is reached, when the heat generation is balanced by the heat losses to the environment (stage 3). This equilibrium temperature has been mostly used for the characterization of fatigue because it depends on the dissipated heat energy which is connected with the area under the mechanical hysteresis. However, the thermal boundary conditions also have a strong influence. In a new approach [8] the temperature change per mechanical loading cycle during stage 1 was measured. It describes the initial slope of the temperature increase. This temperature change does not depend on the thermal boundary conditions because of the adiabatic behavior during the early stages of the loading. The approach was performed in order to develop potential for reliable aircraft component monitoring. It offers the capability of rapid, non contact, high resolution characterization applicable for technical components. For the thermal measurement the fatigue experiment was interrupted at specific cycle numbers. After cooling down the titanium specimen was then subjected to a short-term mechanical loading of approximately 90 cycles at higher frequency cycling. From the so initiated temperature evolution the temperature rise per cycle was calculated. These quantity monotonically increases with the number of loading cycles and is a suitable measuring quantity to characterize fatigue.

For characterizing the fatigue behavior of austenitic stainless steel a GMR-sensor was used. The GMR was controlled by standard eddy-current equipment. In this experimental set-up an electromagnetic yoke is used as a transmitter coil and the GMR as the receiver. Fig. 4 shows a cyclic deformation curve \( \varepsilon_{ap} \) as function of the load cycles obtained at room temperature and at a load of 380 MPa (stress-controlled experiment). Plastic deformation occurs from the start. First of all, slip lines and Lüders bands are observed in the austenitic phase. With the onset of cyclic softening, first of all martensite structures occur followed by micro-cracks in the austenite and enhanced martensite development after a thousand cycles. With the beginning of the secondary hardening (five thousand cycles), the increase of the martensite phase transformation is pronounced. Extrusions and intrusions occur and macro-crack propagation starts after ten thousand cycles combined with a

FIGURE 4. Cyclic Deformation Curve of Steel 1.4541 at Room Temperature (RT).
strong failure localization. The martensitic structure development and residual stress effects influence the magnetic permeability. The electrical conductivity is influenced by the changing dislocation density and arrangement. Both effects lead to changes in the GMR-impedance. In stress-controlled fatigue tests the eddy current-impedance measured by the GMR-sensor was found to be especially suitable to characterize the fatigue behavior. Fig. 5 is an example of a multiple step fatigue test with a load mix of different amplitudes and time dependencies. The impedance clearly shows an average continuous increase due to the martensite development. The impedance curve is modulated with a time function which follows the plastic strain amplitude exactly, an effect which is assumed to be induced by load and residual stresses. The GMR-technique provides the possibility of on-line, non-destructive characterization of the cyclic deformation behavior of the austenitic steel [9].

**Conclusion**

Electromagnetic procedures are suitable for nondestructive evaluation of material degradation by embrittlement caused by copper-precipitates and neutron irradiation. Electromagnetic measuring methods have the potential for in-field embrittlement measurements. This way, electromagnetic predictive maintenance of power plant components can inform the provider about the current state of embrittlement.

Two new NDE approaches for the characterization of fatigue have been presented, namely the thermo-damping approach and the GMR–eddy current approach. The thermo-damping approach based on dissipated heat evaluation is suitable for fatigue damage characterization of Ti alloys. The GMR–eddy current approach based on a GMR technique is suitable for fatigue characterization in steels. Both approaches have proved their reliability in laboratory fatigue tests. However, their applicability on technical components has still to be confirmed.
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REFERENCES


