ESRF	**Experiment title:** Measurement of the volume fraction of the γ' phase of nickel base single crystal superalloy up to complete solutionizing	**Experiment number:** HS243
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Report:

The high mechanical performances of single crystal superalloys are due to a precipitation hardening : an ordered γ' phase with a $L1_2$ structure precipitates inside a matrix of disordered γ phase and blocks the dislocation movements The volume fraction of the γ' phase is an important parameter to understand the evolution of the precipitate microstructure at high temperature and under stress. Its value at the working temperature of these superalloys is not well known. The solutionizing of the γ' phase occurs above 800°C and it becomes complete at about 1260°C. As γ is a FCC disordered phase and γ' is a FCC ordered phase one way to measure the evolution of the volume fraction up to complete solutionizing is to measure the integrated intensity of a superstructure reflection : the volume fraction of the γ' phase as a function of temperature can be measured as reflections with different parity indices are forbidden for the γ phase and not for the γ' phase.

Using the Triple Crystal Diffractometer of the high energy beam line ID15A, we measured the superstructure reflection (100) as a function of temperature for three AM1 superalloy specimens with different thermo-mechanical history and then different precipitate microstructure. The first specimen referenced as H was homogenized at 1280°C and contains fine spheroidal γ' precipitates. The second specimen referenced as S was annealed at 1050°C during 16 hours after homogenization, it contains cuboidal precipitates of average size 0.35-0.40 μm aligned along the cube direction. For these two specimens, measurements have been done using the two axes mode. The third specimen was creep deformed at 1050°C under 150 MPa with a resultant deformation of ε=1.033%, the γ' precipitates present a raft shape perpendicular to the axis of deformation. In this case we studied the reflections (100) and (001) corresponding respectively to the diffraction of planes parallel and perpendicular to the axis of deformation.

In the case of crept sample the mosaicity was large enough to allow an accurate analysis of three axes mode measurements which permit to studied in the same time the temperature dependence of the lattice parameters.

The diffraction profiles of the fundamental reflection (ZOO) of the crept sample show two well split peaks, each one is related to the diffraction of one phase. In the case of fundamental reflection the structure factor of the γ and γ' phases are very close. Then the relative integrated intensity of the diffraction peak related to the γ' phase gives directly the volume fraction of this phase.

The evolution with temperature of the γ' phase volume fraction is shown in figure 1 for specimens H and S and in figure 2 for the crept sample. The integrated intensity of superstructure reflection was normalized at 200°C, the γ' phase volume fraction value was taken equal to .73 at this temperature.

These first results show that the composition and the order parameter of the γ' phase is not constant in all the temperature range. They depend strongly on the temperature and on the thermo-mechanical history of the specimen. The observed anomalies in the temperature behavior suggest an evolution of the composition and should be explained by the diffusion of different chemical compounds. A deeper analysis is needed to explain these phenomena

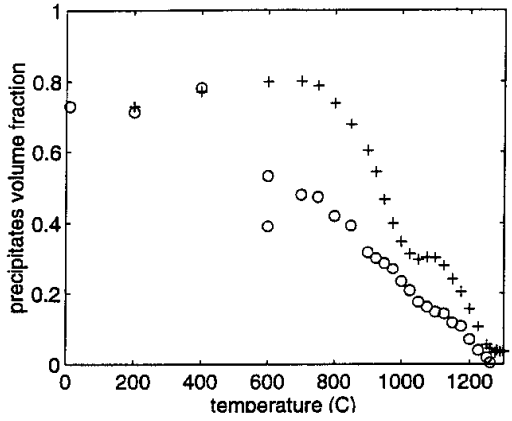

Figure 1: Temperature dependence of the integrated intensity of (100) reflections of specimens H (+) and S (o)

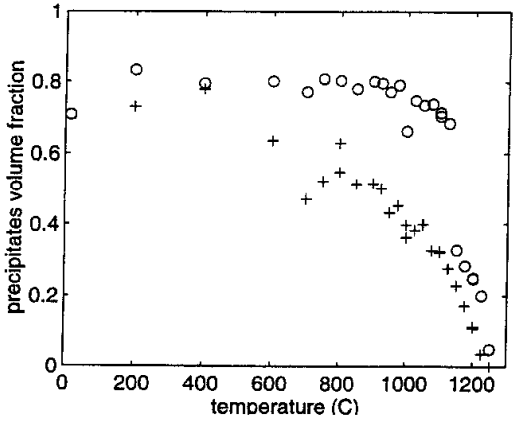

Figure 2: Temperature dependence of the γ' volume fraction obtained from the analysis of (100) superstructure reflection (+) and from (200) fundamental reflection (o).