Transactions, SMiRT-26
Berlin/Potsdam, Germany, July 10-15, 2022
Division VIII

MICROSTRUCTURE-BASED LIFETIME ASSESSMENT OF AUSTENITIC STEEL AISI 347 EXPOSED TO CORROSION AND FATIGUE

Ruth Acosta[1], Christian Boller[2], Klaus Heckmann[3], Jürgen Sievers[4], Tim Schopf[5], Tobias Bill[6], Peter Starke[7], Lukas Lücker[8], Kai Donnerbauer[9], Frank Walther[10]

[1] Research Assistant, LZfPQ, Saarland University, Saarbrücken, Germany (ruth.acosta@uni-saarland.de)
[2] Professor, LZfPQ, Saarland University, Saarbrücken, Germany
[3] Technical Expert, GRS, Cologne, Germany
[4] Chief Expert, GRS, Cologne, Germany
[5] Research Assistant, MPA, University of Stuttgart, Stuttgart, Germany
[6] Research Assistant, WWHK, University of Applied Sciences Kaiserslautern, Kaiserslautern, Germany
[7] Professor, WWHK, University of Applied Sciences Kaiserslautern, Kaiserslautern, Germany
[8,9] Research Assistant, WPT, TU Dortmund University, Dortmund, Germany
[10] Professor, WPT, TU Dortmund University, Dortmund, Germany

ABSTRACT

The austenitic steel AISI 347 (1.4550, X6CrNiNb18-10) is a typical material in the field of nuclear engineering, especially in pressurized pipelines of water-cooled reactors in Germany. The mechanical and thermal stresses due to operational conditions together with corrosive influences lead to aging and degradation of the material, which is associated with microstructural changes. The effects of such influences are currently covered by safety factors in the design of components. Additional qualified information about the microstructural state obtained through monitoring could improve the assessment of an aging condition, contribute to the quantification of safety margins, and possibly considerably delay the replacement of affected components while maintaining the same level of safety. In the joint research project "Microstructure-based assessment of maximum service duration of nuclear materials and components exposed to corrosion and fatigue (MibaLeB)", methods and tools which are intended to enable an improved assessment of the residual service life of metallic components have been developed. These investigations were mainly carried out analyzing the cyclic loading behavior of unnotched and notched specimens tested at different temperatures and medium conditions, based on the mechanical stress-strain hysteresis measurements and additional NDT parameters to monitor the material behavior under cyclic loading, the development of the short time evaluation procedure StrainLife for the determination of S-N curves and its integration in the simulation program PROST (PRObabilistic STructural mechanics), which is a tool for the integrity evaluation of passive components and for the analysis of their structural reliability.

INTRODUCTION

In this paper, an overview of the work performed in the research project "Microstructure-based assessment of maximum service duration of nuclear materials and components exposed to corrosion and fatigue (MibaLeB)", funded by the German Federal Ministry of Economic Affairs and Energy (BMWi) is presented. This project is a collaborative initiative between the five partners: Gesellschaft für Anlagen- und Reaktorsicherheit (GRS), LZfPQ at Saarland University, MPA of Stuttgart University, WPT of TU Dortmund University and WWHK of University of Applied Sciences Kaiserslautern.

The motivation of this research comes from the reorientation of the energy policy on how to assess the effect of an operational change in nuclear power plants. According to the report published by the Alliance for Competence in Nuclear Technology, Kompetenzverbund Kerntechnik (2013), a reliable assessment of power plant conditions as well as the prognosis of the expected operational and age-related changes on their safety conditions is needed. This can be achieved with the further development of validated analysis tools for the simulation of the structural-mechanical behavior of pressurized components and infrastructure, which include validated material models for the description of the material behavior, as well as qualified, non-destructive testing (NDT) methods.

The main objective of the MibaLeB project is, therefore, the development of a methodology for the assessment of the degradation of materials used in power plants' piping, by means of the combination of the expertise of all the project partners involved. For this purpose, the austenitic stainless steel AISI 347 (1.4550, X6CrNiNb18-10), widely used for piping in German nuclear power plants, was selected. The initial characterization by means of microscopical investigations and hardness measurements of the material, was the first step. Then, specimens were manufactured and initially characterized by means of NDT methods and roughness measurements and were finally fatigue tested following an optimized fatigue lifetime methodology which combines the use of different NDT methods and short time evaluation procedures (STEPs) (i.e. Starke et al. (2006) and (2018)) with numerical simulation. This methodology, validated in the first part of the project for fatigue specimens manufactured from a bar material, is to be transferred and validated for a pressurized hot water pipe exposed to thermal transients in the current investigations.

