EUROPEAN SYNCHROTRON RADIATION FACILITY

ESRF User Office

BP 220, F-38043 GRENOBLE CEDEX, France

Delivery address: 6 rue Jules Horowitz, 38043 GRENOBLE, France

Tel: +33 (0)4 7688 2552; fax: +33 (0)4 7688 2020; email: useroff@esrf.fr; web: http://www.esrf.fr

Evolution of the intragranular stress during a tensile test using the Kossel microdiffraction technique

Raphaël Pesci, Denis Bouscaud, Christian Perca, Marc Wary, Sophie Berveiller, Manfred Burghammer

The goal of these experiments was to follow the evolution of intragranular strain and stress in two materials (CuAlBe shape memory alloy and superalloy) through Kossel microdiffraction, during in situ tensile tests. To that effect, flat specimens (60mm long, 4mm wide) were stressed with a small 5kN tensile machine directly installed on the beamline. A CCD camera was put as close to the samples as possible in order to collect a maximum number of Kossel lines and to make the analyses as swift as possible. In addition, energy levels beyond the absorption edge of the materials were preferred (9.2 keV, with a beam size of about 3µm) so as to intensify the X fluorescence (maximum fluorescence).
Many results were obtained, only a part of which are presented here.

Results

1. Superalloy single crystals

Several tests were first performed to check that Kossel patterns were easy to come by with this material. To improve the quality of the obtained Kossel line patterns, an average of various Kossel acquisitions was realized, either with a flat field correction or with a background subtraction (figure 1). The aim was to find out which procedure yielded the most “beautiful” Kossel lines, i.e. those that would be easiest to process (line profiles) to determine thereafter the crystallographic orientation and the strain-stress state. In both cases Kossel lines and circles of good quality were obtained, all well defined, and presenting clean-cut intersections.

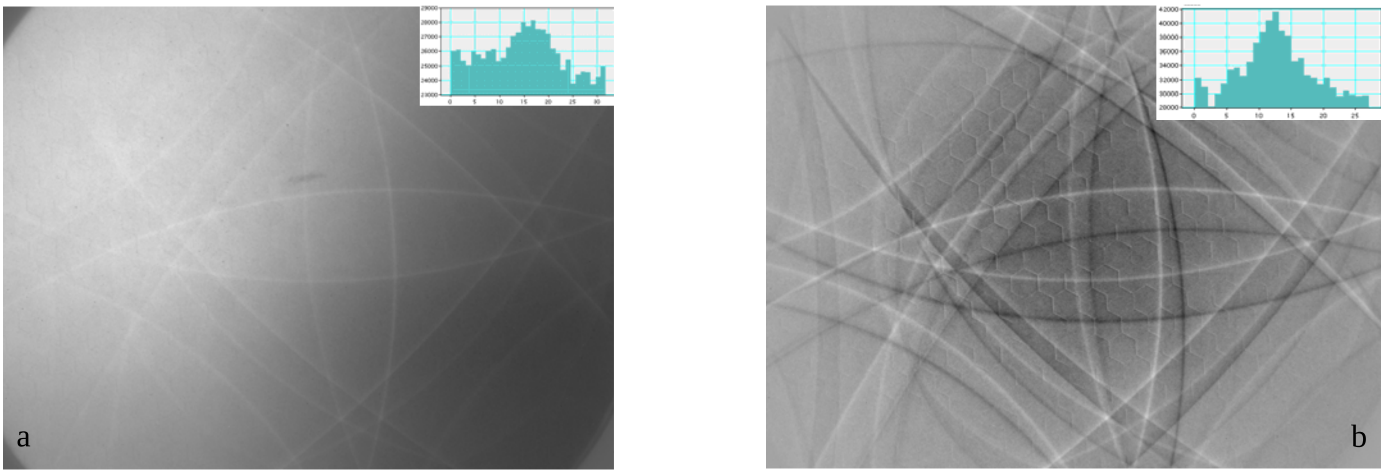

Figure 1 - Kossel pattern on an superalloy single crystal specimen
a) with a flat field correction - b) with a background subtraction

Tensile tests were then carried out to follow the evolution of strain and stress under loading. The goal was to establish a comparison with what would be found through Kossel microdiffraction with the applied stress, since single crystals were being investigated.
Several analyses were therefore performed during loading, both in the elastic range (the single crystals in question have a yield stress of about 1000MPa) and in the plastic range. Only the example of a sample in its initial state and with an applied stress of 820MPa is here presented. The various obtained patterns were first superimposed - always with a flat field correction or a background subtraction - to see if lines could be seen moving under stress (figure 2).

