# Thermal Aging Evaluation of Mod. 9Cr-1Mo Steel using Nonlinear Rayleigh Waves

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## 1. Introduction

Modified 9Cr-1Mo ferritic-martensitic steel has been developed in 1980s for applications in sodium-cooled fast reactors (SFR) [1]. SFR is operated in high temperature above 500°C. Higher operating conditions require a careful consideration of the thermal aging in the components and piping of SFR. Thermal aging can pose a high risk to decreases in the mechanical properties such as strength or creep resistance [2]. This can lead to an unexpected failure during long term operation. Nonlinear NDE techniques are preferred over conventional NDE techniques (linear ultrasonic measurements) because nonlinear ultrasonic techniques have shown their capability to detect a microstructural damage in the structures undergoing fatigue and creep [3-8]. These nonlinear ultrasonic techniques make use of the fact that the dislocation density increases, which will create a nonlinear distortion of an ultrasonic wave; this damage causes the generation of measurable higher harmonic components in an initially mono-chromatic ultrasonic signal. This study investigates the recently developed non-contact nonlinear ultrasonic technique to detect the microstructural damage of mod. 9Cr-1Mo steel based on nonlinear Rayleigh wave with varying propagation distances.

## 2. Nonlinear Ultrasonic Technique

An ultrasonic wave propagating through an elastic solid generates higher harmonics due to the nonlinear distortion of the wave by the material nonlinearity. The nonlinearity of the material can be assessed by measuring the amplitude of these higher harmonics. The non-dimensional acoustic nonlinearity parameter  $\beta$  relates the amplitudes of the fundamental and second harmonic waves and gives therefore information about the nonlinearity of the stress-strain relation of the solid. For an excitation with a plane time-harmonic longitudinal wave of amplitude *A* and frequency  $\omega$ , having a displacement field of form

$$u(x,t) = A\cos(kx - \omega t), \qquad (1)$$

the acoustic nonlinearity parameter  $\beta$  can be obtained in terms of the amplitudes of the fundamental  $(A_1)$  and second harmonic  $(A_2)$  waves as

$$\beta = \frac{A_2}{A_1^2} \frac{8C_L^2}{\omega^2 x_1}.$$
 (2)

The acoustic nonlinearity parameter can also be derived for Rayleigh surface waves as shown by Herrmann *et al.* [9] with the dependency on the amplitudes of the first and second harmonic waves and the propagation distance x. Rayleigh surface waves have certain advantages over the longitudinal waves [9, 10]. Rayleigh waves have longer propagation distance due to the energy concentration near the surface and are very sensitive to near-surface microstructural changes. Rayleigh waves are generated and detected on the same side of a component.

#### 3. Experiments and Results

## 3.1. Specimen preparation

Mod. 9Cr-1Mo steel was in plate form with a thickness of 12.7 mm. It was normalized at 1050°C and tempered at 770°C in air. The dimensions of the specimens are 400 mm  $\times$  45 mm  $\times$  12.5 mm (length  $\times$  width  $\times$  height). Smooth and parallel surfaces are obtained by surface grinding the two opposite sides of the specimen where the Rayleigh wave measurements are performed. Isothermally heat treatment was performed at temperatures 650°C for various thermal aging times (0 h, 200 h, 500 h, 1000 h, 1500 h and 3000 h), as shown in Table 1.

Table 1. Heat treatment of specimen

| Specimen # | Temperature [°C ] | Time [h] |
|------------|-------------------|----------|
| 1          | 650               | 0        |
| 2          | 650               | 200      |
| 3          | 650               | 500      |
| 4          | 650               | 1000     |
| 5          | 650               | 1500     |
| 6          | 650               | 3000     |

#### 3.2. Nonlinear Rayleigh wave measurement

Figure 1 shows the experimental setup using a noncontact air-coupled receiver. A tone bust of 20 cycles with 2.1 MHz is used for the excitation of the transducer with nominal frequency of 2.25 MHz. The desired high voltage excitation signal for the transducer is obtained by amplifying the output signal of a function generator with the RITEC GA-2500A high power gated amplifier. The receiving air coupled transducer centered at 4 MHz receives the longitudinal wave in air, which is leaked from the Rayleigh wave. The obtained output signal is amplified by 40dB to improve the signal-tonoise ratio and the signal is recorded and averaged by an oscilloscope.