Different constant amplitude tests (CAT) and load or strain increase tests (LIT or SIT) were performed under different loading, temperature, and environmental conditions. During these tests, as it is done traditionally, the load and the deformation were recorded. Moreover, the cyclic degradation of the specimens was monitored with different NDT methods, taken as a material response. The determination of the S-N curve of the material was not performed using the classical methods but following STEPs, where a complete S-N curve may be determined with as little as three tests only.

MATERIAL AND METHODS

The Niobium stabilized austenitic stainless steel AISI 347 (1.4550, X6CrNiNb18-10) was selected for these investigations. Round bars with a diameter of 30 mm and the chemical composition presented in Table 1 were used.

Table 1: Chemical composition of the investigated AISI 347 (1.4550, X6CrNiNb18-10) in wt.-%.

	C	Si	Mn	Ni	Cr	S	P	Nb	Nb/C
Min				9.000	17.000				10.000
Max	0.080	1.000	2.000	11.500	19.000	0.015	0.045	1.000	
Batch P-I	0.025	0.401	0.577	10.064	18.147	0.001	0.024	0.402	16.080
Batch P-II	0.051	0.409	0.714	9.121	17.165	0.003	0.029	0.547	10.730

The material obtained from two batches as mentioned in Table 1, was ordered from the same manufacturer to optimize comparability. However, their chemical compositions vary slightly as well as their grain sizes, presented in Table 2. This causes a difference in metastability of the material, which does affect the α'-martensite transformation rate and the α'-martensite volume fraction achieved at the end of the test when tested under the same conditions.

Metallographic investigations of the cross section of the bars revealed a squared stirring mark in the center of the bar (Figure 1 a)), which is a trace of the manufacturing process of the bars having been hot rolled from a casted squared bar to a round bar finally. After a heat treatment in a furnace at a temperature of 1060 °C for 30 minutes and water quenching, the mark remained in the cross section. In general, the grain size of the P-II material is smaller than the grain size of the material from P-I, which is also reflected on the hardness measurements on both cross sections with values given in Table 2. Figure 1 a) shows a light microscopy image of the cross section of the bar after this was heat treated for 30 minutes at 1060° C and quenched in water. Figure 1 b) shows a high magnification EBSD from the center of the bar material, it is shown that the center of the bar is composed of an inhomogeneous multiphase structure (Austenite γ and α'-martensite). The edges of the rod are composed of a deformed and fine-grained austenitic structure.

Table 2: Average grain diameter and hardness of the investigated AISI 347 (1.4550, X6CrNiNb18-10).

Section	**Grain diameter [µm]**		**Hardness measurements [HV10]**	
	P-I	**P-II**	**P-I**	**P-II**
Center	14.18	10.05	147	176
Intermediate	14.18	10.05	152	181
Edges	10.05	7.09	188	195

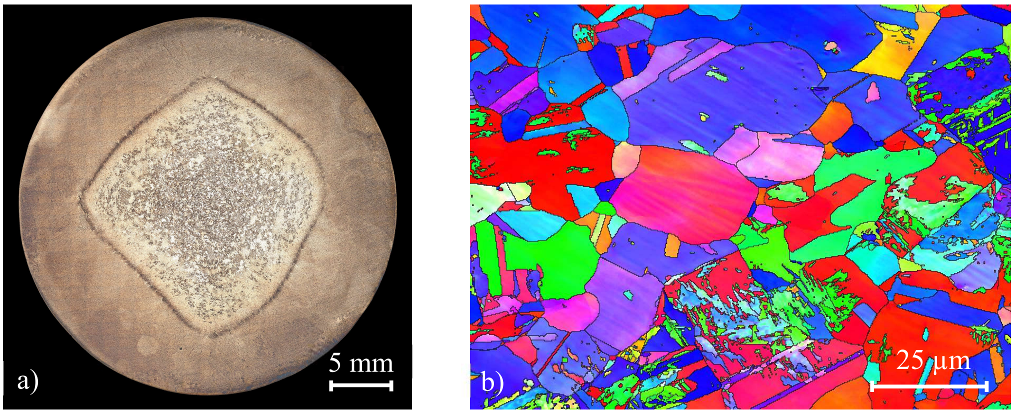

Figure 1. a) Light microscopic image of the AISI 347 (1.4550, X6CrNiNb18-10) after heat treatment and b) High magnification EBSD from the center of the bar material.

