ESRF	**Experiment title:** In-situ time-resolved combined SAXS/WAXS study of precipitation in a Ni-base superalloy	**Experiment number**: MA3418
Beamline: ID31	**Date of experiment**: from: 2017-03-16 to: 2017-03-19	**Date of report**: 2017-06-13
Shifts: 12	**Local contact(s)**: Veijo Honkimäki	*Received at ESRF:*

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Background

The purpose of the experiments was the in-situ characterization of precipitation kinetics in a newly developed Ni-base superalloy (Haynes 282) under specific heat treatments by wide and small angle scattering. The Haynes 282 superalloy has a unique combination of high temperature strength, creep resistance, thermal stability and fabricability. This alloy has hardening γ' precipitates (~20 vol.%), primary MC carbides and secondary carbides like M_6C and $M_{23}C_6$ as its micro constituents. The precipitation, distribution and morphology of these micro constituents are controlled during processing and heat treatments in order to optimize the properties. A recent study on Haynes 282 sheet shows that small variations in heat treatment temperature can cause drastic changes in the mechanical properties due to microstructural changes. For example, by reducing the temperature by only 14 °C compared to recommended practice during the first aging step, a bimodal distribution of γ′ with two different morphologies was observed – small (20 nm) spherical and large (100–200 nm) cuboidal. It also showed the presence of a mix of carbide and γ′ in the grain boundaries. With the inclusion of an additional solutionizing step before ageing, the grain boundary carbide morphology changed from discrete particles seen after standard treatment, to an interconnected structure that lead to a significant drop in room temperature tensile ductility. The goal was to use *in-situ* time-resolved synchrotron diffractometry and small angle scattering during heat treatments of a newly developed Ni-base superalloy (Haynes 282) in order to study the effect of different parameters on the precipitation of carbides, hardening precipitates and possible high-temperature phases. The aim is to develop sufficient knowledge to allow tailoring and optimization of the microstructure and properties for different applications. The work is part of a PhD project with the goal to design improved heat-treatments of Haynes 282 for aerospace applications.

Experimental setup and issues

Experiments were performed at an energy of 63 keV and a beam size of 0.2 x 0.2 mm^2. Stick shaped specimens with a 1x1 mm cross-section were placed in a quartz dome, allowing the creation of an inert environment (Fig. 1). This solution also allowed rotation of the specimen during exposure for powder averaging. The specimens were heated using induction heating, and the temperature was monitored using a multi-wavelength pyrometer. WAXS patterns were obtained on a Pilatus3 X CdTe 2M detector with an exposure time of 10 s. After obtaining the WAXS pattern, the Pilatus detecter was moved out of the beam path, allowing 5 s acquisition of SAXS patterns at a Varex XRD 1621 xN ES detector at the end of the vacuum fligh tube. Due to the required movement of the detecter between WAXS and SAXS acquisitions, the acquisition of each set of WAXS and SAXS pattern took about 4 minutes. Combined SAXS and WAXS was done at this frequency throughout the tests. The main issue encountered during the experiment was that the temperture could not be accurately measured. The sample area needed for pyrometry required that the

temperature was measured on the specimen holder, instead of the specimen. Even though some cross-correlations using a thermo-couple placed at the beam position were attempted before the measurements, it was not possible to obtain a reliable calibration function between sample temperature and pyrometer read-out. Instead, the appearance and dissapearance of γ' supperlattice diffraction rings were used to set pyrometer control points corresponding to the critical temperatures above and below the γ' solvus for the first stage of heat treatments.

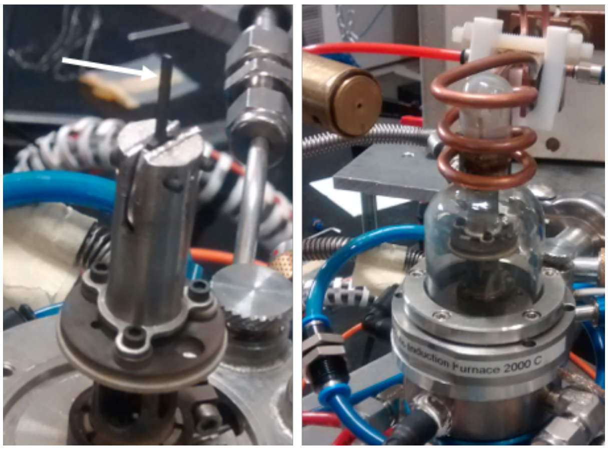

Figure 1. Experimental set-up. Left: Stick shaped specimen, indicated by white arrow, clamped in the specimen holder. Right: Specimen enclosed in the quartz dome, with induction heater in place.

Key scans

Out of the proposed six heat treatment cycles, four were completed within the allocated time. These consisted of subjecting two different starting conditions (as-received and solution treated) to two different heat treatment cycles (carbide stabilisation above or below the γ' solvus, followed by a γ' precipitation treatment for 6 h) In addition, one constant heating/cooling rate scan was performed in order to establish accurate precipitation/dissolution temperature for the different phases.

Evaluation strategy

The experiments will be evaluated using standard techniques regarding data reduction and analysis. Due to the problems with temperature measurements, lattice parameter data as a function of temperature from a separate in-situ synchrotron diffraction experiments with the same material but more accurate temperature measurements, will be used to calculate the temperature from the observed γ lattice parameter during the cycles.

Initial results

Some preliminary evaluations have been done at this stage, indicating that the tests were completed succefully. Figure 2 show some examples of evaluated WAXS data obtained during the temperature ramp cycle, where the appearance and dissapearance of all phases of interest can be seen. Similar evaluations of the SAXS data also shows that the size of the γ' precipitates can be tracked during precipitation and dissolution. Further analysis of the data sets, and in particular the heat treatment cycles, will be reported in scientific publications.

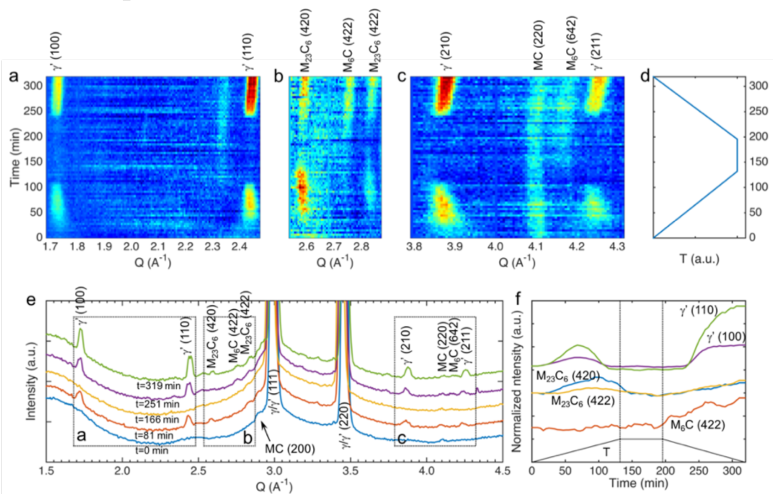

Figure 2. Preliminary WAXS results from the temperature ramp cycle. (a)–(c) shows evolution of the γ, γ' M23C6, M6C and MC peaks during the temperature cycle defined in (d) (note that the temeprature has not been calibrated yet). (e) shows spectra at different times and locations of the Q-ranges in (a)–(c). (f) shows the evolution of the normalized integrated intensities of selected peaks, shifted in y-direction for clarity.