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Use of large scale facilities for research in metallurgy

Stress relaxation through ageing heat treatment – a comparison between in situ and ex situ neutron diffraction techniques

Relaxation des contraintes lors de traitements thermiques – une comparaison des techniques in-situ et ex-situ de diffraction des neutrons

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ABSTRACT

The effect of ageing heat treatment on the relaxation of residual stress in a water quenched polycrystalline nickel-base superalloy has been measured using neutron diffraction. Two separate experiments have been conducted; the first experiment was an ex situ study in which samples were individually processed with varying degrees of age time before measurement. The second experiment was an in situ heat treatment, which required heating and then holding the sample at ageing temperature while measuring strain using neutron diffraction. The in situ experiment was carried out twice using the same setup to assess the repeatability of the technique and found to be repeatable within experimental error. The agreement between in situ and ex situ experiments was found to be reasonable, particularly the manner in which the stresses relaxed with time. In both studies it was found that initial stress relaxation was rapid, approximately 200 MPa in 15–30 min, after this a slower linear relaxation remained for the rest of the ageing heat treatment. This behaviour suggests creep may be the means by which stress relaxation takes place in this material during ageing.

RÉSUMÉ

Nous présentons l'étude par diffraction des neutrons de l'effet de traitements thermiques de vieillissement sur la relaxation des contraintes résiduelles dans des polycristaux de superalliages à base nickel trempés à l'eau. Deux expériences distinctes ont été menées ; la première consiste en une étude ex-situ au cours de laquelle les échantillons ont subi différents niveaux de vieillissement avant les mesures. La deuxième est un traitement thermique in-situ, qui a nécessité de chauffer puys de maintenir l'échantillon en température pendant la mesure par diffraction des neutrons. La technique in-situ a été conduite deux fois en utilisant le même dispositif expérimental afin de déterminer sa répétabilité, qui s'est avérée bonne, à l'incertitude des mesures près. Un agrément satisfaisant entre les techniques ex- et in-situ est obtenu, particulièrement en ce qui

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concerne la manière avec laquelle les contraintes sont relaxées au cours du temps. Dans les deux études nous obtenons que la relaxation des contraintes est initialement rapide, environ 200 MPa en 15 à 30 min, puis on observe une relaxation linéaire plus lente pendant le reste du traitement. Ce comportement suggère que la relaxation des contraintes au cours du traitement de vieillissement a lieu par un mécanisme de fluage.

1. Introduction

Residual stresses can be introduced into a material through a number of widely used manufacturing techniques including forging, heat treatment and machining. The level of residual stress within a component is known to affect performance as it can promote or retard failure mechanisms [1]. Characterisation of residual stress following manufacture is therefore a crucial part of understanding the in-service capabilities of any single component. Neutron diffraction is one of the variety of techniques available to measure strain and hence calculate residual stress within engineering alloys [2]. The technique makes use of Bragg's law; $\lambda = 2d\sin\theta$, to relate the diffraction angle (θ) to inter-planar spacing (d) within the crystal structure. By measuring diffracted neutrons of known wavelength, the crystal lattice structure of a material becomes a '3D atomic strain-gauge', where inter-planar spacings can be read from the diffraction angle and used to calculate strain.

The main advantage of neutron diffraction over other techniques is the ability to measure strain deep within the material bulk [3]. However, as a means to measure stress relaxation following ageing heat treatment, current studies have largely been limited to ex situ experiments. This means that in order to measure the level of stress relaxation, a sequence of samples must be measured each having experienced a different level of ageing. Two major issues arise with this method. The first is the shear quantity of samples required obtaining detailed time resolution in stress relaxation; a sample must be prepared and measured for each data point. The second issue is in sample to sample variability; heat treatment processes such as quenching and ageing are difficult to repeat to a high precision. As a result, stress relaxation measured using ex situ samples are subject to a scatter generated by even the most minor uncertainty in processing.

The alternative to measuring ex situ is to carry out stress relaxation in situ. This involves heating the sample to ageing temperature on the instrument whilst diffracting neutrons. In measuring in situ, the issues affecting ex situ are overcome; time resolution is only limited to the speed of the instrument, and sample-to-sample variation is non-existent. In addition to this, possible alignment errors are removed since alignment need only be carried once for an entire ageing experiment. In the present paper we compare the results obtained using both in situ and ex situ neutron diffraction ageing experiments on an advanced nickel based superalloy for disc application.

2. Experimental description

2.1. Sample preparation

2.1.1. Ex situ samples

Four sub-scale forgings of the nickel-base superalloy RR1000 were provided by ATI Ladish Forging. The nominal composition of RR1000 is given in Table 1. RR1000 is an advanced polycrystalline superalloy for high temperature application that is strengthened by almost 50 vol% γ'. The samples were of simple disc shape with a diameter of 76.2 mm, and a thickness of 25.4 mm (Fig. 1). Prior to any measurement, the samples were processed as follows.