Fatigue tests were performed on unnotched and notched specimens. The specimens used for fatigue tests are presented in Figure 2. Figure 2 a) shows the unnotched specimen's geometry, whereas Figure 2 b) shows the two notched specimen's geometries, with a notch radius of R_N=0.35 and R_N=0.50 mm respectively.

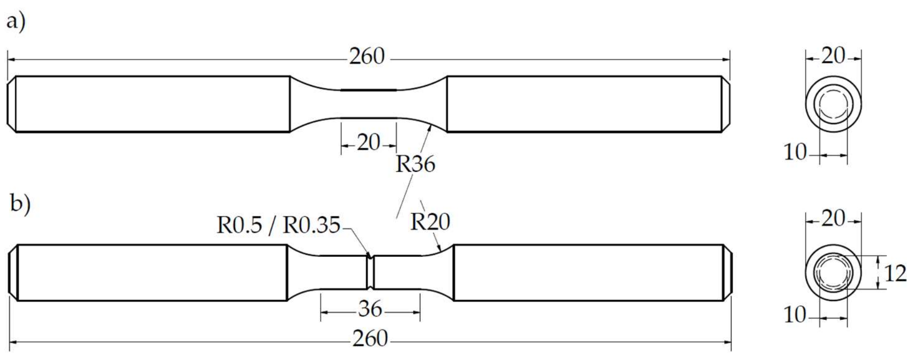

Figure 2. a) Unnotched specimen's geometry and b) Notched specimen's geometries.
All measurements are in mm.

All fatigue tests on unnotched specimens were strain controlled, with a constant total strain rate of $\dot{\varepsilon}_{a,t}$ = 4 × 10^{-3} s^{-1} and all fatigue tests performed on notched specimens were stress controlled tests with a constant nominal stress rate of $\dot{S}$ = 800 MPa·s^{-1}.

MATERIAL'S DEGRADATION ASSESSMENT USING NDT

It is well known that the description, measurement and quantification of damage in metals is a very complex subject. Palmgren (1924) was the first to introduce the concept of linear summation of fatigue damage and Miner (1945) was the first to express this concept mathematically, based on the number of applied cycles at a specific load level and the number of cycles to failure for that specific level. Since then, numerous damage models and theories have been developed over the years (See Fatemi et al. (1998) and Hectors et al. (2021)), but none of them has gained enough acceptance to replace the classic Palmgren-Miner damage rule in fatigue design of engineering structures. Those models could be related to a 'unique' material response to cyclic loading, recorded with possibly different NDT methods, giving the development of more reliable methods. The questions are: To which extent can those degradation mechanisms be detected with NDT methods? Which of the available methods can best describe the degradation of the material analyzed? The first step here is to understand, how the material will degrade when it is cyclically loaded. Table 3 shows how the AISI 347 (1.4550, X6CrNiNb18-10) is expected to respond to cyclic loading in the conditions to be analyzed. The information presented there is based on the work by Krupp et al. (2008), Smaga et al. (2006, 2019), and the experiments performed with the Batch P-I material.

Table 3: Degradation mechanisms of AISI 347 (1.4550, X6CrNiNb18-10) due to cyclic loading at different temperatures and fatigue regimes.

