	Experiment title: Bragg Coherent Diffraction Imaging of coherent nanoprecipitates using superstructure reflections	**Experiment number**: 85467
Beamline: ID01	**Date of experiment**: from: 04/09/2020 to: 08/09/2020	**Date of report**:
Shifts: 12	**Local contact(s)**: Ewen Bellec	*Received at ESRF:*

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- Objective & expected results

Our goal was to demonstrate the ability of Bragg Coherent Diffraction Imaging (BCDI) to measure the shape and strain of an assembly of precipitates using superstructure reflections at the ID01 beamline.
The measurements were carried out on Ni-base superalloys: materials which are designed to resist extreme thermal and mechanical conditions. In this regard an essential factor is their microstructure consisting of a high volume fraction of ordered $L1_2$ precipitates (γ' phase) embedded in a disordered solid-solution FCC matrix (γ phase). Not only the volume fraction, size and distribution of the precipitates but also their shape have a large influence on the mechanical properties of these alloys. It is thus clear that the detailed knowledge of the precipitate shape ,and strain can provide very valuable quantitative data which can be confronted to microstructural modeling (atomic scale simulations, finite element and phase field method).
The good spatial resolution and very high strain sensitivity of BCDI make it the only one suitable to image the 3D strain field of the nanoprecipitates. Additionally, such measurement would be the first experimental demonstration of BCDI on coherent precipitates using a superstructure reflection. These new capabilities could open the door to BCDI as a microscopy tool for studying complex real-world materials.

Results and the conclusions of the study

We intended to measure two distinct microstructures. The first one, M3 consisted of small coherent γ' nanoprecipitates (200 nm, Fig. 1c) while in the second one, M2 the precipitates were much larger (1-2 μm, Fig. 1b) and incoherent. Both microstructures were prepared by mechanical polishing and chemical etching. Focused

Ion Beam (FIB) milling was then employed to prepare two lamellae for each microstructure: a large one: 10x3x1 µm and a much smaller one ~1.5x1.5x1 µm (2x2x2 µm for the M3 microstructure). The lamellae were then attached to two TEM copper grids (one for each microstructure, Fig. 1d).
The lattice orientation of the samples was determined by micro-Laue X-ray diffraction on the BM32 beamline. The indexation of the Laue patterns turned out to be quite challenging due to the small size of the lamellae, and the overlap with the peaks from the Cu grid. In addition; the beam was significantly larger than the typical beam size used on BM32 (2 µm vs 0.5 µm). Therefore we only managed to index reliably one of the samples, *i.e* the large TEM lamella with the M3 microstructure.

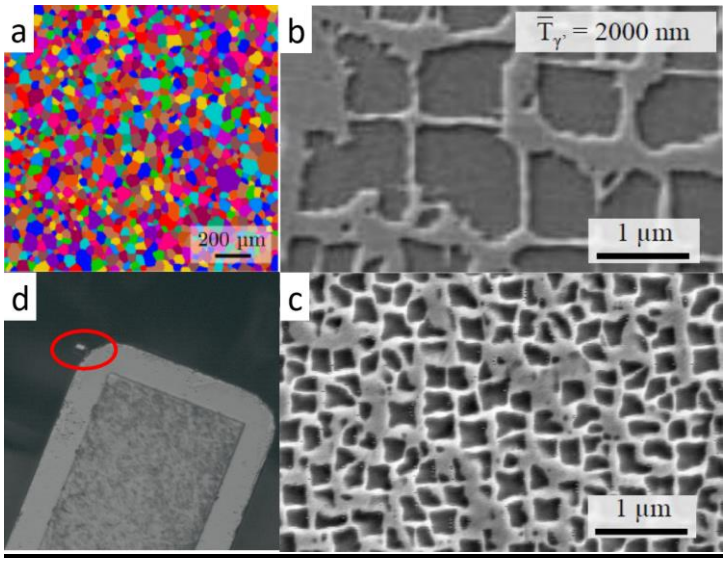

Figure 1 a) EBSD map of the M2 microstructure (average grain size ~ 40 µm) b) –c) SEM image of the M2 and M3 microstructures revealing the average precipitate size for the 2 microstructures

At the ID01 beamline, the 9 keV beam was focused down to about 400x400 nm using KB-mirrors. The lamella was located by using the ID01 microscope together with the fluorescence of the Ni edge at 9 keV. Using the orientation matrix obtained from BM32 we managed to align a total of 11 Bragg reflections: 2 fundamental (2 0 0 and 3 1 1) and 9 superstructure (1 1 0, 2 -1 -1, 2 1 1, 2 0 1, 2 0 -1, 2 1 -1, 3 0 0, 3 0 -1 and 3 0 1). Given the large size of the precipitates, the signal of the superstructure was quite intense, proving the ability of the setup to measure such reflections.
We first tried to find isolated precipitates and measure them using superstructure reflections. This turned out to be quite challenging given the beam size and the large volume fraction. We considered several candidates and acquired Coherent X-ray Diffraction patterns at the 1 1 0 superstructure reflections (Fig. 2).

