



	<b>Experiment title:</b> <i>In situ</i> nanodamage and strain interactions: three-dimensional multiscale <i>in situ</i> laminography experiments and simulations	<b>Experiment number:</b> ma3777
<b>Beamline:</b> ID16b	<b>Date of experiment:</b> from: 08/12/2017 to: 13/12/2017	<b>Date of report:</b> 02/20
<b>Shifts:</b> 15	<b>Local contact(s):</b> Jussi-Petteri Suuronen	<i>Received at ESRF:</i>
<b>Names and affiliations of applicants</b> (* indicates experimentalists): <b>*Dr. Thilo Morgeneyer, Mines ParisTech</b> <b>*Dr. Lukas Helfen, KIT, ESRF</b> *Dr. Heikki Suhonen, University of Helsinki * Andrei Shkarin, <b>KIT</b> * Simon Haaga, <b>KIT</b> * Dr. Daniel Pino Munoz, Mines ParisTech Daniel Hänschke , <b>KIT</b>		

### Report:

In the frame of ma3777 we imaged *in situ* the behavior of thin, notched Al sheets under mechanical load via X-ray nanolaminography at ID16B. This reveals the mechanisms of void nucleation up to crack propagation in the investigated materials for high stress triaxiality conditions ahead of a severe notch.

1. Low-resolution laminography scans have been acquired successfully for 2198T8R- and 2196Mn Al alloys *in situ* at different loading steps up to the failure of the material. At every loading step for each specimen a low resolution scan has been acquired with 240 nm voxel size.
2. Fast reconstruction of the acquired projection data was performed via single-distance phase retrieval [1, 2, 3] (integrated in our reconstruction library and pipeline (UFO-KIT) available as open-source code [4,5]) which enabled us to precisely select a number of ROIs for high-resolution imaging.
3. Based on the reconstructed 3D data of the low resolution scans, particular Region of Interests (ROIs) have been selected to be investigated into more detail. In subsequent high-resolution scans with 100nm effective pixel size at different ROIs, the microstructure evolution could be observed on-line at such spatial resolutions.
4. Additionally, we acquired correlative surface data via visible light microscopy. This will provide macroscopic surface information on local loading conditions.