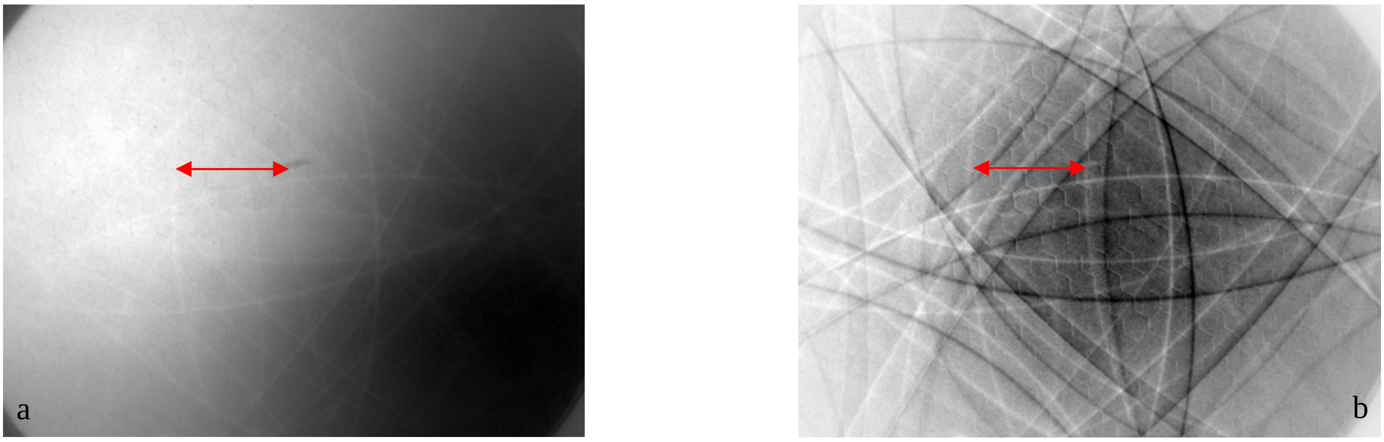

Figure 2 - Two Kossel patterns superimposed (initial state and with a 820MPa applied stress)
a) with a flat field correction - b) with a background subtraction

The Kossel line pattern obtained for an applied stress of 820MPa was then indexed using a software (of our own making) that specifically takes into account the distance between the center of projection on the material and the center of the screen, the wavelength of the emitted X-rays, as well as the crystalline structure and the lattice parameter of the tested material. The strain tensor in the crystal coordinates was calculated from this pattern; the final stress state calculated in the specimen coordinates remains to be compared to the applied stress of 820MPa.

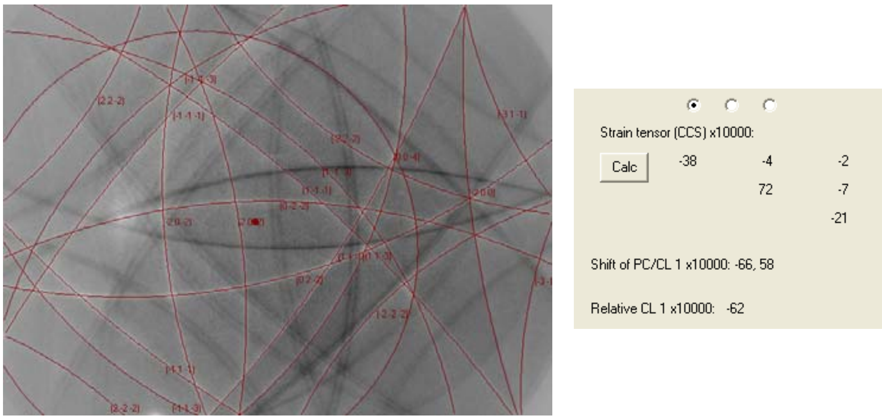

Figure 3 - Kossel pattern (820MPa applied stress) fully indexed and the corresponding strain tensor in the crystal coordinates

2. CuAlBe polycrystalline specimens

Several tests were also performed on CuAlBe polycrystalline specimens with coarse grains (size: 1-5mm); only the background subtraction approach was worked with in this instance, because it yielded more satisfying results regarding line profiles.
The evolution of the strain and stress states in two different grains was thus followed in the same way as previously, in order to gauge the influence of crystallographic orientation on behaviour.

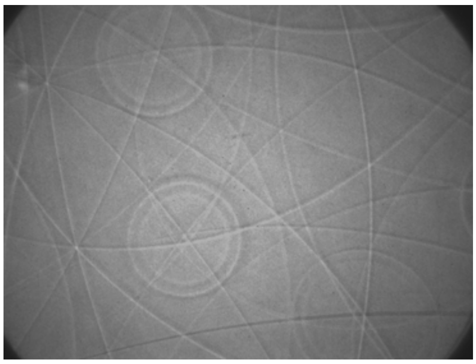

Figure 4 - Kossel pattern on a CuAlBe specimen

Prospects

It is thus possible to obtain some really high quality Kossel line patterns through synchrotron emission, in order to determine the strain and stress states inside grains.
It is important to go one step further now by making the first cartography of one whole grain in order to show the intragranular heterogeneity, especially near grain boundaries. It is also important to show that it is possible to obtain Kossel patterns with a smaller beam size (100 to 200nm: analysis of nanomaterials and microelectronics components, for example), and for materials with high wavelength - like aluminium (pseudo-Kossel technique). Two new proposals will soon be submitted in this regard.