Standard Project

Experimental Report

Proposal title: Influence of shot-preening, grain size and microstructure on residual stresses in a nickel-based superalloy		**Proposal number:** 20140989
Beamline: BM32	**Date(s) of experiment:** from: 2015-04-08 to: 2015-04-13	**Date of report:** 2015-11-22
Shifts: 15	**Local contact(s):** O. Robach	*Date of submission:* *2014-09-12*

Objective & expected results (less than 10 lines):

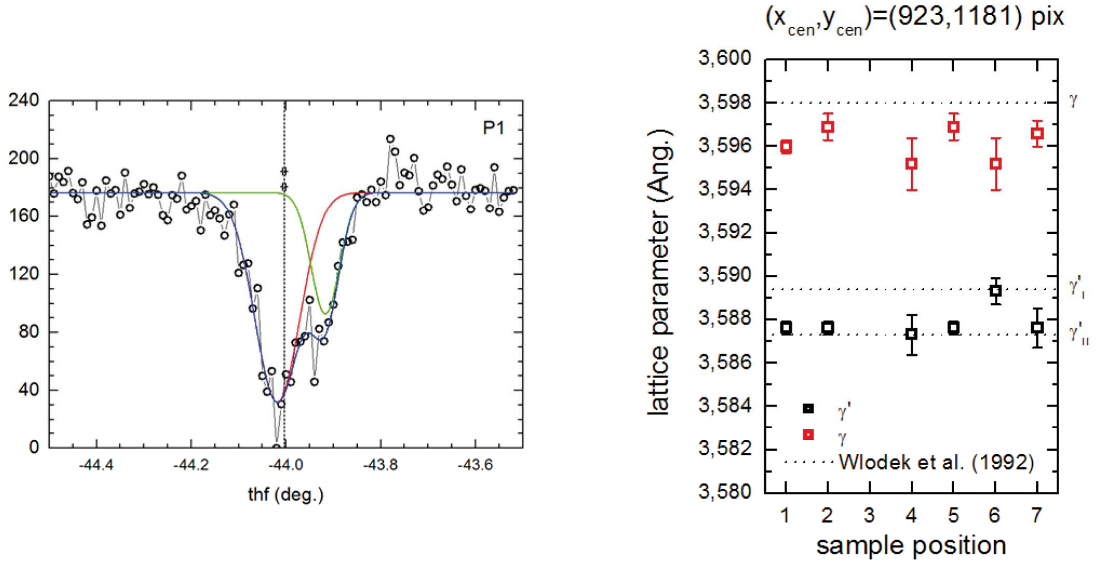
The fatigue properties of turbine disks of aircraft engines are improved by shot-peening. The compressive residual stresses introduced in the surface layer by the plastic deformation tend to reduce cracks initiation and propagation. In this study, we want to examine the connections between residual stresses caused by shot-peening, the mechanical behavior and the fatigue life time of a N18 superalloy for different types of microstructures. The aim of this experiment is to map local strains, grain orientations and dislocation densities using the micro-Laue technique in the section of fatigue test specimen. With the submicronic beam size, the orientation of the crystal and the components of the deviatoric strain/stress tensors are obtained at different length scales: from grain to grain with a large mapping and inside grains with a fine mapping. The "rainbow" method implemented in 2013 at BM32 [1] is assessed to determine the lattice parameters of the γ and γ' phases at specific sample locations and calculate full strain/stress tensor components. Some samples are prepared post mortem from specimen subjected to shot-peening, isothermal ageing at 450°C or fatigue tests at 450°C interrupted at the half lifetime in order to investigate the stress relaxations.

Results and the conclusions of the study :

During the experiment, 2 types of microstructures with a 40 µm average grain size have been investigated : microstructrures denoted M1 and M2 in the following have 200 nm and 2 000 nm γ' precipitate sizes respectively. The thermomechanical treatments applied to the investigated microstructures are the following:(a) no treatment, (b) 300 cycles fatigue tests at 450°C. (c) shot-peening (d) shot-peening +1h40 isothermal ageing at 450°C (e) shot-peening + 300 cycles fatigue test at 450°C.

