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Rafting and elastoplastic deformation of superalloys studied by neutron diffraction☆

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ABSTRACT

The rafting of monocrystalline Ni- and Co-based superalloys has been studied by neutron diffractometry. Lattice parameter misfit values and the difference in phase stiffnesses at room temperature, 650 °C, and 900 °C are presented. These microstructural parameters should assist in refining computer models that aim to predict precipitate evolution in superalloys and aid future alloy design. The nature of rafting is shown experimentally to be dependent upon the lattice parameter misfit. The 900 °C yield strength of the γ-phase of the Co-based superalloy with a rafted microstructure occurs at ~100 MPa, when loaded at a low strain rate.

Ni-based and the latest Co-based superalloys are strengthened by ordered $L1_2$ γ′-precipitates which are embedded coherently within a disordered fcc γ-matrix. Neutron diffractometry has been applied extensively to the study of polycrystalline [1–6] and monocrystalline [7] Ni-based superalloys to characterise the constrained (or effective) phase elastic constants, $E^{\gamma',\mathrm{eff}}$ and $E^{\gamma,\mathrm{eff}}$, and constrained lattice parameter misfit values

$$\delta^{\mathrm{eff}} = \frac{2(d^{\gamma',\mathrm{eff}} - d^{\gamma,\mathrm{eff}})}{d^{\gamma',\mathrm{eff}} + d^{\gamma,\mathrm{eff}}} \quad (1)$$

where $d^{\gamma',\mathrm{eff}}$ and $d^{\gamma,\mathrm{eff}}$ are the constrained, measured *d*-spacing values at the temperatures and stresses of interest. Evaluation of the constrained phase elastic constants gives confidence in more advanced neutron diffraction studies of Ni-based superalloys, such as intergranular and interphase load partitioning [1–3,5–7], deduction of slip modes [4], and lattice strain evolution during creep regimes [18]. Similarly, in-situ X-ray diffraction at synchrotron facilities has been utilised to gain insight into alloy behaviour including: the effect of alloying on lattice parameter misfit [8]; the kinetics of phase transformations [9]; the temperature dependence of lattice parameter misfit [10–13]; and, the effect of applied stress at elevated temperatures [14–17]. However, to date, the study of Co-based superalloys by neutron diffraction has been limited. The variation of lattice parameter misfit with temperature has been deduced in polycrystalline ternary and quaternary alloys [19–23], and constrained phase

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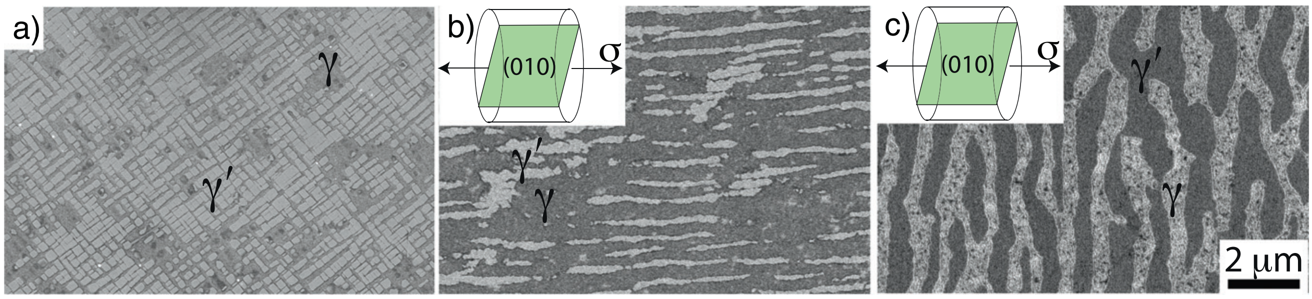

Fig. 1. Representative secondary emission SEM images of etched cross-sections showing a) the cuboidal γ' microstructure and b) the rafted γ'-microstructure of the single crystal Co-based superalloy (L19C); and c) the rafted γ'-microstructure of the single crystal Ni-based superalloy (CMSX-4). Insets: The loading direction is shown relative to the precipitate orientation. The γ-phase was etched with an aqueous solution of 2.5 vol% phosphoric acid at 2.5 Vdc for ~1 s.

elastic constants have been measured at room temperature and 650 °C [21].

The deduction of phase elastic constants and lattice parameter misfit values by neutron diffraction are of fundamental interest, as the strengthening γ'-precipitate size and morphological evolution at elevated temperatures (with or without applied stresses) are dependent on these microstructural parameters [24]. The direction and rate of rafting (directional coarsening at elevated temperature and stress) are dependent on the direction and magnitude of the applied load, the lattice parameter misfit value, and the fractional difference in constrained elastic constants of γ' ($E^{\gamma',\mathrm{eff}}$) and γ ($E^{\gamma,\mathrm{eff}}$) [24]

$$m^{\mathrm{eff}} = \frac{2(E^{\gamma',\mathrm{eff}} - E^{\gamma,\mathrm{eff}})}{E^{\gamma',\mathrm{eff}} + E^{\gamma,\mathrm{eff}}} \quad (2)$$

The influence of dislocations on precipitate rafting must also be considered during creep. Specifically, it is known that raft orientation and particle growth rate are dependent upon the relief of lattice parameter misfit strains in specific channels by dislocations at the γ/γ' interface [25,26].

