Materials Science & Engineering A 681 (2017) 32–40

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Visco-plasticity during in-situ cooling from solidification of a nickel-base single crystal superalloy using neutron diffraction

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ARTICLE INFO

Keywords:
Neutron diffraction
In-situ cooling
Visco-plasticity
Stress relaxation
Lattice strain

ABSTRACT

In-situ neutron diffractometry is performed to study the visco-plasticity in a nickel-base single crystal superalloy during in-situ cooling from high temperatures. It is found that visco-plastic deformation has two contributions from creep and stress relaxation, which are subject to the accumulation of dislocation activity and dislocation annihilation, respectively. Use has been made of the lattice strain evolution of the (200) γ+γ′ peak to confirm this effect. The decrease in lattice strain and macro-stress during in-situ cooling has been observed and confirms that there was softening taking place before thermal strain dominates at lower temperatures. Therefore, in-situ isothermal cyclic loading and relaxation tests under strain control, akin to thermal contraction during casting, have been carried out. A visco-plasticity law was then developed based on macro-strain development during creep and lattice strain evolution during stress relaxation within an appropriate timescale, where transient effects are captured. The constitutive law developed has been used to independently determine the evolution of stress and strain during in-situ cooling. The implementation of these findings into thermo-mechanical modelling during cooling from solidification is also discussed.

1. Introduction

Mechanical response during the investment casting of single-crystal superalloys is governed by the thermo-mechanical histories induced by mechanical deformation arising from differential thermal contractions of the metal (superalloy) and ceramic (mould and internal core), under casting conditions [1,2]. Cooling rates [3] and dendritic growth rates [4,5] can be controlled through optimising the furnace parameters and appropriate component design to minimise defects in single-crystal castings that also conform to acceptable dimensional tolerance. On cooling during and after solidification of a single crystal superalloy casting, little is known about the in-situ introduction of high temperature deformation [6,7] and the quantification of processing-induced deformation [8]. To the best of the authors' knowledge, finite element calculations that determine thermo-mechanical behaviour are traditionally contingent on the quality of the as-cast materials data and thermo-physical properties used in the model [9]. Such an approach has advantages, as it confers a predictive capability on turbine blade designers enabling them to produce a design space for achieving a minimal-waste manufacturing route and design criteria with functional integrity.

Processing-induced plasticity can be introduced at temperatures close to the γ′ solvus temperature in single crystal Ni-base superalloys [10–12]. In that study it was proposed that 2–3% plasticity induced at high temperatures leads to a dislocation network [1], which results in re-crystallisation during subsequent solution heat treatment to release the stored strain energy within the microstructure. Typical mechanisms resulting in a reduction in the dislocation density include creep and stress relaxation [13–19]. On the other hand, larger stresses arising at lower temperatures can lead to other commonly encountered casting defects, such as hot tears [20,21]. For a prediction of such casting defects, it becomes challenging to address how processing-induced plasticity should be quantitatively treated on the one hand, as well as whether time-dependent plasticity should also be considered [1]. The latter is important, since it is accompanied by relaxation and reduction in the stress and which is dependent on the time that the casting resides in the critical temperature regime during cooling [3].

Materials modelling used in thermo-mechanical analysis follows usually plastic flow (time independent) or visco-plastic flow (time dependent) approaches, the applicability of which depends upon the working temperature regimes [22,23]. Within process modelling of investment casting, owing to the limited high temperature materials

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http://dx.doi.org/10.1016/j.msea.2016.11.013
Received 16 August 2016; Received in revised form 21 October 2016; Accepted 2 November 2016
Available online 10 November 2016

data, the extrapolation of intermediate temperature tests permits the elasto-plastic analysis to be performed. Uniaxial tensile or compression testing using constant strain rate at elevated temperatures is conventionally carried out to derive the empirical or phenomenological laws for plastic flow for use in this analysis [1]. However, the predictive capability can be further improved by taking into account visco-plasticity [3]. Visco-plasticity is conventionally incorporated through consideration of secondary (steady state) creep testing using constant stress at elevated temperature. Although creep has technological significance [24–28] and offers a convenient means of including visco-plasticity in the analysis, it is not altogether relevant to investment casting process modelling. The principal objections here are related to the material condition, i.e. as-cast, segregated and not homogenous, but also the fact that visco-plastic behaviour has to be considered at small time scales to accurately capture the stress/strain transients during cooling, i.e. few minutes as compared to hours [3].

On the other hand, advent of in-situ testing capabilities equipped with synchrotron [29,30] or neutron [31,32] light sources provide substantial benefits for the observation of lattice strain evolution, which dictates the underlying macroscopic response at the component level. Following such an approach, the aim in this study is therefore three-fold.