<table>
<tr><th></th><th>LCF</th><th>HCF</th></tr>
<tr><td rowspan="5">RT</td><td colspan="2">The paramagnetic γ-austenite can transform into ferromagnetic α'-martensite. Causing:
Reduction ← Fatigue life of the specimen → *Increase*</td></tr>
<tr><td>Martensite is expected to act as preferential crack initiation site.</td><td>Nucleation of very fine martensite particles in the areas of local plasticity hinder the dislocation movement.</td></tr>
<tr><td colspan="2">Incubation period with no α'-martensite transformation followed by a continuous increase in the α'-martensite volume fraction, leading to cyclic hardening.</td></tr>
<tr><td colspan="2">*Shorter* ← Incubation period → *Longer*</td></tr>
<tr><td colspan="2">*Larger* ← Volume fraction at the end of the test → *Smaller*</td></tr>
<tr><td>T > 200 °C</td><td colspan="2">No α'-martensite transformation occurs in the LCF and HCF regimes. Furthermore, the cyclic deformation behavior is characterized by initial cyclic hardening and depending on the applied strain amplitude, a subsequent cyclic softening might occur.</td></tr>
</table>

Based on that, it becomes evident that electromagnetic based measurements are an attractive option to detect the α'-martensite transformation due to plastic deformation at room temperature (RT). In the following, some examples of the NDT methods applied will be shown:

Tangential Magnetic Field and Eddy Current Measurements

Changes in the tangential magnetic field have been monitored using a self-developed magnetic field sensor, which consists of a permanent ring magnet, which magnetizes the specimen in the longitudinal direction, a Hall element positioned perpendicular to the specimen and thermocouples to compensate variations due to temperature changes. Figure 3 a) shows the evolution of the change in the tangential magnetic field for three strain-controlled CATs with total strain amplitudes $\varepsilon_{a,t}$ of 0.25%, 0.5% and 0.7%. It can be well seen that the period where no deformation induced α'-martensite takes place becomes longer the lower $\varepsilon_{a,t}$ is, whereas the volume fraction at the end of the test is smaller, understanding that a higher change in the tangential magnetic field corresponds to a larger α'-martensite volume fraction.

Additionally, eddy current measurements have been applied. This method is only feasible in conductive materials and consists of the excitation of a coil-probe with an alternating current, which produces a primary field. When the probe is near a conductive material, eddy currents are induced in the material, which in turn induce a secondary magnetic field that can be monitored by measuring the probe's electrical impedance. Therefore, the changes of the signal in the impedance plane reflect changes in the electromagnetic properties of the material. In this investigation, a multifrequency eddy current excitation of 500, 250, 100 and 50 kHz has been used. This has the advantage, that different penetration depths can be achieved with the same set up. Figure 3 b) shows the change in the phase for the 4 different frequencies in a strain increase test with initial total strain amplitude $\varepsilon_{a,t,start}$ = 0.025%, increased in steps of $\Delta\varepsilon_{a,t}$ = 0.025% and Δt = 900 s until $\varepsilon_{a,t}$=0.20% and then in steps of $\Delta\varepsilon_{a,t}$ = 0.05% and Δt = 1800 s until specimen's failure. A deeper penetration depth achieved with 50 kHz results in an earlier detection of the α'-martensite. As eddy current is known to be a reference NDT method, these changes in the phase could be related to α'-martensite volume fraction using reference standards.

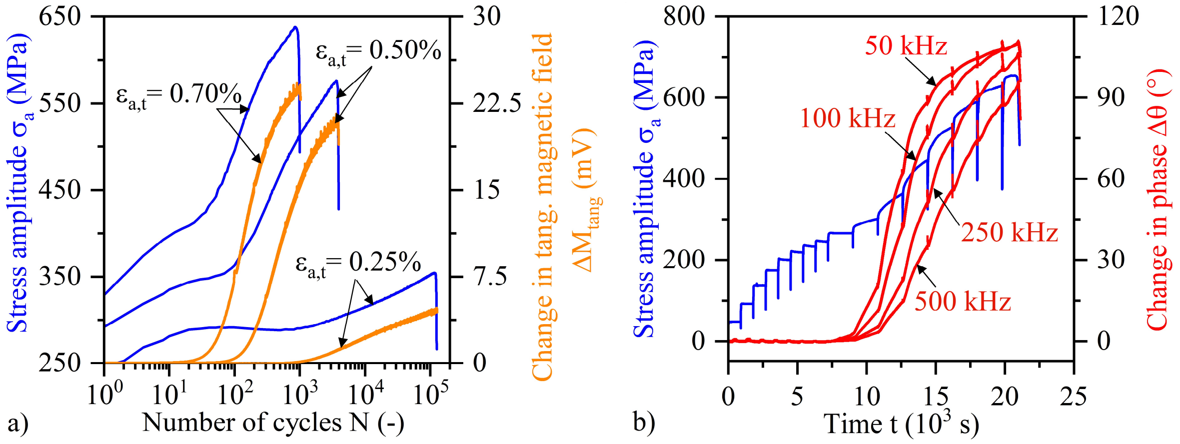

Figure 3. a) Change in tangential magnetic field ΔM_{tang} results of three CATs at RT with $\varepsilon_{a,t}$ = 0.25%, 0.50% and 0.70% and b) Change in Phase $\Delta\Theta$ results of a SIT using 4 excitation frequencies.

