ESRF	**Experiment title:** In-situ coherency strain measurements in advanced nickel base superalloys	**Experiment number**: Me 776
Beamline: ID31	**Date of experiment**: from: 28.06.2004 to: 02.07.2004	**Date of report**: 15.02.2005
Shifts: 9	**Local contact(s)**: **Francois Fauth**	*Received at ESRF:*

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Report:

In-situ and ex-situ measurements of the misfit between the matrix (γ) and precipitates (γ') in next generation nickel base superalloys have been successfully carried out. The measurements have demostrated that the misfit between the two phases in the next generation alloys is significanlty smaller than predicted. In-situ experiments at 800°C have also shown that the misfi varies, and in some cases even cycles, with time. It should be noted that the time dependant misfit variations at high temperature are small and it is only due to the high resolution of the powder diffraction beam line ID31, that the change of misfit could be detected.

Nickel-base superalloys are widely used in turbine engines because of their high temperature capability. The key feature that imparts high temperature strength to superalloys is a high volume fraction of the intermetallic γ' ($Ni_3(Al, Ti)$, $L1_2$ structure) phase that is coherently precipitated within γ (Ni, fcc). A large intragranular volume fraction of γ' gives better high temperature properties compared to current nickel base superalloys but microstructural stability and welding becomes an important issue. The size, morphology and distribution of the γ' phase is critical in determining alloy strength. It is therefore desirable to use a combination of heat treatment temperatures, hold times and quench rates to provide an optimised distribution of the γ' phase. Recent microstructural studies have shown that during annealing/aging of prospective next generation nickel-base superalloys, γ' coarsens to a critical size and then divides into smaller particles. The experiment carried out on ID31 aimed to undertake ex-situ and in-situ experiments, which would clarify the role of misfit between γ and γ' in terms of microstructural instability.

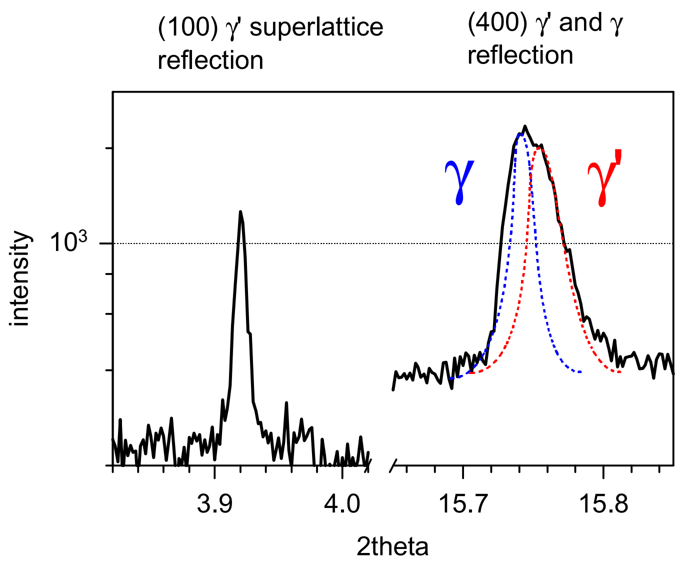

Fig.1: Example of the (100) superlattice reflection and (400) γ and γ' reflections recorded during me776. Note the small misfit between γ and γ' observed for the (400) reflection. The determination of the misfit was only possible by using the information of the (100) superlattie reflection.

The experiment was carried out using cylindrical samples of 1 mm diameter, which were placed on the powder diffraction rotation stage. High resolution scans were carried out using all nine detectors of the diffractometer. The energy used for this experiment was 60keV and the 2θ range scanned for each sample was between 3.5 and 25°. The misfit

between γ and γ′ was determined by measuring the position of the superlattice reflections of the coherent, ordered γ′ phase together with the main peaks. The measurement of the superlattice reflection allows determining the lattice parameter of the γ′ phase. This information can then be used to deconvolute the overlapping γ and γ′ main reflections to determine the exact position of the matrix lattice parameter. It was found that the highest accuracy for the γ lattice parameter was achieved by studying the (400) reflection. The (400) reflection gave a good combination of strong signal and due to its relatively high diffraction angle sufficient resolution to observe the separation of γ and γ′ (see Fig. 1). One difficulty encountered during this experiment was the asymmetric peak shape at low diffraction angles, which affected the determination of the γ′ lattice parameter (only the low angle (100) and (110) superlattice reflection could be used to identify the lattice parameter of γ′). By using a standard powder diffraction calibration sample the peak shift in the low angle region was measured and a correction factor as a function of diffraction angle was calculated. In this way it was possible to directly relate the lattice parameter measured on the (100) and (110) superlattice reflection with the (400) γ′ peak position. A double peak fitting routine with the peak position known for γ′ was then applied to determine the d(400)-spacing of the matrix.

