	Experiment title: Topologically close packed phases in nickel-base superalloys	**Experiment number**: MA2690
Beamline: ID22	**Date of experiment**: from: 9 July 2016 to: 11 July 2016	**Date of report**: 27 Feb 2022
Shifts: 6	**Local contact(s)**: Catherine Dejoie	*Received at ESRF:*

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Report:

The turbine discs at the core of gas turbine aeroengines are made from polycrystalline Ni-base superalloys. These alloys typically comprise an fcc Ni-base matrix (γ) reinforced by a fine dispersion of Ni_3Al-base (γ') precipitates. The new superalloys being developed for future, more efficent civil aviation typically utilise higher γ' volume fractions and increased concentrations of refractory metals to deliver the strength and creep resistance required at elevated temperatures. However, such developments typically increase susceptibility to topologically close-packed (TCP) phase formation that compromise alloy service life, including the μ, σ, P & R phases. It is therefore critical that TCP phase precipitation is understood. Regrettably, state-of-the-art thermodynamic models cannot predict TCP phase occurrence in these multi-element systems with the fidelity required, necessitating experimental characterisation. However, this can be challenging as TCP precipitation is kinetically inhibited and typically requires in excess of 1000 hours at temperatures between 700-900˚C. Further, the extremely low volume fractions of TCP phases produced (~2%) and the low symmetry of their crystal structures prohibits reliable characterisation using laboratory X-ray diffraction of solid samples. The compositional and morphological similarity between the TCP phases, and the carbides that form, also prevents unambiguous discrimination between the phases using scanning electron microscopy. While the crystal structure of these phases can be determined using transmission electron microscopy, the localised nature of the method prevents reliable assessment of their volume fractions. As a consequence, quantification of TCP phases is typically achieved by X-ray diffraction of electrolytically extracted powders. However, serious concerns exist over the potential loss of TCP phases during the extraction and filtration processes. This casts doubt over the reliability of quantitative information obtained from such studies.

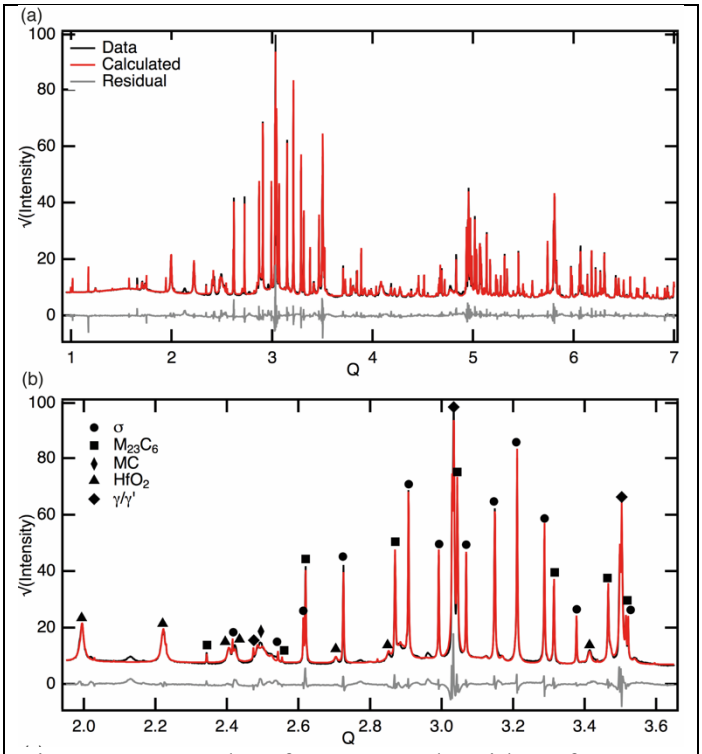

Figure 1: XRD data for extracted residue after 5000 hours at 800°C. (a) Synchrotron XRD data from ID22, (b) magnified view of synchrotron XRD data.

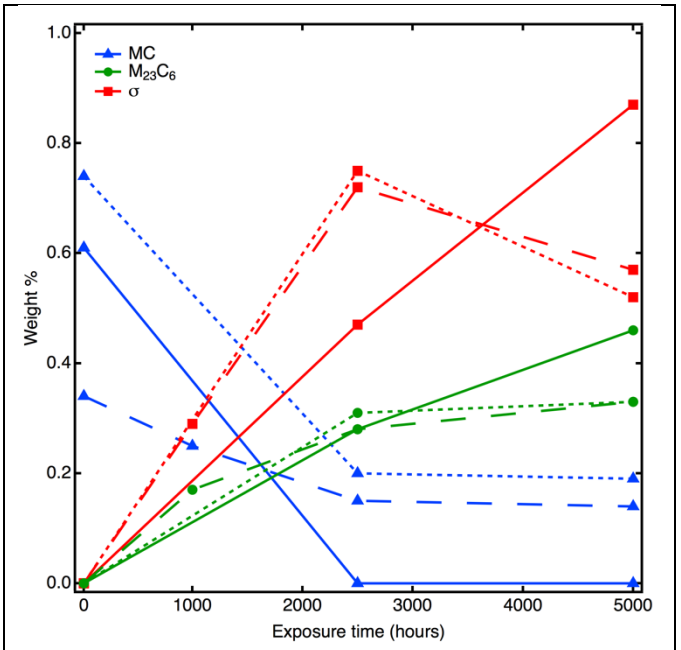

Figure 2: Weight % of minor phases in RR1000 at 800°C calculated from data collected by different experimental methods (solid line = solid sample measured using synchrotron XRD, dashed line = extracted sample measured using synchrotron XRD, dotted line = extracted sample measured using laboratory XRD).

In this study, high-resolution synchrotron X-ray diffraction was used to quantify the volume fractions of the different TCP phases produced in a Ni-base superalloy after thermal exposure at 800˚C for 100, 500, 1000, 2500 & 5000 hours. Data were acquired from both solid and electrolytically extracted powder samples. This enabled direct comparison of the effectiveness of synchrotron diffraction for the quantification of TCP volume fractions and provide a benchmark for assessing the stability of other alloys. Examples of the data acquired are provided in Figure 1 along with data acquired using a laboratory X-ray source for comparison. These data effectively demonstrated the potential of synchrotron X-ray diffraction for identification of the TCP phases formed and their associated volume fractions.

The study showed that synchrotron diffraction of solid specimens allows the detection of very low phase fractions (~0.3 wt.%) of minor phases in a Ni-base superalloy. Although, the exact detection limit depends on the phase under consideration as it is affected by various factors including the positions of the peaks relative to the other peaks in the data and the peak breadths.

The phase fractions obtained for the solid and extracted samples were of a similar order of magnitude providing encouraging evidence that the results produced by the extraction method are generally representative of the phases present in the material. The low phase fractions of σ present in all of the superalloy samples prevented a more detailed assessment of the accuracy of the method from being made. However, it can be concluded that synchrotron diffraction analysis of solid samples is not suitable for the quantification of phases present in quantities below ~0.3 wt.% in commercial alloys as these result in peaks with extremely low intensities that can be difficult to fit with confidence.

The findings of this experiment have been published in:

Comparison of Methods for Quantification of Topologically Close-Packed Phases in Ni-Based Superalloys
A. S. Wilson, K. A. Christofidou, A. Evans, M. C. Hardy & H. J. Stone
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