Electrical Resistance

The resistivity ρ of a material can be used to characterize imperfections in metallic conductors, given that it is inversely proportional to its mean free path of electrons. According to Kittel (2005), the net electrical resistivity ρ can be expressed using Matthiesen's rule as the sum of $\rho_L(T)$ and ρ_i, where $\rho_L(T)$ is the resistivity caused by the thermal phonons and ρ_i the resistivity caused by scattering of electron waves by imperfections like chemical impurities, vacancies, and dislocations, which disturb the periodicity of the lattice structure. Therefore, ρ_i is generally not dependent on temperature, but on the imperfections present in the specimens of a material. On the other hand, $\rho_L(T)$ should have the same value for various specimens of a material and change its value with temperature. During the fatigue tests, changes in the geometry of the gauge length can be neglected and the change in electrical resistance ΔR can be used to characterize fatigue damage instead of ρ.

In Figure 4, the evolution of the change in electrical resistance in shown for three strain-controlled CATs with total strain amplitudes $\varepsilon_{a,t}$ of 0.25%, 0.5% and 0.7%. Contrary to the change in tangential magnetic field for the same tests (Figure 3 a)), the electrical resistance changes linearly with a not steep slope since the beginning of the test, showing to be more sensitive to degradation mechanisms happening before the α'-martensite transformation starts. When the α'-martensite transformation starts, there is a change in the slope, which becomes steeper. At the end of the test, an exponential increase in the electrical resistance indicates the macrocrack propagation and failure of the specimens.

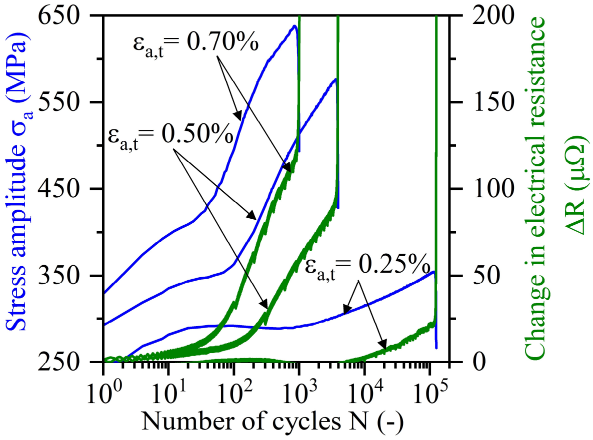

Figure 4. Change in electrical resistance ΔR results of three CATs at RT with $\varepsilon_{a,t}$ = 0.25%, 0.50% and 0.70%.

Temperature Measurements

According to Piotrowski et al. (1995), irreversible plastic deformation work during cyclic loading is converted approximately 90 to 95% into heat, therefore temperature measurements have been widely used to characterize fatigue damage. Temperature measurements were performed using an IR camera. Three areas were recorded, one in the center of the gauge length T_1 and two at the shafts as reference T_2 and T_3. The change in temperature ΔT is calculated by subtracting the average of T_2 and T_3 to T_1.

Figure 5 shows the results of temperature measurements for three strain-controlled CATs with total strain amplitudes $\varepsilon_{a,t}$ of 0.25%, 0.5% and 0.7%. It is observed that the temperature increases from the beginning of the tests in the incubation period, changing its trend to a decrease, which indicates the hardening of the material due to α'-martensite transformation. Before specimens' failure, there is an increase in the temperature, which is related to macrocrack propagation.