1) γ/γ' phase sensibility and full stress tensor determination with the "Rainbow" method [1]

All Laue patterns exhibit fundamental spots associated with the γ and γ' phases as well as superstructure spots associated with the γ' phase. The incident beam energy has been reduced to 5-17keV instead of 5-22 keV to reduce the number of reflections at the same spot position and the beam penetration depth. The data analysis (grain orientation, cell shape and deviatoric stress tensor components) is realized with the Laue Tools and Xmas softwares. The analysis is easier for superstructures reflections since there is no overlapping of γ and γ' contributions. For the sample M2(a), we clearly observe some spatial fluctuations of the composition from the fluorescence signal close to Ni,Ti, or Al edges. To determine all cell parameters at specific locations in a given grain, the rainbow method is used. After a calibration work, the diffraction of a given reflection of a diamond single crystal by the incident beam cause the attenuation of given sample spots and thus enables to determine their energy. As example, **figure 1a** represents the integrated intensity of a fundamental spot during the rotation of the diamont single crystal close to a Bragg angle. We assume that the attenuation profile corresponds to the convolution of γ and γ' contributions. The lattice parameters of the two phases (cubic approximation) obtained at different sample positions from the preliminary analysis of such profile are represented in **figure 1b**. Values for the γ' phase are close to those given by Wlodek et al. (1992) corresponding to unstrained lattice parameters (dotted lines). The γ phase is more strained.



(a) (b)

Figure 1: (a) integrated intensity of a fundamental spot as a function of the diamond crystal rotation angle. Red and green lines correspond to Gaussian fittings of local minima (b) γ and γ' lattice parameters obtained from the analysis of the intensity attenuation recorded at different sample positions. Results are compared to values given by Wlodek for unstrained phases.

2) Deviatoric stress maps of samples subjected to different thermomechanical treatments.

During the beam time, Laue patterns are recorded at different sample positions in order to determine deviatoric strains and crystal orientations (4 spots analysis) inside grains and from grain to grain. As example, grain orientations (ψ Euler angle) and stress (σ Von Mises) maps obtained for the microstructure M1 and corresponding to thermomechanical treatments denoted previously by (a) and (b) are shown in **figure 2**. Stresses of 200-300 MPa are homogeneously distributed in the microstructure for the reference sample (Fig. 2a). By contrast, after a fatigue test at 450°C, 400-700 MPa Von Mises stresses are mainly localized at grain boundary and are the signature of accumulated plasticity (Fig. 2b).

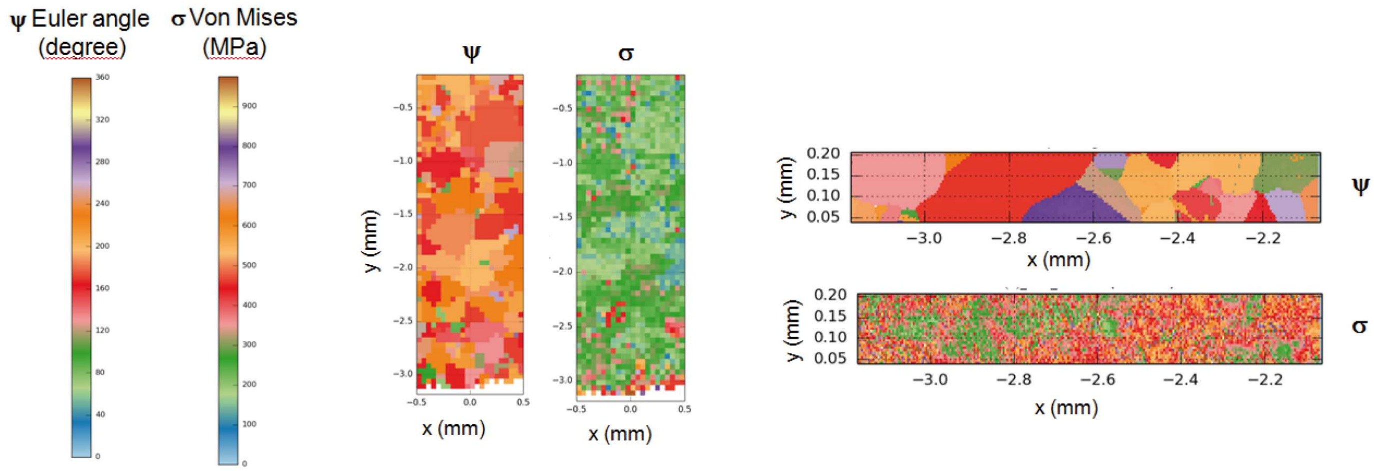


Figure 2 : Orientation and Von Mises stress maps determined by the micro-Laue technique in (a) a reference sample and (b) in a sample which have been subjected to fatigue test at 450°C.