Fig. 1. Schematic diagram of experimental setup using noncontact air-coupled receiver.

The nonlinear Rayleigh wave measurement is performed by detecting the Rayleigh wave for increasing propagation distance. The exciting wedge is clamped to the specimen at one location for one set of measurements and measurements are taken over the propagation distance by positioning the air-coupled transducer at different spots along the propagation distance. In Figure 2, the steady state portion in the detected time domain signal is identified and a Hann window is applied to eliminate the ringing effects from the transducer. In order to obtain the electrical amplitudes of the fundamental  $A^{el}_1$  and second harmonic wave  $A^{el}_2$  components we need to map the windowed time domain signal in the frequency domain.



Fig. 2. (a) Time-domain signal; (b) Amplitudes of the first and second harmonics in the frequency-domain, obtained by performing a Fast Fourier transformation on the steady state portion of the time domain signal.

The nonlinearity parameter in equation 2 is proportional to the ratio  $A^{el}_2/A^{el}_1^2$ . However, this is only valid when the propagating wave is a plane wave, where there is no change in the wave front. In the three dimensional case diffraction and attenuation affect the propagating Rayleigh wave, but these effects can be neglected for small propagation distances and therefore a linear fitting of the obtained ratio  $A_2/A_1^2$  over propagation distance can be used as a proper model to obtain the nonlinear parameter, as shown in Figure 3.



Fig. 3. The ratio,  $A_2/A_1^2$ , as a function of propagation distance for the reference sample (specimen 1).



Fig. 4. Nonlinear parameter as function of holding time for 9Cr-1Mo steel.

Figure 4 shows the relative nonlinear parameter  $\beta'$ , normalized by the mean value of the untreated specimen, as function of holding time. The relative nonlinearity parameter is rapidly decreased in the initial thermal aging and then slightly increased after a holding time of 500 h. The decrease in  $\beta'$  can be qualitatively explained by the reduction of the dislocation density and the following increase can be explained with the growth of the precipitated particles.

## 3.3. Microstructure and hardness measurement

Figure 5 shows a sequence of optical micrographs (OM) at 1000X magnifications of modified 9Cr-1Mo steel samples treated at 650°C for (a) 0 h (b) 200 h, (c) 500 h, (d) 1000 h, (e) 1500 h and (f) 3000 h. All samples show large concentration of precipitated particles. Initially, particles seem to be uniformly distributed in the matrix and the boundary of the grains.

As aging time increases particles seem to concentrate on grain boundaries.

Figure 6 shows that Rockwell C hardness drops rapidly for 200 h and 500 h holding time and slowly decreases for holding times higher than 500 h. It is clear that the diffusion process generated by heat treatment is affecting the microstructure of the mod. 9Cr-1Mo steel; this process creates a loss of chromium and molybdenum in the matrix. The loss effect is reflected on the hardness of the mod. 9Cr-1Mo steel which decreases as aging time increases.



Fig. 5. OM micrographs at 1000X magnifications of modified 9Cr-1Mo steel specimens treated at 650 °C for (a) 0 h (b) 200 h, (c) 500 h, (d) 1000 h, (e) 1500 h and (f) 3000 h



Fig. 6. Rockwell hardness C as function of holding time for mod. 9Cr-1Mo steel.

## 4. Conclusions

Nonlinear Rayleigh surface wave measurements using a non-contact, air-coupled ultrasonic transducer have been applied for the thermal aging evaluation of modified 9Cr-1Mo ferritic-martensitic steel. Thermal aging for various heat treatment times of mod. 9Cr-1Mo steel specimens is performed to obtain the nucleation and growth of precipitated particles in specimens. The amplitudes of the first and second harmonics are measured along the propagation distance and the relative nonlinearity parameter is obtained from these amplitudes. The relative nonlinearity parameter shows a similar trend with the Rockwell C hardness. The results show the potential of nonlinear Rayleigh surface wave measurement technique for the assessment of thermal aging in mod. 9Cr-1Mo ferritic-martensitic steel.

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