Each sample was cut from a large pancake, forging at the same radial distance to ensure a closely matched thermo-mechanical history for each part. Sample 1 (S1) was sub-γ'-solvus heat treated followed by water quench. Samples 2 (S2), and 3 (S3) were both processed as S1 but with the addition of a 15 and 120 min ageing heat treatment at 760 °C respectively.

The water quench process was chosen in this sub-scale geometry to achieve high levels of residual stress despite the small geometry of the sample, whilst an ageing temperature of 760 °C was chosen since it is a typical ageing temperature of a γ' strengthened nickel-base superalloy.

2.1.2. In situ samples

As with the ex situ study, ATI Ladish Forging provided sub-scale forgings of the nickel superalloy RR1000 in the same geometry. Two samples were required for this study; sample 4 (S4) and sample 5 (S5), each of which were subjected to the same sub γ'-solvus heat treatment as S1–S3 followed by a water quench. Thus before any in situ ageing, S1, S4, and S5 were all processed identically, and were expected to have identical residual stress distributions.

Table 1
Alloy composition.

Alloy	Ni	Cr	Co	Mo	Ti	Al	C	B	Ta	Zr	Hf
RR1000	52.3	15.0	18.5	5.0	3.6	3.0	0.027	0.015	2	0.06	0.5

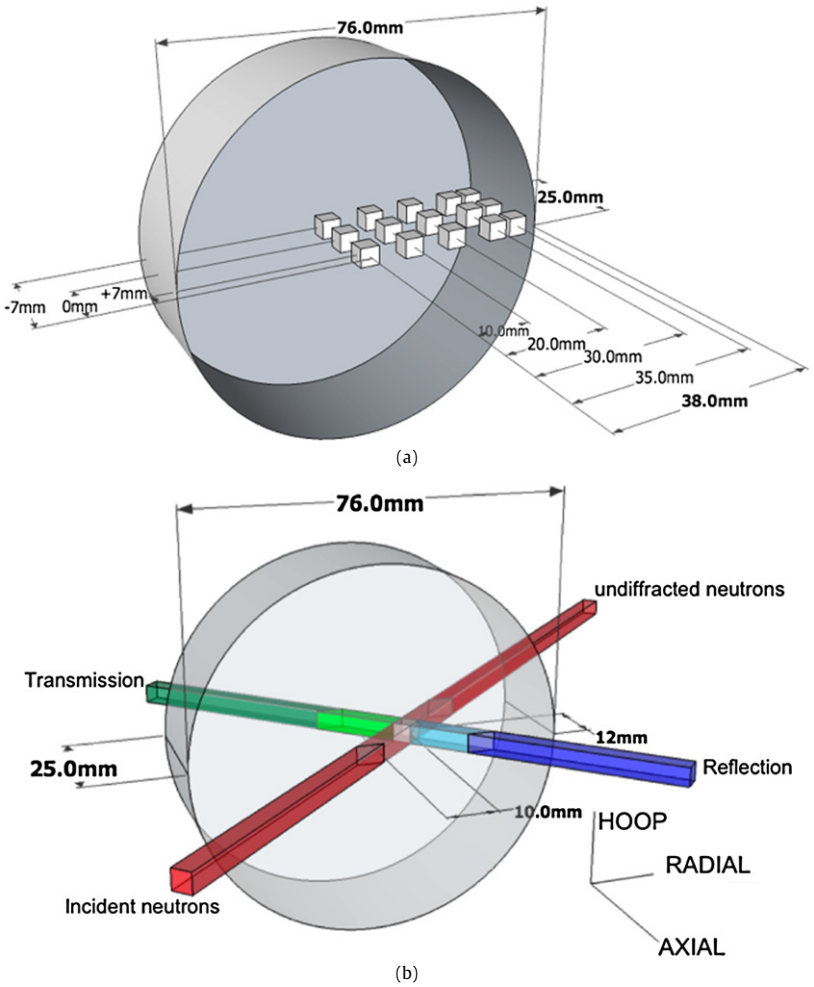

Fig. 1. Schematic diagram of the ex situ experimental sample setup; with (a) the location of gauge volumes within the sample, and (b) the measured strain directions.

2.2. Ex situ experiment – setup

All ex situ experiments were carried out at the dedicated strain scanning instrument STRESS-SPEC at FRM-II, Germany [4].