After shot-peening, the stress field is inhomogeneous with amplitudes close to the elastic limit (1200 MPa) of the material at the sample surface (see **figure 3**). Due to strong crystal distortions, the analysis of Laue patterns recorded close to the sample surface is not possible. In figure 3d, we observe that stress amplitudes near the sample center are of the same order of magnitude than those recorded in reference samples.

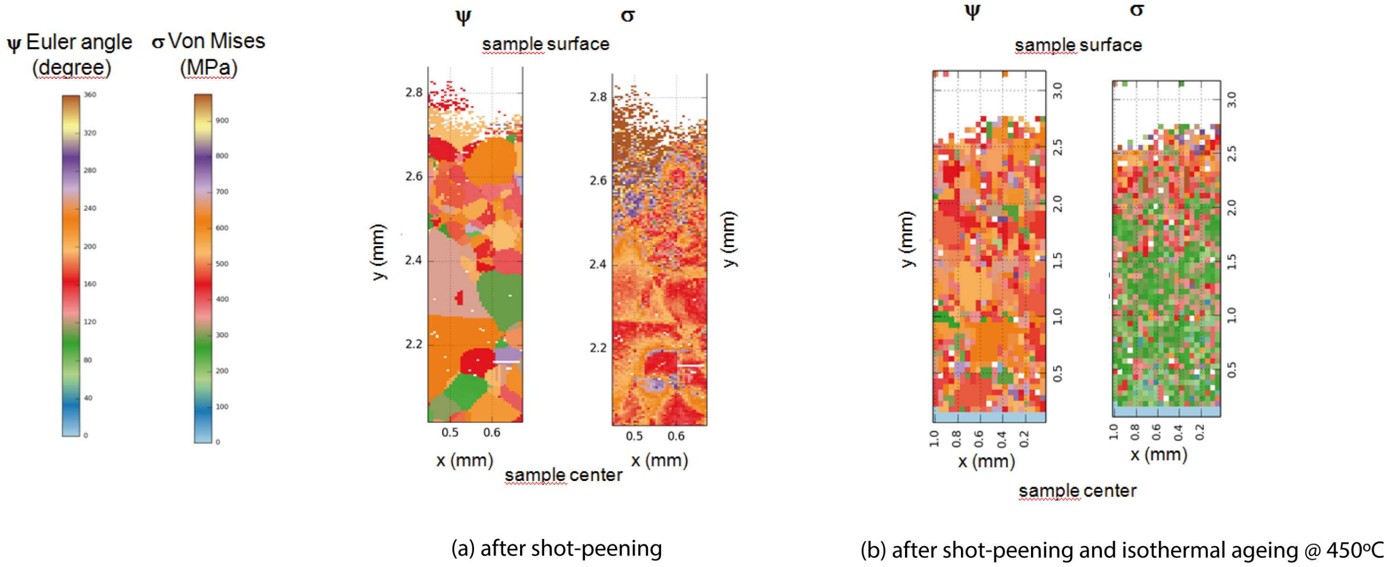


Figure 3 : Orientation and Von Mises stress maps determined by the micro-Laue technique in (a) a shot-peened sample and (b) in a sample which have been subjected to shot-preening and isothermal ageing at 450°C.

3) Conclusion

- Data obtained during this experiment show us how different alloy microstructures react to thermomechanical loadings especially to shot-peening. Deeper analysis of rotation gradients should give us estimate of geometrically necessary dislocations densities. Using averaging, stress profiles with respect to the distance from the surface to the center of the sample will be obtained.
- The rainbow method has been assessed in a nickel based superalloy. Its sensibility seems to be good enough to separate the γ and γ' phases contributions.
- For the microstructures investigated here (40 µm < average grain size < 80 µm), the use of the micro-Laue technique is the only way to obtain quantitative values of strains at the micrometer length scale. The laboratory methods such as the sin^{2}y or the Ortner method are ineffective.

[1] O. Robach O, J.-S. Micha, O. Ulrich, O. Geaymond, O. Sicardy O, J. Härtwig and F. Rieutord, Acta Crys. A. 69 (2013) 164-70

Justification and comments about the use of beam time (5 lines max.):

The micro-Laue technique is well suited for strain measurements in nickel bases superalloy with grain sizes larger than the beam penetration (~20 µm). The rainbow method implemented in BM32 is a great technique which enables to have all crystal cell parameters and thus all the components of stress tensors without removing the sample to change the incident beam wavelength. In the surface layer strongly affected by shot-peening, the Laue technique is ineffective to determine stress levels.

Publication(s):