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Combined thermo-electric power and resistivity measurements of embrittlement recovery in aged JRQ ferritic steel

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Abstract

The detection of microstructural changes by means of non-destructive techniques is a severe challenge. A significant amount of work has been carried out over the last 20 years to validate methods based on thermo-electric power and resistivity measurements.

It has been shown that both thermo-electric power (TEP) and resistivity measurements have the potential to provide information and assessment of microstructural changes such as precipitation of copper and matrix damage, whereas grain boundary segregation of particular species like phosphorus remains difficult to assess.

This paper investigates the possible advantages of combined TEP and resistivity measurements of embrittlement recovery due to annealing of an embrittled ferritic RPV steel. The ferritic steel chosen for its wide use as reference in several IAEA international programmes is JRQ, particularly sensitive to embrittlement.

Keywords: Reactor pressure vessel steel; Irradiation damage; Non-destructive techniques; Ageing monitoring; Thermo-electric power; Resistivity

1. Introduction

Detection of microstructural changes, caused by embrittlement and thermal treatments or service history of the component, can provide useful support to the safety and economy of plant. The results obtained in previous studies [1–8] demonstrate that non-destructive techniques based on the measurement of electrical properties can have interesting applications to non-destructive embrittlement assessment of reactor pressure vessel ferritic steels. The Institute for Energy of the Joint Research Centre (JRC-IE) has in particular developed two devices, focussed on the measurement of the electrical properties, which prove to give a good non-destructive assessment of the embrittlement state of ferritic steels.

As shown in [9,10], the first technique, called Seebeck and Thomson effects on aged material (STEAM), is based on the measurement of the relative Seebeck coefficient, characteristic of the material and related to the microstructural changes induced by embrittlement. With the same aim the second technique, named resistivity effects on aged material (REAM), measures the resistivity of the material.

The present paper focuses on the applicability of both techniques to the assessment of recovery by annealing of thermally embrittled JRQ steel. This is a ferritic steel used as reference material in many international round-robin projects supported by the International Atomic Energy Agency and the European Commission, which has proven to be particularly sensitive to embrittlement.

2. Experimental procedure

As described hereinafter, measurements of thermo-electric power and resistivity by means of the STEAM and REAM devices, respectively, have been performed on JRQ ferritic steel in the virgin and thermally embrittled/annealed conditions. Cross-comparison with hardness measurements was used to investigate possible correlations with hardening.

2.1. Measurements of the relative Seebeck coefficient (STEAM technique)

The STEAM technique measures the thermo-electric power generated by the so-called Seebeck effect. The basic principle is that the induction of thermoelectric power is caused by the

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Table 1
Composition of JRQ ferritic steel (mass%)

	C	Si	Mn	P	S	Cu	Ni	Cr	Mo	V	Al
JRQ	0.18	1.42	0.24	0.017	0.004	0.14	0.84	0.12	0.51	0.002	0.012

temperature-sensitive distribution of conduction electron velocity. Applying a temperature gradient across the material, equilibrium is reached only when a voltage difference of the order of a few millivolts per degree Celsius of imposed temperature gradient is established (Thomson effect). Therefore, under limiting conditions, the matrix damage and the microstructural changes caused by both thermal and neutron embrittlement can be correlated with electric potential measurements.

Moreover, since the conductor tested is also coupled with two other metals at different temperatures, a voltage difference is generated by the thermocouple effect at the interfaces (Peltier effect). The two combined phenomena form the Seebeck effect.

The applicability of STEAM to measurements following annealing treatments has been proved in comparison with other NDE techniques [1,2]. For a complete description of the STEAM device and the test set-up see [3].

References to previous studies of thermoelectric power effects in materials and their application to non-destructive evaluation of metals can be found in [4–8].

2.2. Measurements of resistivity—REAM technique

JRC-IE has also developed a prototype device named resistivity effects on aged material (REAM) for the measurement of a material's resistivity. The layout is similar to that of STEAM, the driving force imposed being a current (1 A) instead of the temperature gradient imposed in STEAM.

The technique can be applied to Charpy or even mini-Charpy size specimens. Due to the limited size of the specimens tested, the property measured is only proportional to the absolute resistivity (the ASTM standard foresees a minimum length of 300 mm). The application is therefore for monitoring relative changes of resistivity between virgin and aged materials, rather than for measuring absolute values. Details of the experimental set-up are described in [9,10].

2.3. JRQ steel

The material tested is JRQ ferritic steel, an ASTM A533-B Cl.1 steel plate used as reference in many IAEA international projects for its high neutron embrittlement rate [11]. JRQ chemical composition in wt% is reported in Table 1.

The JRQ plate material was manufactured by Kawasaki Steel Co. The heat treatment followed was:

Normalisation 900 °C—air cooled
Quenching 880 °C—water quenched
Tempering 665 °C—12 h—air cooled
PHWT 620 °C—40 h—furnace cooled.

A JRQ plate was then thermally treated at JRC-IE to 900 °C for 1 h and water quenched.

Subsequently, from the embrittled steel plate 15 standard Charpy specimens ($10\times10\times55$ mm^3) were machined, and then annealed according to the following scheme:

(a) 400 °C for 1 h and cool-down in air
(b) 475 °C for 1 h and cool-down in air
(c) 575 °C for 1 h and cool-down in air
(d) 600 °C for 1 h and cool-down in air
(e) 650 °C for 1 h and cool-down in air
(f) 675 °C for 1 h and cool-down in air
(g) 715 °C for 1 h and cool-down in air
(h) 750 °C for 1 h and cool-down in air

This whole range of temperatures was chosen to span the potential range for recovery from the original embrittled state.

3. Results

Measurements of as-received JRQ (before thermal treatment to 900 °C for 1 h and water quenching) are reported in Table 2.

Subsequently, all different annealing levels have been characterized by hardness, resistivity and TEP measurements.

3.1. Results analysis: Pearson product moment correlation coefficient

The Pearson product moment correlation coefficient is defined as

$$r = \frac{\sum(X-\mu_x)(y-\mu_y)}{N\sigma_x\sigma_y}$$

where μ_x and μ_y are the mean values of the X and Y variables, and σ_x, σ_y are the standard deviations.

In our analyses, the Pearson correlation coefficient (omitting product moment which just clarifies its definition) was chosen because it is the most widely used measure of correlation or association. It is a number that can range from -1 (perfect

Table 2
JRQ: as received measurements

ρ (μΩ m)	S (μV/°C)	S/ρ (μA/°C m)	HV$_{10}$
0.277	3.96	14.296	198