The FRM-II neutron source is of the reactor type, which means a single neutron wavelength and hence single reflection is chosen for the duration of the experiment. For the measurement of strain in RR1000 the (311) reflection was chosen for its low sensitivity to plastic strain and good representation of the bulk [5]. Using a wavelength of $\lambda = 1.55$ Å the (311) diffraction peak was recorded at $2\theta = 92^\circ$, which provided an approximately cubic gauge volume. It should be noted that the two main phases in nickel-based superalloys are the fcc matrix (γ) and the intermetallic $L1_2$ precipitates (γ'). Due to the similar crystal structure and size of the two phases, any fundamental reflection consist of overlapping γ and γ' reflections.

Samples were mounted in pairs on a computer controlled translator table and aligned initially using a theodolite, and then again using entry scans with the neutron beam. In order to calculate stress it was necessary to measure strain in three perpendicular directions, namely hoop, radial and axial as indicated in Fig. 1(b). Measurements were made along three line scans with five points per line at -7 mm, 0 mm, and $+7$ mm with respect to the centre thickness. In order to balance spatial resolution with count statistics, a gauge volume of $4 \times 4 \times 4$ mm^3 was chosen and defined using primary and secondary slits.

Measurement of strain using diffraction always requires a value for d_0 or strain-free lattice spacing. In this study d_0 values were obtained by extracting cubes from each sample of sufficiently small dimension as to be relieved of all macrostress [6]. A $6 \times 6 \times 6$ mm^3 cube was taken from each sample at mid-diameter and thickness, using electro-discharge machining (EDM) to extract a core, and conventional cutting to form a cube. Each cube was then measured in a follow up experiment at the SALSA strain scanning instrument at the ILL Grenoble [7]. A $4 \times 4 \times 4$ mm^3 gauge volume was aligned using entry scans to ensure full gauge volume immersion within the sample after which two measurements were made

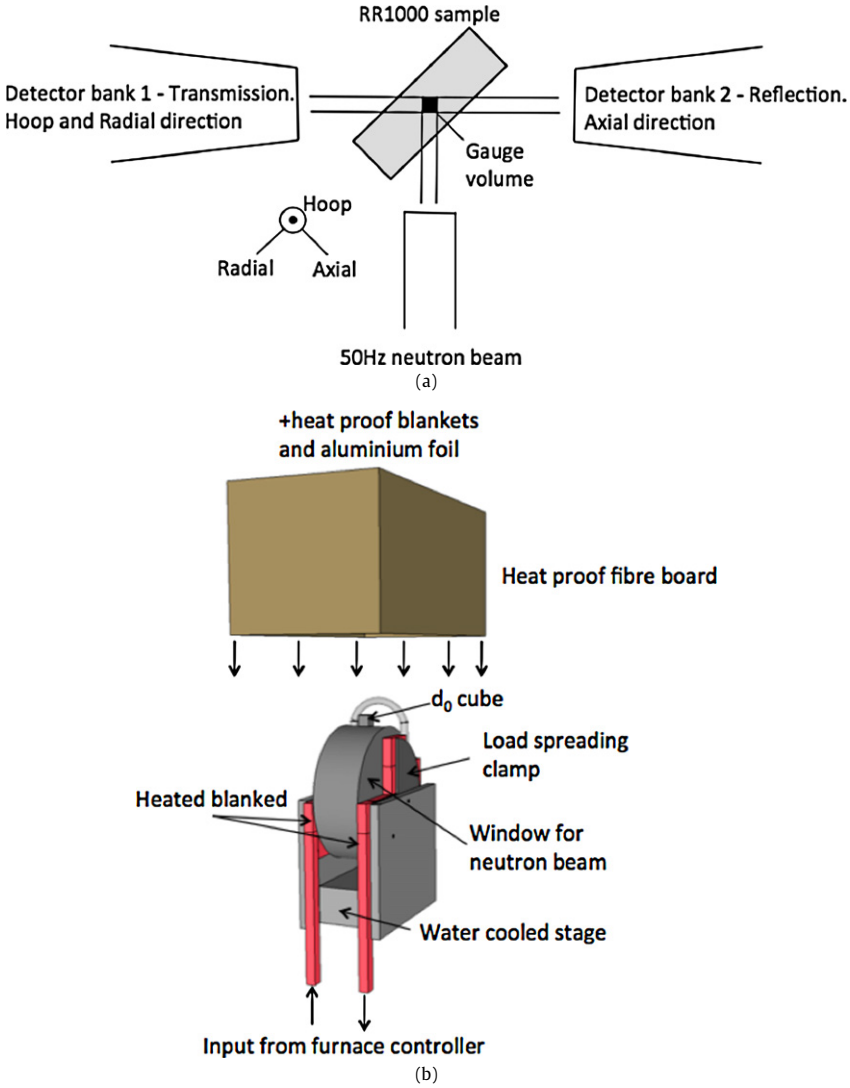

Fig. 2. Schematic diagram of the in situ experimental sample setup; with (a) the sample as placed on the instrument, and (b) the heated blanket arrangement and insulation.

using the same 311 *hkl* plane as the bulk measurements. In each case a relatively long count time was used to ensure good count statistics and a low peak position uncertainty. It was expected that absolute *d*-spacing values would differ between instruments; therefore, to calibrate between the two experiments a container of RR1000 powder was measured in each case.