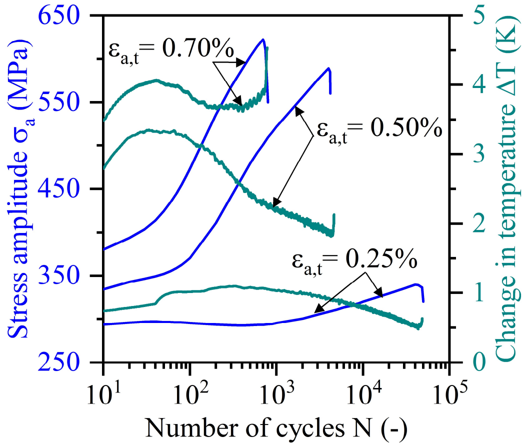

Figure 5. Change in temperature ΔT results of three CATs at RT with $\varepsilon_{a,t}$ = 0.25%, 0.50% and 0.70%.

SHORT TIME EVALUATION PROCEDURE: STRAINLIFE

STEPs for the determination of S-N curves of metallic materials based on NDT were introduced by Starke et al. (2006) with the PHYBAL approach, which allows the determination of a complete S-N curve with 3 fatigue tests only. This approach has been improved and extended to specific boundary conditions, like for example with the SteBLife method, developed by Starke et al. (2018), which is able to determine a trend S-N curve with one fatigue test only.

Within the frame of the MibaLeB project, the StrainLife approach was developed. The schematic representation of the method is shown in Figure 6. One SIT and two CATs need to be conducted, recording not only the stress, total strain and plastic strain amplitudes, but also the material response using any of the NDT methods previously described. Using the results of the SIT, the total strain amplitude is plotted in a double logarithmic scale vs. the material response taken at the middle of each step and this relation can be divided into a mostly elastic and a mostly plastic portion, from which coefficients $n_e{}'$ and $n_p{}'$ are determined, that characterize this relationship. These coefficients can then be used in the empirical equations developed by Morrow (1965), to determine the slopes b and c of the $\varepsilon_{a,e} - N$ and $\varepsilon_{a,p} - N$ relationships, respectively. Using these slopes b and c, the elastic and plastic strain amplitudes, as well as the number of cycles to failure determined from the fatigue lives of the two CATs, performed at different total strain amplitudes, the coefficients B and C of the Basquin and Manson-Coffin relationships can be determined and the complete ε-N curve can be evaluated as to Equation (1).