During me776, first ex-situ experiments of various nickel-base superalloys heat treated to different conditions were carried out. Each scan took about 25 minutes, which enabled us to measure the misfit with an accuracy of better than 10 microstrain. In-situ experiments using a hot-air blower at 800°C were carried out with a 8 times faster scanning rate than during ex-situ experiments. However, due to a relatively large scatter in the data, data averaging over seven measurement points was applied. Figure 2 plots the time dependent misfit variation for two different conditions. The smoothness of the curves indicates a strain accuracy better than 5 microstrain. When the material was slowly cooled (billet cooling) the misfit is slightly smaller than when an intermediate cooling rate was applied. More interestingly is the difference in time dependent misfit variation between the two samples. As it can be seen in Figure 2, the slowly cooled sample (billet cooled) displays a steady misfit increase for seven hours before it reaches a plateau. The intermediate cooled sample on the other hand seems to display misfit cycling for almost 10 hours before a sudden increase of misfit is observed. It should be emphasised that the misfit and its variation seen in Figure 2 are of very small magnitude. It is therefore unlikely that the γ/γ′ misfit causes splitting of γ′. However, the misfit variation might be a result of the γ′ coarsening/splitting and therefore would be the first ever in-situ observation of this behaviour. More work is required to clarify the relationship if any between the cycling of the γ/γ′ misfit and the coarsening/splitting behavour of γ′.

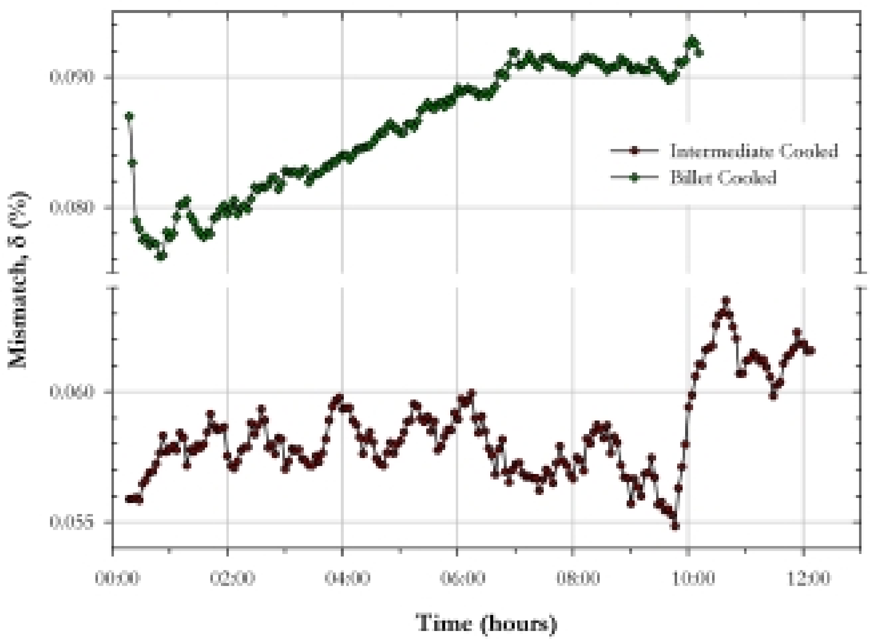

Figure 1: Mismatch as a function of time at 800°C after two different cooling conditions. Note the significant difference in mismatch variation for the two different samples.

Publications arising from me776: Papers to be submitted for publication:

1. Influence of composition and cooling rate on the constrained and unconstrained lattice parameters in advanced polycrystalline nickel-base superalloys, *R.J. Mitchell, M. Preuss, M.C. Hardy and S.Tin, to be submitted to Scripta Met.*
2. Interrelationships between composition, γ′ morphology , hardness and γ-γ′ mismatch in advanced polycrystalline nickel-base superalloys: Part I: Solutioning and cooling rate – Ex-situ studies, *R.J. Mitchell, M. Preuss, M.C. Hardy and S.Tin, to be submitted to Met Trans A*
3. Interrelationships between composition, γ′ morphology , hardness and γ-γ′ mismatch in advanced polycrystalline nickel-base superalloys: Part II: Ageing at 800°C – In-situ studies, *R.J. Mitchell, M. Preuss, M.C. Hardy and S.Tin, to be submitted to Met Trans A*