2.3. In situ experiment setup

The strain-scanning instrument ENGIN-X (ISIS, UK) was chosen for in situ heat treatment. To bring each sample up the required temperature a heated 'blanket' arrangement was used. The blanket was made up from bead and/or block shaped elements of sintered alumina each containing resistance heating Ni Cr wire. From the sample geometry, heating elements were designed to wrap around the surface and thus through conduction, heat each sample to the required temperature. To allow the neutron beam to pass into the sample and back out unobstructed, a window was left open in the heated blanket as indicated in Fig. 2(b).

Prior to any in situ measurement, a sequence of thermo-couple heating tests was carried out on a spare nickel super-alloy sample. A schematic indicating thermo-couple location is given in Fig. 3. Through comparison to thermo-couple data provided by ATI Ladish Forging it was found that heating and cooling rates could be matched well to the ex situ samples. In particular, the cooling rate from 760 °C was found to be well replicated by turning off the furnace and then removing any insulation around the sample at approximately 500 °C. Results from this test also indicated that the quantity and positioning of insulation around the heated blanket and sample were crucial to achieving temperature uniformity. In a layer by layer structure our final setup was; nickel sample, heated blanked, stage and clamping, a 'box' arrangement of heat proof fibre

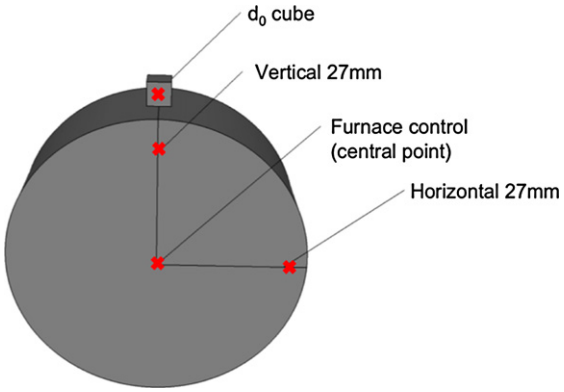

Fig. 3. Location of the type-K thermo-couples on the 'hockey puck' sample during the in situ experiment.

board placed over the top, heat proof blankets wire tied around the box, and finally aluminium foil wrapped around the outside (Fig. 2(b)). A second test was made on the beam line at room temperature to assess the impact of the insulating material on the neutron count statistics. It was found that while aluminium foil and fibreboard made little difference to the data, the heatproof blankets strongly attenuated the beam. As a result, the placement of the blankets was adjusted so as to maintain a similar window to the heating blanket.

For the measuring of a d_0 value, four $6 \times 6 \times 6$ mm^3 cubes had been extracted from an identically processed water quenched sample using EDM machining prior to the experiment. Before mounting on the instrument, each sample had one of the cubes attached to the sidewall using ceramic glue (Fig. 2(b)). Great care was taken in this process to maintain conductive contact between the cube and the sample by only using a small amount of glue around the edge of the cube. To ensure the cube reached the same temperature as the sample, part of the heating blanket was designed to loop over the top of this region to add heat. Extra insulation was also placed around the cube mounting area to prevent heat loss from the sample top. We were able to verify the effectiveness of these measures in maintaining temperature uniformity using a thermo-couple spot welded to the cube.

Each sub-scale forging complete with d_0 cube was placed in the heating blanket and then the entire setup clamped to a small stage containing a water-cooled base. The stage was mounted on a computer controlled translation table, which in combination with a theodolite allowed for sample alignment to within 0.2 mm. Sample clamping was made using two load spreading steel plates and four small bolts to sandwich the blanket against the sample face. The use of just four small diameter bolts for clamping was important to reduce conduction from the heating blanket to the stage.

The d_0 cube was aligned separately using the theodilite and measured using a $4 \times 4 \times 4$ mm^3 gauge volume to ensure full immersion of the gauge volume. The d_0 cube was measured at room temperature, while heating to temperature, at the beginning and end of the 760 °C hold period, and finally once back at room temperature again. This allowed data to be gathered on the thermal expansion of the alloy during heating, plus account for d_0 variation before, during, and after heat treatment.