First, neutron diffraction experiments are used to investigate the processing-induced visco-plasticity during the in-situ cooling of uniaxial tensile samples under representative casting conditions. Towards this end, the strain control mode was employed, where the length of the sample is fixed during cooling, which is akin to metal freezing around a rigid (immovable) ceramic core. As mentioned before, to capture the transients in stress/strain during cooling it is required to use a small neutron data acquisition time, but without compromising the error in strain measurement. Our previous work [31] has used 10 min acquisition time; however more recently a lower acquisition time of 3 min has been observed to be statistically reliable [32]. Additionally, from the measurement of macro-stress a key insight will be obtained on the efficacy of this choice of lower acquisition time, as the micro-strain directly measured using diffraction can be compared with the macro-strain deduced from the stress.

Second, isothermal relaxation testing is carried out to rationalise the visco-plasticity and stress relaxation behaviour at 950 °C and 1000 °C, and corresponds to the temperature range of the cooling experiments. Using the discrete data, best-fit equations for creep and stress relaxation can be obtained to quantify the visco-plasticity over the stress and temperature range, which is akin to the creep modelling approach that has been conventionally adopted [1–3]. Using these equations the visco-plasticity can be calculated during cooling and subsequently compared with the experimentally measured values. The rationalisation of process-induced visco-plasticity during cooling from solidification is then discussed.

One criticism for such an approach is that during solidification and subsequent cooling, alloys might not have not reached kinetic equilibrium and γ/γ' might not possess a good coherent relationship. It has been however demonstrated that γ' precipitation kinetics is diffusion controlled and therefore accompanied by interfacial equilibrium [33]. Consequently, the coherency is unaffected, as this is determined by the interfacial composition. On the other hand, the other important drawback of using isothermal creep/relaxation tests to determine visco-plasticity is to ignore the history dependence of the prior deformation existing within the solid, as it has cooled to the given temperature. One possible way of taking this into account was by adopting the cyclic relaxation tests, where the deformation in prior cycles is incorporated within the relaxation tests. Therefore, a third and important objective is to assess the validity of using isothermal loading tests by re-heating to calculate visco-plasticity. This is achieved by directly comparing the experimentally measured visco-plastic strain in the in-situ cooling experiments to that predicted using the relaxation/creep tests.

Table 1
The nominal compositions (in wt%) of CMSX4 single-crystal superalloy.

Cr	Co	Mo	Re	W	Al	Ti	Ta	Hf	Ni
6.5	9	0.6	3	6	5.6	1	6.5	0.1	Balance

Implications of this work will be beneficial for establishing a better mesoscopic description for the process modelling of investment casting by examining the conditions in which this approach can be adopted.

2. Method

2.1. Material

Tensile test pieces with a diameter of 5.85 mm and a gauge length of 29 mm of CMSX4 (nominal composition in Table 1) following the design used in [31], have been fabricated using the state-of-the-art investment casting process at the Precision Casting Facility (PCF), Rolls-Royce plc, Derby, UK. Prior to casting, moulds were seeded with the required orientation to ensure an axial orientation of the single crystals to within 5 ° from [100]. The single crystals were then directionally solidified in a small-bore furnace using a withdrawal rate of 5×10^{-5} m/s. In this manner, the orientation of the seed was conferred on to the test pieces that subsequently solidified; more details are included in [31]. Tensile bars were subject to electro-discharge machining (EDM) at the shoulders and grips, but the gauge length portion remained in the as-cast condition.

2.2. Neutron diffraction measurement

A series of samples were examined during in-situ heating, loading and subsequent relaxation of stress on the ENGIN-X instrument, at the ISIS pulsed neutron facility, Rutherford Appleton Laboratory, Didcot, UK. An optical furnace was used to heat the samples in air and a K-type thermocouple was held in contact with the sample to monitor temperature profiles. Isothermal stress relaxation tests were conducted at two temperatures, 950 °C and 1000 °C. The samples were heated at a ramping rate of 10 °C/min up to 800 °C and thereafter at a rate of 5 °C/min to the set temperatures. Prior to loading, each sample was held at the requisite testing temperature for nine minutes for thermal equilibrium and also to get the extensometer stabilised. At each temperature, a range of initial tensile stresses were used for stress relaxation testing and the stresses were applied at a rate a strain rate of 0.2% min^{-1}. Specifically, the loading started with a relatively small stress and dwelt at the stress for nine minutes under load control, which was then followed by stress relaxation under strain control mode for an additional 12 min. The initial stress was then increased by an increment of 10 MPa from the previous applied stress before another dwelling and stress relaxation test was conducted. The predicted stress ranges derived from a previous modelling study of solidification and cooling of simple one-dimensional and three-dimensional bobbin-type geometries has been taken as a reference for the selection of the initial stress for each temperature [2,3].

The loading axis in these experiments was horizontal and at 45° to the incident beam, allowing simultaneous measurement of lattice spacing both parallel and perpendicular to the loading axis. The samples were mounted such that the stress was applied along specimen axis. The positioning of the rig was such as to yield the longitudinal lattice spacing in one detector and the transverse lattice displacement in the other. The detectors were fixed at 90° to the incident beam and the data are obtained from the full ± 15° detector bank [31]. In all cases the neutron measurement volume was typically of the order of few cubic mm [34]. For the single crystal to be correctly aligned measurements are taken and the sample subsequently rotated until the desired lattice planes, in this (100) and (010), are located in the north-