This paper studies the lattice strain response of both γ- and γ'-phases during loading of monocrystalline Ni- and Co-based superalloys with cuboidal and rafted γ'-microstructures at room temperature, 650 °C and 900 °C. δ^{eff} values of the Ni-based superalloy, with rafted microstructures, are found to be highly anisotropic, while those of the Co-based superalloy values are isotropic. Rafting is shown to be dependent on the lattice parameter misfit sign and magnitude, in agreement with theory [27]. Load partitioning from γ to γ' occured on yielding of the Co-based superalloy with rafted γ'-microstructures. The quantification of the constrained lattice parameter misfit values and elastic constants from room temperature to elevated temperatures will facilitate models of the precipitate evolution in Co-based superalloys, as has been attempted for the Ni-based superalloys, *e.g.* [28].

(100) oriented single crystal bars of the Ni-based superalloy CMSX-4 were provided by Rolls-Royce plc.[1] , Derby, U.K., following a proprietary solution heat-treatment and a two-step aging process of 1140 °C/2 h + 870 °C/16 h. (100) oriented bars of single crystal Co-based superalloy with the composition Co-27.3Ni-2.7Al-1.4Ti-5.8W-4.2Mo-2.8Nb-2.8Ta wt% (Co-28.8Ni-6.2Al-1.8Ti-2.0W-2.7Mo-1.8Nb-0.9Ta at.%), determined by inductively coupled plasma optical emission spectrometry and labelled L19C, were cast by Alcoa-Howmet Research Center and Exothermics Inc., New Jersey, U.S.A.; with a final heat-treatment of 1300 °C/24 h + 900 °C/24 h. The microstructure following this heat-treatment is shown in Fig. 1a.

12:7 mm gauge diameter Ni-based superalloy single crystal and 6:35 mm gauge diameter Co-based single crystal superalloy cylindrical tensile specimens were machined from the heat-treated bars, with 40 mm gauge lengths. Samples of the Co-based superalloy were crept under tension at 900 °C/100 MPa for 20 h with 0.2% strain accumulation, producing P-type γ'-rafts (aligned parallel to the tensile loading direction), Fig. 1b. Samples of the Ni-based superalloy were crept under tension at 1150 ° C/100 MPa for 10 h with 0.7% strain accumulation, producing N-type γ'-rafts (aligned normal to the tensile loading direction), Fig. 1c. The samples were heated by an induction coil with the sample grips chilled. Displacement was recorded with a 12 mm high-temperature extensometer centred on the gauge length. The temperature gradient across the extensometer length was always within 10 °C of the target temperature, and thus can be considered isothermal.

In order to study the lattice strain response of each phase in the linear elastic loading regime, *in-situ* neutron diffraction measurements were performed by sequentially stepping the applied tensile stress after each measurement at room temperature, 650 °C and 900 ° C with samples of: (i) a Co-based superalloy with cuboidal γ'-microstructure (Fig. 1a); (ii) a Co-based superalloy with P-type γ'-rafts (Fig. 1b); (iii) a Ni-based superalloy with N-type γ'-rafts (Fig. 1c). All 900 °C measurements were taken between 15–150 MPa. The room temperature and 650 °C measurements of the rafted γ' Co-based superalloy were taken between 15–300 MPa, while those of the cuboidal γ' Co-based superalloy and rafted γ' Ni-based superalloy were taken between 15–550 MPa. These stress ranges were selected to try to ensure that the alloy under test remained in the elastic regime during testing. Each ramp in stress between diffraction measurements was linear with time and occurred over a 1 min time period.

Neutron diffraction measurements were performed on VULCAN [29], the time-of-flight (TOF) engineering neutron diffractometer at the spallation neutron source (SNS), Oak Ridge National Laboratory (ORNL), Tennessee, U.S.A., in a similar manner to described previously [7]. The incident beam, sample and detector banks were positioned to give the longitudinal lattice plane displacement in one detector and the transverse lattice plane displacement in the other. The beam frequency was 60 Hz in the high resolution mode with count times of 20 min for each diffraction measurement performed and a 7 mm irradiated gauge volume was centred at the middle of the specimen and extensometer.

Pseudo-Voigt peak functions were fitted to the diffraction spectra peaks by an iterative least-squares error minimisation procedure, in a similar manner to [1,7]. For the case of the Co-based superalloys, the intensities of the {300} γ' peaks were too weak to determine accurately the γ' d-spacing, but were sufficient to identify the γ' peak-position in the {200}$\gamma + \gamma'$ doublet peak, by $d^{\gamma',\mathrm{eff}}_{\{200\}} = 1.5 d^{\gamma',\mathrm{eff}}_{\{300\}}$. The Co-based superalloy {200}$\gamma + \gamma'$ peaks were widely separated (indicating a large lattice parameter misfit value) and fitted with a

[1] Any mention of commercial companies or products herein is for information only; it does not imply recommendation or endorsement by NIST.