The heating blanket was controlled using a Eurotherm furnace controller, which set the input current according to a centrally mounted control thermo-couple. Temperature uniformity was monitored throughout every experiment using type-K thermo-couples spot welded to the sample using small vanadium plates, as shown in Fig. 3. Using the described setup, we were able to maintain temperature to within 10 °C across the entire sample.

As a spallation source, ISIS generates neutron pulses with wavelengths ranging from ~ 0.5 to 6 Å. ENGIN-X receives the neutron pulse through a neutron guide tube via two rotating chopper arms which can reduce the neutron bandwidth depending on the needs of each experiment [8]. In this experiment the beam chopper was set to 50 Hz to give a bandwidth of $\lambda \approx 0.7$–2.2 Å, which improved count times and included the Ni(311) reflection. It was important to include this reflection as it was planned to use single peak fitting of the (311) reflection to maintain consistency across both experiments.

Whilst at heat treatment temperature, strain was scanned at a single location at the centre of the sample, this location was chosen for two reasons. Firstly, it is the location of peak tensile stress in the hoop and radial direction (Fig. 4). Secondly, at the sample centre the hoop and radial directions are the same. Thus on ENGIN-X the two detector system allowed to measure effectively all three strain directions simultaneously (Fig. 2(a)). Before and after the heat treatment, measurements were made at room temperature using longer count times to give an accurate assessment of the level of stress relaxation.

In planning this experiment, the gauge volume was chosen to be the same $4 \times 4 \times 4$ mm^3 gauge volume as the ex situ experiment, which on ENGIN-X is defined by the choice of collimator in addition to motorised slits. However, with this gauge volume and a path length of 35 mm count times approached 40 min per point, a time resolution less than that of the ex situ experiment (15 min). It was therefore necessary to open the slits and rock the collimators to increase the gauge volume to $8 \times 8 \times 8$ mm^3 and reduce count times to 15 min. Normally this would create an issue in averaging out stress gradients over such a large gauge volume. However from ex situ studies (Fig. 4) and finite element modelling it was known that the sample centre did not contain any steep stress gradients over this gauge volume. When measuring at locations other than sample centre, such as the d_0 cube, the gauge volume was reduced back to $4 \times 4 \times 4$ mm^3.