$$\varepsilon_{a,t} = B \cdot (2N_f)^b + C \cdot (2N_f)^c \tag{1}$$

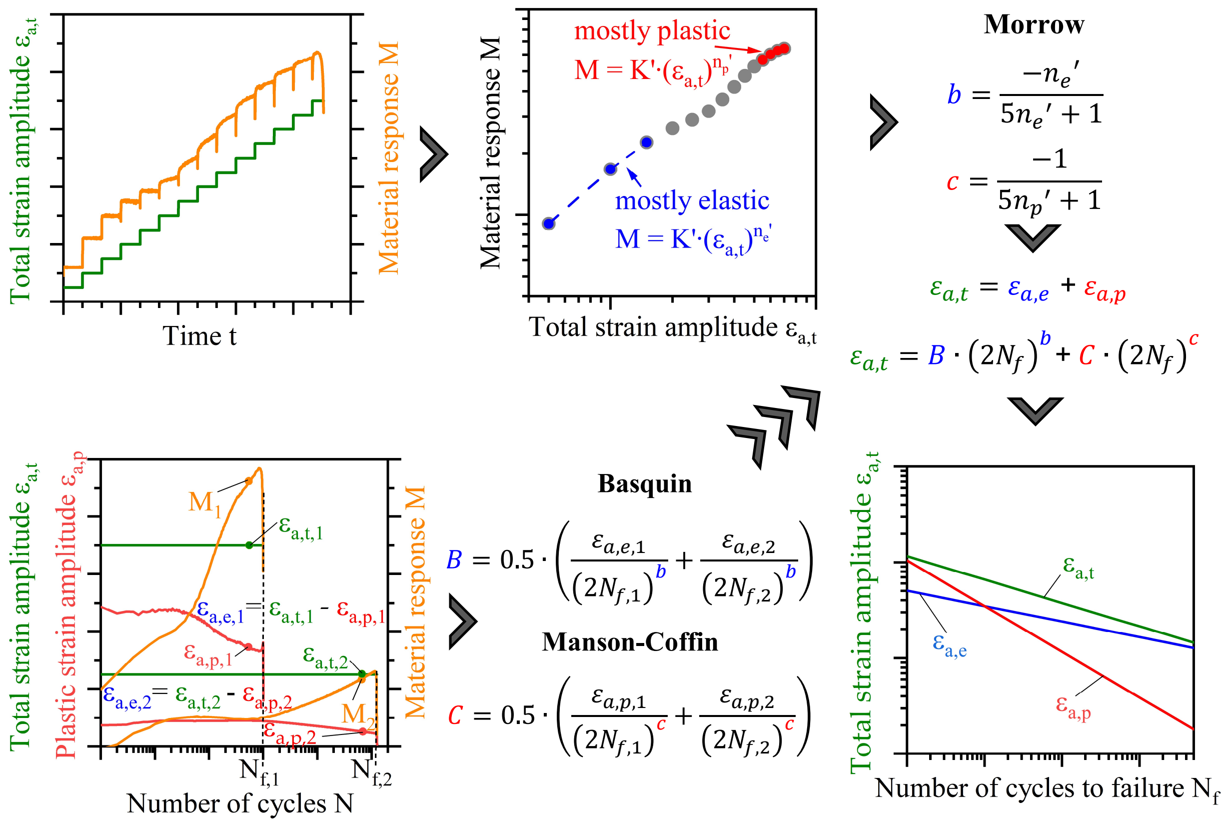

Figure 6. Schematic representation of the StrainLife method for the determination of a complete ε-N curve.

TRANSFER OF MATERIALS DATA INTO NUMERICAL MODELS

In order to assess the integrity of structural components, an appropriate description of the materials behavior based on mechanical models has to be made. Using the materials data obtained from fatigue tests on unnotched specimens, the parameters of the three component Chaboche plasticity model were determined and validated on notched specimens. This model describes the nonlinear kinematic hardening of a material. Moreover, FEM simulations were carried out to determine the stress and strain in the notch root of the notched components, given that this stress and strain cannot be directly determined from experiments. Details of this transfer can be obtained from Heckmann et al. (2022).

TRANSFER OF METHODOLOGY TO COMPONENTS

Summarizing what has been presented above, these are three major conclusions to be drawn from the MibaLeB project so far for the development of a methodology for integral assessment of aging structures:

- NDT methods provide additional useful information beyond stress and strain to describe aging phenomena in metallic materials. The degradation mechanisms observed go beyond a visible crack and do include mechanisms such as martensitic transformation, possibly even dislocation movements, persistent slip bands' formation and possibly more. It is important to identify to which extent the NDT methods are sensitive to those degradation mechanisms.
- The generation of S-N data can be made in a fast but reliable way using the different STEPs.
- Reliable numerical models that describe a component's behavior under different loading and environmental conditions are crucial to understand what is ongoing in locations, that cannot be accessed experimentally in a reasonable way, but which may have to be considered from a safety criticality point of view. The NDT methods considered may then again benefit from those numerical models in a way, that the results of the models can provide guidance on how to optimize the monitoring of the structures to be assessed.

The methodologies presented have been validated on simple notched fatigue specimens so far only. However, current work ongoing in MibaLeB Phase II is expanding this validation to the case of a pressurized hot water pipe made of AISI 347 (1.4550, X6CrNiNb18-10) and exposed to a series of pressurized hot water flow conditions, which change the material's condition, without having a crack to be seen. This will include aspects of fluid-structure interaction (FSI), pressurization and thermal transients, all being linked to NDT methods where feasible. The big challenges that arise are as follows:

- Which microstructural phenomena will happen on the component as it degrades and to which extent can those phenomena be detected with NDT methods? Is it possible to use in-situ monitoring to detect all these degradation mechanisms and to predict the remaining lifetime?
- Can a microstructural configuration be associated with the degree of degradation of a material or component? For each microstructural configuration, only one S-N curve is valid and therefore the determination of S-N data for aged structures is difficult, since the material to be made available for analysis is extremely limited. In that regard STEPs could be of essential help.
- To which degree can material's data bases obtained using STEPs and NDT be used to develop reliable numerical simulations of components, whose loading condition become way more complex than a simple uniaxial fatigue specimen.