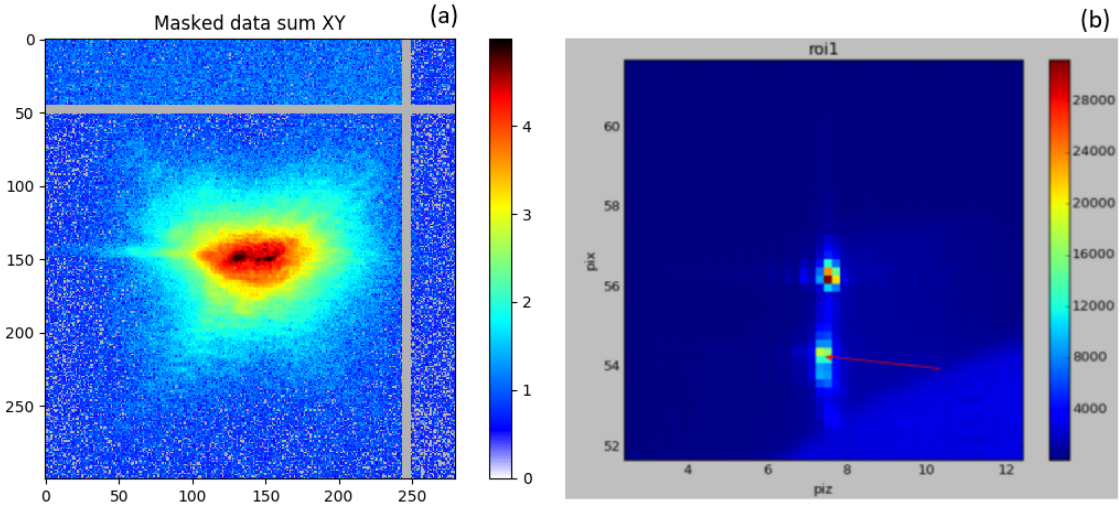

Figure 2 a) Coherent X-ray diffraction pattern from the 1 1 0 superstructure reflection b) KMAP showing the location of the precipitate in lamella (designated by a red arrow)

We then decided to change our measurement strategy and perform scanning X-ray diffraction microscopy on the sample, after reducing the beam size to 250x250nm. We used overlapping positions such that Bragg

ptychography could be used to improve the spatial resolution and recover the 3D strain field from several reflections. We selected the 6 reflections (2 fundamental and 4 superstructure reflections) and carried out the measurements over night. For each reflection we collected 60x60 points over an area of 10x10 microns for 20 angular steps (step size of 167 nm). This represents a measurement time of approximately 2 hours per reflection with some additional time required to realign the sample when changing the reflection.
Fig 3 shows the distribution of the maxima of intensity in the lanella for several Bragg reflections:

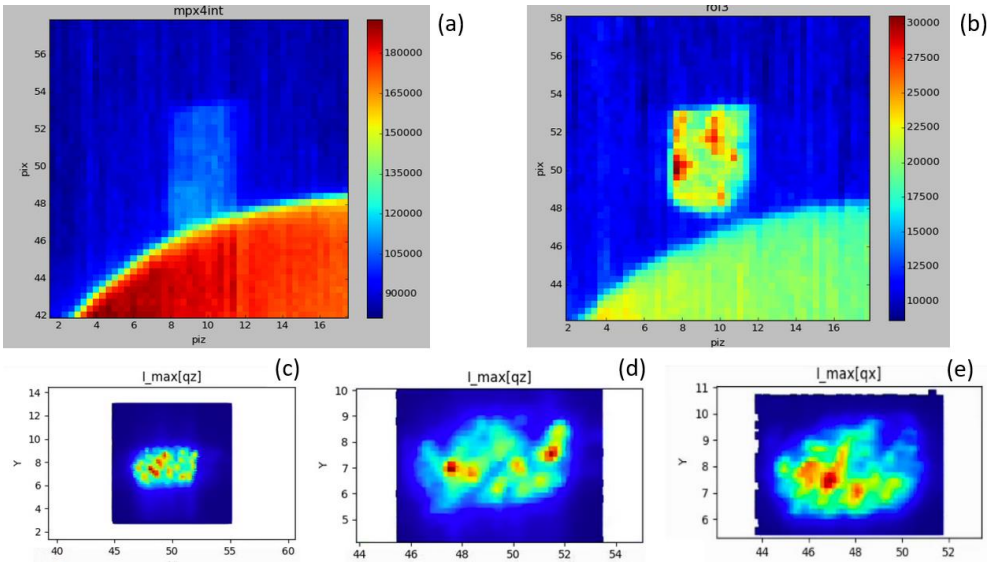

Figure 3 a) Fluorescence map at 9 keV b –e) Maximum intensity as a function of the sample position for **g** = 2 0 0 (b-c), g = 2 0 -1 (d) and g = 2 -1 -1 (e)

One can observe that the integrated intensity is less homogeneous for the superstructure reflections (Fig.3 d-e° than for the fundamental (Fig. 3c) which would tend to indicate that the distribution of the γ' precipitates is not completely homogeneous.

- Justification and comments about the use of beam time

We succesfully managed to measure a large number of fundamental and superstructure reflections using scanning X-ray diffraction microscopy. The reconstruction of the BCDI data turns out to be challenging and would probably require a sample with a lower volume fraction of precipitates. For such complex microstructure, scanning X-ray diffraction and 3D Bragg ptychography are more suitable and we aim at use these techniques to study the evolution of the strain field during formation and dissolution of tertiary γ' precipitates in a follow up proposal. The analysis of the data is still ongoing .The first step will be to extract the tilt and strain from the collected 3D reciprocal space maps. The large number of collected reflections should allow to extract the full strain tensor. Subsequently, we will try to reconstruct the 3D strain distribution in the sample using Bragg ptychography.