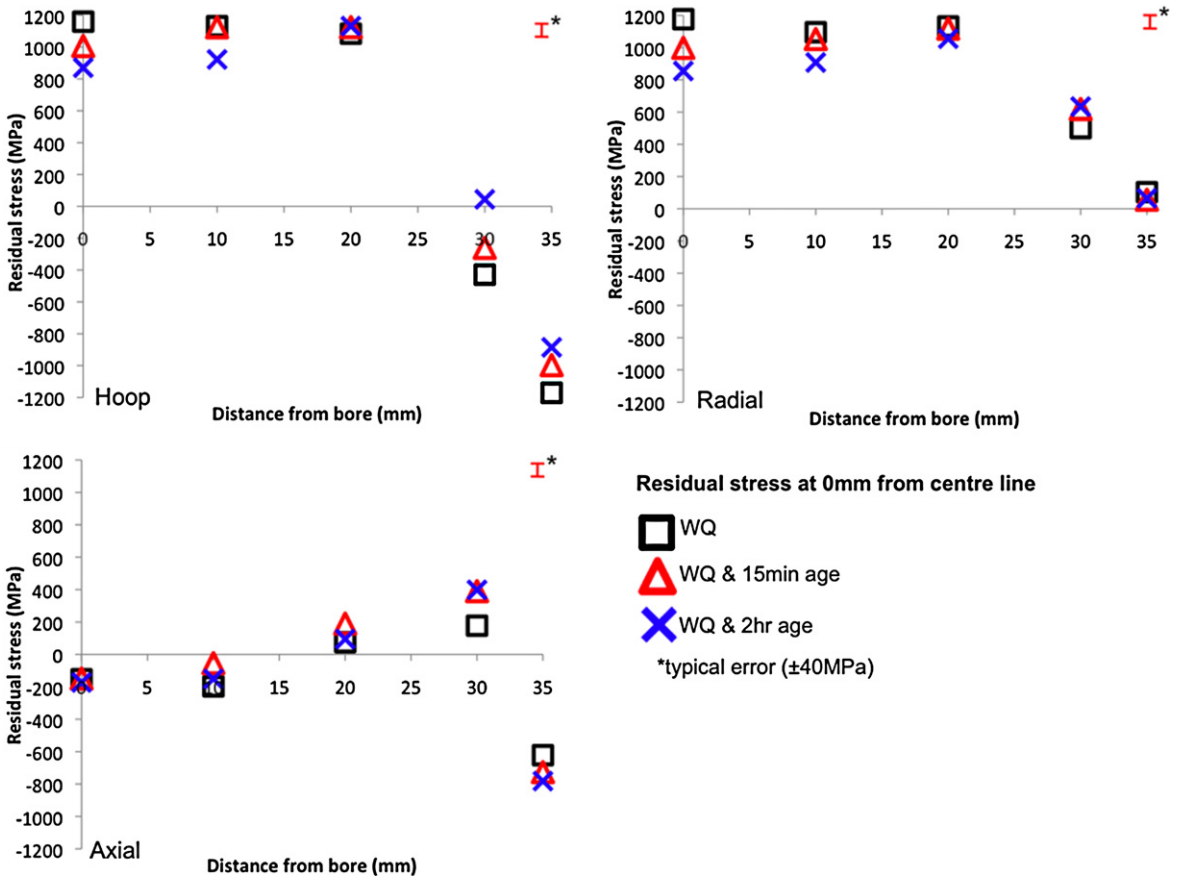

Fig. 4. Residual stress measurements made ex situ after water quench (WQ), WQ plus 15 min age at 760 °C, and WQ plus 2 h age at 760 °C.

Table 2
Material properties of nickel-superalloy RR1000.

Material properties of RR1000	
Young's modulus (bulk)	224 GPa
Poisson ratio (bulk)	0.326

Although a large gauge volume was used in this experiment to reduce count times, typical peak fit uncertainties in this experiment were high at approximately ±250 με or ±150 MPa in this particular material. Although this is greater than the recommended 100 με accuracy it was a necessary compromise to achieve the required time resolution in stress relaxation.

The first in situ sample, S4, was heated in stages from 25 to 200, 400, 600 and finally 760 °C and held for 5 min at each temperature to allow a d_0 measurement to be carried out. Once at temperature the gauge volume was positioned at the centre of the sample and measurements were made at approximately 15–20 min intervals throughout the hold time. Sample 4 was held to within ±1 °C of 760 °C for 8 h by controlling the furnace using the arrangement of thermo-couples as indicated in Fig. 3. Before cooling, a final d_0 was measured to check for any variation during the hold time. The furnace was then turned off and all insulation removed at ∼ 500 °C to cool the sample back down to room temperature. This process was repeated as closely as possible for sample 5.

2.4. Diffraction data analysis

Data analysis for both STRESS-SPEC and ENGIN-X was carried out by using in-house software to fit a Gaussian profile to the (311) reflection and hence derive the (311) lattice plane (d) spacing. In the same way, the data from each stress-free cube measurement was converted into a value of stress-free d-spacing, or d_0. The d and d_0 values were used to calculate strain in each of the three principal directions and then residual stress calculated from Eq. (1).

$$\sigma_x = \frac{E_{hkl}}{(1+\nu_{hkl})(1-2\nu_{hkl})}\left[(1-\nu_{hkl})\varepsilon_x + \nu_{hkl}(\varepsilon_y+\varepsilon_z)\right] \quad \text{and} \quad \sigma_y = \cdots, \quad \text{etc.} \tag{1}$$

where the principle strain directions are denoted as x, y, z and E_{hkl} and ν_{hkl} represent plane specific diffraction elastic constants. The elastic constants used in this particular study of the alloy RR1000 are given in Table 2.

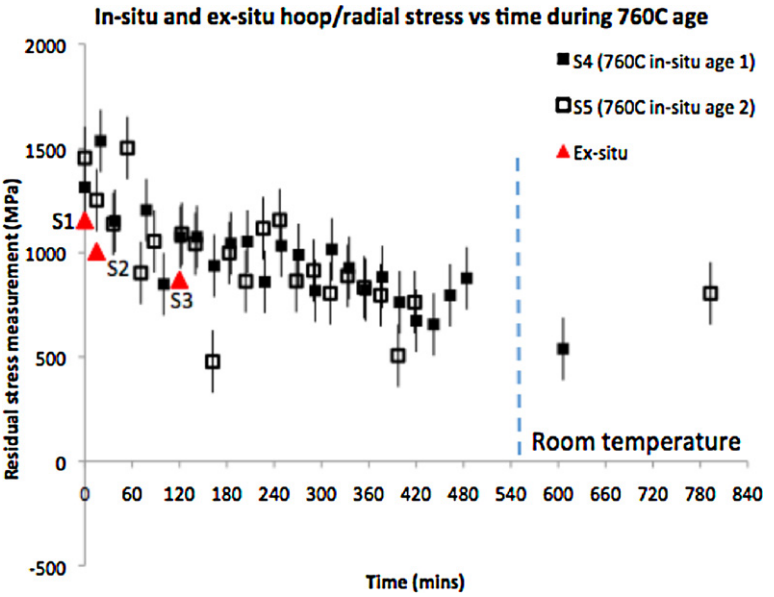

Fig. 5. Hoop and radial stress at the sample centre during an in situ ageing heat treatment at 760 °C. Data shown from both the in situ and ex situ experiments.