The approach that will be followed to overcome those challenges is basically the generation of a damage matrix based on different NDT parameters and on various fatigue tests performed under different conditions, analyzing the microstructure of the specimens after certain intervals, and correlating those microstructural changes to the different NDT parameters. Afterwards, the generated data could be analyzed using neural networks, in view of recognizing degradation patterns. It will be finally evaluated, to which extent the measured signals compare to the damage matrix and how this all matches with the pipe component tests.

FUNDING

This research was funded by the German Federal Ministry of Economics Affairs and Climate Action (BMWK), within the framework of the joint project "Microstructure-based determination of the maximum service life for corrosion fatigue loaded materials and components of nuclear technology", part I, funded grant numbers 1501528A, 1501528B, 1501528C and RS1545 and Part II, funded grant numbers 1501610A, 1501610B, 1501610C, 1501610D and RS1594E respectively.

REFERENCES

Fatemi, A.; Yang, L. (1998). "Cumulative fatigue damage and life prediction theories: A survey of the state of the art for homogeneous materials," *International Journal of Fatigue*, 20, pp. 9-34.

Heckmann, K., Sievers, J., Acosta, R., Boller, C., Schopf, T., Bill, T., Starke, T., Luecker, L., Donnerbauer, K., Walther, F. (2022). "Evaluation of the fatigue life of AISI 347 specimens with small notched based on local strain considerations," *26th International Conference on Structural Mechanics in Reactor Technology (SMiRT 26),* Berlin, Germany.

Hectors, K., De Waele, W. (2021). "Cumulative Damage and Life Prediction Models for High-Cycle Fatigue of Metals: A Review," *Metals*, 11, 204, DOI: 10.3390/met11020204.

Kittel, C. (2005), *Introduction to solid state physics*, 8th ed., John Wiley and Sons, New York.

Kompetenzverbund Kerntechnik (2013). *Nukleare Sicherheitsforschung, Neuorientierung an aktuellen energiepolitischen Rahmenbedingungen,* Germany.

Krupp, U., West, C. and Christ, H. J. (2008). "Deformation-induced martensite formation during cyclic deformation of metastable austenitic steel: Influence of temperature and carbon content," *Materials Science and Engineering: A,* 481-482, pp. 713-717, DOI: 10.1016/j.msea.2006.12.211.

Miner, M.A., (1945). "Cumulative damage in fatigue," *Applied Mechanics Transactions (ASME),* 12, pp. 159-164.

Morrow, J.D. (1965). "Cyclic plastic strain energy and fatigue of metals," In *Internal Friction, Damping, and Cyclic Plasticity*; ASTM International: West Conshohocken, PA, USA, pp. 45–87, ISBN 978-0-8031-5995-2.

Palmgren, A.G. (1924). "Die Lebensdauer von Kugellagern (Life Length of Roller Bearings or Durability of Ball Bearings)," *Zeitschrift des Vereines Deutscher Ingenieure (ZVDI),* 68(14), pp. 339-341.

Piotrowski, A., Eifler, D. (1995). "Bewertung zyklischer Verformungsvorgänge metallischer Werkstoffe mit Hilfe mechanischer, thermometrischer und elektrischer Meßverfahren," *Materialwissenschaft und Werkstofftechnik*, 26, pp. 121–127, DOI:10.1002/mawe.19950260305.

Smaga, M., Walther, F., Eifler, D. (2006). "Investigation and modelling of the plasticity-induced martensite formation in metastable austenites," *International Journal of Materials Research*, 97 (12), pp. 1648-1655, DOI: 10.3139/146.101396

Smaga, M., Boemke, A., Daniel, T., Skorupski, R., Sorich, A. and Beck, T. (2019). "Fatigue behavior of metastable austenitic stainless steels in LCF, HCF and VHCF regimes at ambient and elevated temperatures," *Metals,* 9, 704, DOI: 10.3390/met9060704.

Starke, P., Walther, F. and Eifler, D. (2006). "PHYBAL-A new method for lifetime prediction based on strain, temperature and electrical measurements," *International Journal of Fatigue*, 28, 9, pp. 1028-1036, DOI: 10.1016/j.ijfatigue.2005.07.050.

Starke, P., Bäumchen, A. and Wu, H. (2018). "SteBLife – A new short-time procedure for the calculation of S-N curves and failure probabilities," *Materials Testing*, 60, 2, pp. 121-127, DOI: 10.3139/120.111139.