



# DELISA<sup>LTO</sup>

DEscription of the extended Lifetime and its influence on the Safety operation and construction materials performance – Long Term Operation with no compromises in the safety



## Report on applicability of ultrasonic measurement for the evaluation of phase changes caused by thermal ageing

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## About DELISA-LTO

The aim of the project is to determine the most affected and threatened components from the point of view of the long-term operation (LTO) and describe the effect of the LTO on the material properties as well as develop a simulation tool able to predict the non-acceptable state of the material. The project is specifically focused on the Water-Water Energetic Reactor (VVER), nevertheless, the approach is to maintain the easy transferability to other light water reactor technologies, as well.

The outputs of the project will lead to the increase of operational safety at the extended lifetime due to the in-time prediction of the potential failure. The basic approach of the project is to combine the development of the simulation tools, experimental work (material analyses), in-service and/or non-destructive inspection techniques to develop the effective “early warning” tool for the assessment of the system integrity for the LTO of the current LWR with a specific focus on the VVER technology.

The project's aim specifically focuses on the thermal aging and swelling of the loaded constructional materials. One of the most affected components from the LTO's point of view are the heat exchanging tubes of steam generators (thermal ageing) and reactor internals (swelling). The experimental material was screened and selected with the main criteria: to support and validate the proposed methods in the most accurate way, and to be “on stock” and available at the expected start of the project, the latest. The experiments are planned to be performed at the available material with clear and well-described operational history as well as the material from the original batch in the “as-received” state to gain the most relevant and valuable information with high impact to the community.

## Project information

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## 1. Background

Within the DELISA-LTO project, the possibilities of using non-destructive testing methods to determine the long-term thermal impact on material properties are verified. Samples of artificially aged material by heat treatment as well as samples taken from components operated at power plants are available for testing.

## 2. Introduction

NDT methods selected for testing and verifying any microstructure changes include ultrasonic testing using the Pulse-echo (UT) technique, eddy current (ET) testing method and magnetic material memory (MMM).

The ultrasonic method is used from two perspectives. One of them is the inspection of the material for the presence of internal defects, including the verification of the patency of the material for ultrasonic waves realized by measuring attenuation in selected locations. These measurements will be verified by metallographic evaluation of the microstructure of the material. The second approach of UT is to determine the properties of a material by measuring the propagation velocities of longitudinal and transverse waves to calculate the modulus of elasticity  $E$  of the material. The  $E$  value determined by the Pulse-echo (PEUT) technique can then be verified by mechanical tests.

Microstructural methods of Light Optical Microscopy (LOM) and Scanning Electron Microscopy (SEM) were used for the validation of NDT methods. The methodology of specimen preparation and microstructural evaluation was optimized on the as-received base metal (BM) 08CH18N10T and 10GN2MFA materials to describe the microstructural changes in specimens after artificial heat treatment and also on specimens aged on-site. The time and temperature to which the material has been exposed has a major influence on the change in material properties.

## 3. Ultrasonic measurement of material properties for characterization of thermal aging

### Description of the measurement and evaluation methodology

Measurement of repeatedly evaluable material properties is carried out by ultrasonic testing using the Pulse-Echo technique. Direct probes of longitudinal and transverse waves are used for measurements. Ultrasonic probes are used to measure the speed of ultrasound propagation in each material. The speed of ultrasound propagation is an essential property from the point of view of non-destructive material testing and its change indicates a possible change in the structure. The ultrasonic speed measurement is performed at a calibration point where the sample thickness determined by mechanical measurement using a digital calliper is known. On each sample one calibration point is determined.

Part of the ultrasonic testing of samples is the measurement of attenuation by both types of probes. All measurements are carried out in selected locations marked with numbers according to the type of material. The attenuation values obtained at individual points are averaged for the given sample and the values of the samples of the given material type are compared with each other.

Both types of measurements are carried out before and after thermal aging at the same measuring points. The obtained longitudinal and transverse velocity values ( $V_L$ ,  $V_T$ ), together with the generally known material density values ( $\rho$ ), are used to calculate the Poisson number ( $\nu$ ), Young's modulus of elasticity ( $E$ ) and shear modulus ( $G$ ) according to the equations below.

$$\begin{aligned}\text{Young's Modulus } E &= \rho \cdot V_T^2 \cdot (3V_L^2 - 4V_T^2) / \\ &\quad (V_L^2 - V_T^2) \\ \text{Shear Modulus } G &= \rho \cdot V_T^2 \\ \text{Bulk Modulus } B &= \rho \cdot (V_L^2 - 4 \cdot (V_T^2) / 3) \\ \text{Poisson's Ratio } \nu &= (V_L^2 - 2 \cdot V_T^2) / \\ &\quad (2 \cdot V_L^2 - 2 \cdot V_T^2)\end{aligned}$$

### Equipment required

Ultrasonic device e. g., Epoch 650 for PEUT.

Direct probe (longitudinal wave) – frequency 10 MHz, transducer diameter 10 mm.

Direct probe (transversal wave) – frequency 20 MHz, transducer diam. 6,35 mm.

Coupling medium – GE gel.

### Scope of tests

Ultrasonic measurement results for relevant materials in the initial state and after artificial thermal ageing, as well as available materials with known operational exposure, will be correlated with other material characteristics determined by destructive tests: tensile tests on micro samples.

References: [1], [2], [3] and [4]

## 4. Validation of NDT by microscopy methods

The specimens were cut in longitudinal and transverse direction using the SiC circular saw, then prepared by mechanical grinding and polishing with a diamond paste of particle size up to 1  $\mu\text{m}$ . The surface was chemically etched with the Kallings acid mixture used for austenitic stainless steels.

The microstructural assessment was firstly carried out by LOM. The average grain size and the average size of the particles were calculated. The LOM technique is sufficient to evaluate the average grain size and to evaluate larger particles (micrometre size such as TiC carbides). Austenitic grains were evaluated using the ImageJ macro – microphotographic mark measurement, their approximation by central circles and determination of diameter according to the standard ASTM 112. The average size was defined as the median and the corresponding standard deviation was calculated from a larger number of measurements (in the order of tens). [5]

Chemical analysis of particles present in the microstructure was performed by Energy Dispersive X-ray Spectroscopy (EDS) within SEM to determine the particle type (carbide, carbonitride, sulphide, etc.). To obtain the chemical contrast of particles/matrix and complete the LOM observations, the Backscatter Electron (BSE) imaging was used.

## 5. Validation of NDT by mechanical tests

The modulus of elasticity from mechanical testing can be determined according to Hooke's law from the stress-strain dependence. The stresses, between which the modulus of elasticity was determined, shall be stated, provided that the permanent elongation shall not exceed 0,003 % of the measured length. The modulus of elasticity can be obtained by measuring the extension with a sufficiently sensitive extensometer in two ways:

1. from the engineering stress-strain diagram test as a slope of the initial straight-line part of the diagram
2. from the stress-strain diagram under gradual loading and relief

Both methods are fraught with different errors resulting from the detection method. A variance of 2% of the results on the same sample is considered acceptable. The values of the modulus of elasticity calculated from the total deformations are 3-4% higher than the values calculated from the elastic deformations only [6].

The loading approach can be tensile, compression or 3-point bend test. The optimal approach of testing and evaluating elastic modulus will be chosen with respect to available amount of testing material.

## References

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## 6. Conclusions

The above-described approach of UT testing with measurement of individual parameters and their destructive verification by mechanical tensile tests and metallographic evaluation of the microstructure will be applied to all samples of selected materials artificially and/or naturally aged.