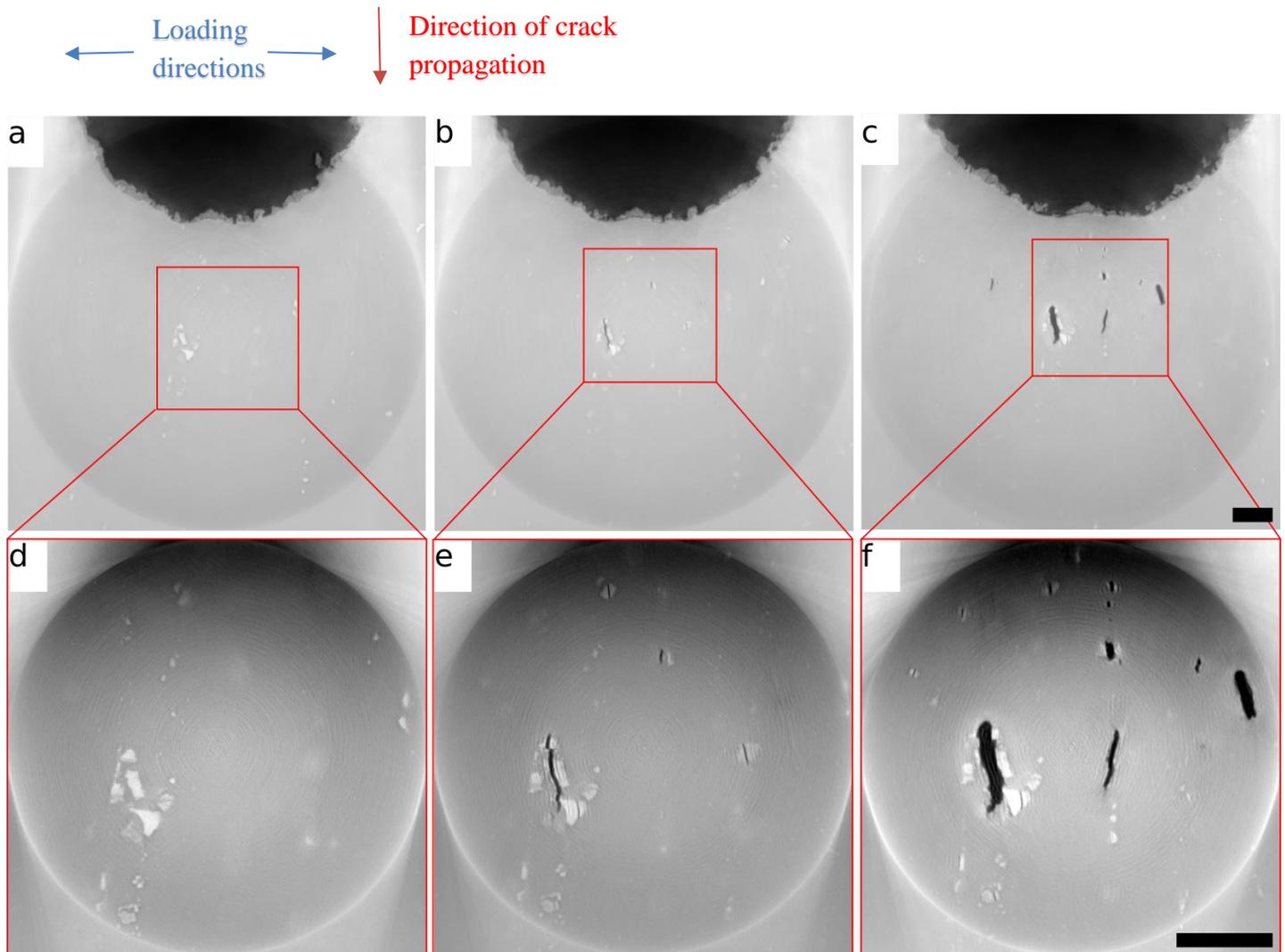
We made a test of one additional scan with 25nm pixel size, but the resolution for this particular scan is not significantly increased probably due to the sphere of confusion (excentricity and wobble) of the current nanolaminography setup (mechanical bearing specified at ~ 100 nm run-out).

All data has been processed using a newly developed single-distance phase retrieval algorithm for cone beam projection microscopy (adopted version of quasiparticle phase retrieval [1, 2, 3]) and laminography reconstruction. Before reconstruction the phase retrieval parameters have been carefully optimized. The image quality has been compared to multi distance phase retrieval acquired during ma2787. It turned out that single-distance artefacts can be accepted in order to achieve a higher throughput or aiming towards higher

temporal resolution. These artefacts and the worse signal-to-noise ration observed for single-distance measurements we expect to improve strongly, due to increased flux delivered by ID16b after the EBS upgrade. During ma3777 still multi distance scans have been acquired, since the impact of single-distance phase retrieval was not investigated clear enough at this point of time.

A publication dedicated to single-distance phase retrieval for cone-beam based microscopy for *in situ* 3D imaging is in preparation [2, 3]. A patent for the dedicated in situ loading device [6] submitted through the CNRS is pending.

The 3D images obtained allow for the evaluation of the distribution and number of secondary phase particles. Furthermore, the mechanisms of crack initiation (e.g. delamination, particle cracking) in the vicinity of such particles can be observed *in situ* and described which is shown in Fig. 1 for the case of Mn-containing 2196 aluminium alloy. These information will be used in our future finite-element analysis modeling efforts to predict damage initiation and progression under various stress triaxiality and stress level conditions [7,8,9,10].



*Figure 1 Hierarchical single-distance laminography scans for the case of a 2196 Mn-containing aluminium alloy. (a)-(c) Overview scans showing the damage with respect to the notch tip position: (a) Distribution of secondary particles (notch opening  $d=0.19$  mm), (b) crack initiation at secondary particles (notch opening  $d=0.47$ mm), (c) early stage of crack propagation (notch opening  $d=0.71$ mm). (d)-(f) High resolution scans showing the chosen ROI (determined based on the former low resolution scans) at 100 nm voxel size allowing novel insight into the damage mechanisms. Scale bars correspond to 50  $\mu$ m.*

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