3. Results and discussion

The residual stress distribution in S1, S2, and S3, as obtained from the ex situ experiment are shown in Fig. 4. The results indicate peak levels of tensile residual stress approaching 1200 MPa are present in the hoop and radial direction at the centre of S1. Hoop stresses fall with radial distance to become strongly compressive towards the rim region, whereas radial stress falls to zero as it approaches the free surface at the rim. In comparison, axial stress in S1 is near zero from 0 to 20 mm radial distance. Further towards the rim of the disc a tensile 'spike' is observed followed by a rapid drop of the axial stresses into compression. Comparing residual stress levels in each of the three samples, we can assess the level of stress relaxation occurring during ageing by direction and location within the sample. It is clear from Fig. 4 that at 760 °C stress relaxation is greatest in the hoop and radial directions, and virtually non-existent in the axial direction. In the hoop direction stress relaxation of up to 300 MPa is seen in both the bore and rim regions, but this is reduced in the region in-between. Similar levels of relaxation are seen at the bore in the radial direction as the two directions as the same at the sample centre. However, moving out from the bore the level of stress relaxation in the radial direction falls until at the rim it reaches zero. In summary, the ex situ experimental data shows that the most significant stress relaxation occurs in and around the sample centre, making this location the most appropriate for the in situ experiment.

Results obtained from the in situ experiment are compared to those from the ex situ experiment in the hoop and radial direction in Fig. 5. The in situ data shows reasonable agreement between S4 and S5, indicating the experiment was repeated as faithfully as possible. However, the data does indicate a degree of scatter and anomalous measurement points, S5 in particular shows two at 150 and 380 min. Unfortunately scatter and anomalous results were a known risk in this investigation since time resolution was generally favoured over reducing experimental error. Peak fitting uncertainties in this experiment translated to ± 150 MPa uncertainty in stress in this material, this will have generated some scatter although the anomalous points for S5 exceed this level of uncertainty. Comparing absolute values obtained from the two experiments, it appears the in situ data is consistently 150–200 MPa greater than the ex situ. The reason for this is not clear, although a number of factors may have contributed given the different nature of each experiment. To overcome this offset and add a further comparison between the in and ex situ experiments, the stress relaxation with time was calculated as the difference between each measurement point and the initial as-quenched measurement.

Hoop and radial stress relaxation as a function of time for both the in situ and ex situ experiments is shown in Fig. 6; agreement between the two experiments is now much stronger. The behaviour of all five samples follow a similar trend, a rapid relaxation of ~ 200 MPa during the first 30 min and then a more linear trend for the remainder of the heat treatment. This behaviour may be indicative of a primary and secondary creep process of stress relaxation. It also suggests that even though samples S1–S5 all had been quenched in the same way, the starting stresses were different for the sample used during the in situ experiment compared to the samples generated for the ex situ experiment. This highlights the issue of possible sample to sample variations and the advantage of using in situ experiments to provide residual stress data that can be used to validate process modelling efforts.

Residual stress measurements from both the in situ and ex situ experiments in the axial direction are shown in Fig. 7; agreement between both in situ samples is high, indicating that the experimental process was repeated successfully.

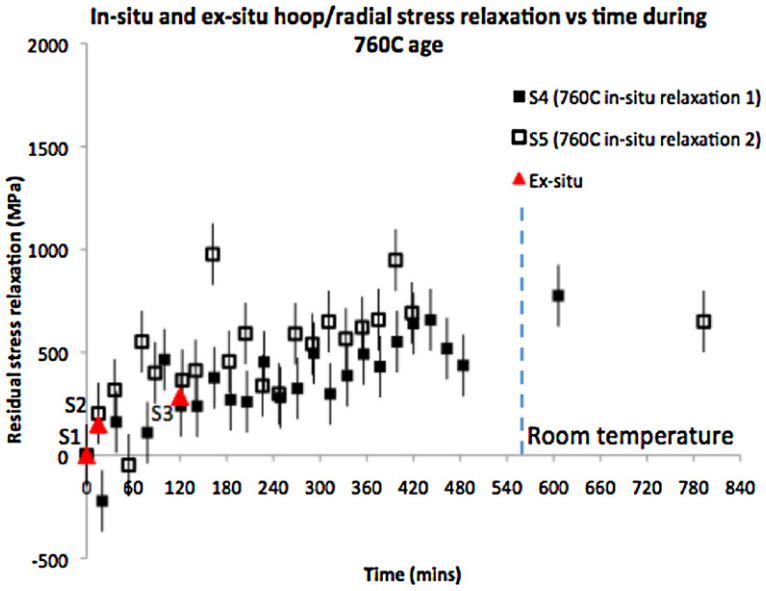

Fig. 6. Stress relaxation in the hoop and radial stress at the sample centre during an in situ ageing heat treatment at 760 °C. Data shown from both the in situ and ex situ experiments.

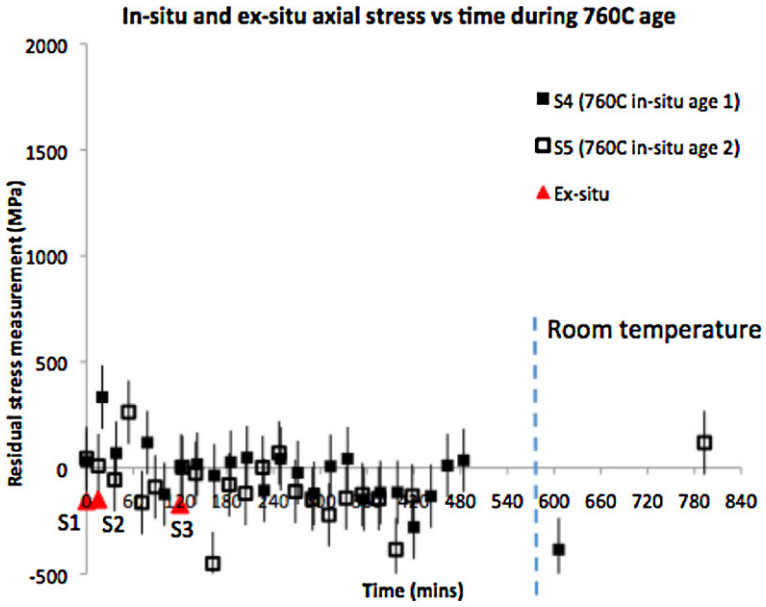

Fig. 7. Axial stress at the sample centre during an in situ ageing heat treatment at 760 °C. Data shown from both the in situ and ex situ experiments.

S4 and S5 show levels of stress very close to zero in the as-quenched state and remain relatively close to zero throughout the heat treatment although a reasonable degree of scatter is again seen in the data. In comparison, the ex situ samples, S1, S2 and S3, all indicated residual stress values of approximately −150 MPa, a difference of ∼ 150 MPa when compared to the in situ experimental data. Interestingly this same difference was seen in comparing in situ and ex situ data in the hoop/radial direction.

In terms of stress relaxation, Fig. 6 indicates that in both experiments the axial stress altered very little during the heat treatment; this being the case, axial stress relaxation with time has not been plotted. However, it is clear that whilst near zero stress relaxation was observed, it was observed equally in both experiments, which signifies agreement between in situ and ex situ measurement techniques.

4. Conclusions

The relaxation of residual stress as a result of an ageing heat treatment has been investigating using an ex situ experiment, followed by an in situ experiment. The ex situ experiment indicated that following a water quench process (S1) residual stress was found to peak at +1200 MPa in the sample centre in both the hoop and radial directions. The axial stress was found to be largely negligible with the exception of the near rim region. Following ageing heat treatments of 15 min (S2) and 120 min (S3), the bore region was found to show the greatest level of stress relaxation.

The in situ experiment was aimed at the bore region to best observe the stress relaxation during an ageing heat treatment. Two samples (S4 and S5) were measured in identical setups heating the sample to 760 °C whilst taking measurements in the sample centre every 15–20 min. Agreement between S4 and S5 was good although a large degree of experimental scatter was present in the data due to the need to reduce data acquisition times as far as possible. Comparing absolute stress values between the ex situ and in situ experiments showed almost identical trends but an offset between the two of approximately 150–200 MPa in all three strain directions. The trends in each experiment were compared by plotting the hoop/radial stress relaxation with time (Fig. 6). In terms of stress relaxation, the agreement between ex situ and in situ experiments was good; both experiments followed an initial rapid stress relaxation process followed by a slower linear rate of relaxation. It is thought that this indicates stress relaxation is occurring by a primary followed by a secondary creep process. After a 2 h age time, stress relaxation levels were: S3 (ex situ) – 285 MPa, S4 (in situ 1) – 240 MPa, and S5 (in situ 2) – 360 MPa. This places the ex situ data within the scatter of the in situ data, thus in terms of stress relaxation the two experiments are in agreement.

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