ESRF	**Experiment title:** Tomographic study on self healing of creep damage by nanoscale precipitation in Fe-Au alloys	**Experiment number**: MA2210
Beamline:	**Date of experiment**: from: 13-06-2015 to: 15-06-2015	**Date of report**: 09-09-2016
Shifts:	**Local contact(s)**: Yang Yang, Peter Cloetens	*Received at ESRF:*

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Report:

Self-healing of deformation damage is a promising new approach to prolong the lifetime of Fe-based alloys for high temperature structural applications. It has been proposed that creep cavities can be healed by the formation precipitates at the cavity surface. We have recently demonstrated that self healing of creep damage can indeed be achieved by gold precipitation in Fe-Au alloys by combined creep tests and electron microscopy studies of the 2D microstructure [1]. To explore the healing mechanism in detail nano-imaging tomography of the cavities (damage) and Au precipitates (healing) was performed in creep loaded Fe-Au alloys for varying applied creep loads. The experimental aim was to resolve the size, shape and spatial correlations between cavities and Au precipitates. A unique insight in the site-specific autonomous repair mechanism is achieved by a direct 3D characterization of the nano- and micro-scale cavities and precipitates.

Four different creep-failed Fe-Au alloy samples were studied, which were subjected to varying applied loads (60, 80, 100 and 117 MPa) at a temperature of 550 ^{o}C prior to the tomography experiments. This allows us to compare the 3D structure between the efficient healing (low load) and inefficient healing conditions (high load). A high energy of 33.6 keV was chosen in this experiment to optimize the transmission through the samples. Besides the benefits of the ability to perform nano-tomography with a high energy, the magnifying geometry of the cone beam allows the switch between a large field of view (FOV) with 100 nm voxel size, and a finer FOV at a voxel size of 25 nm. As shown in **Fig. 1**, a sample was placed downstream of the focus and magnified radiographs were recorded onto an X-ray detector using a FReLoN charged-coupled device (CCD) with a 2048×2048 binned pixels array. For one tomography scan, 1500 projections were acquired with an exposure time of 1.00 s for a resolution of 100 nm and 1.25 s for the higher resolutions of 25 and 50 nm. Tomographies at four different focus-to-sample distances were acquired to complete one holotomography scan, which were subsequently used for phase retrieval. The 2D phase maps retrieved from the angular projections were then used as input for a tomographic reconstruction based on the filtered back projection (FBP) algorithm method (ESRF PyHST software package). The reconstructed 3D volumes were visualized and rendered with 3D visualization software Avizo 8.1 (FEI).

A region of interest shown in **Fig. 2** is extracted from the creep-failed sample at 80 MPa and 550 °C. By projecting this microstructure from different angles, it is found that the positions of the precipitates clearly delineate the grain boundaries. This is in agreement with the previous SEM studies which showed that the precipitates in Fe-Au alloy are formed exclusively at grain boundary and at pre-formed cavities. With the grain-boundary positions identified, it is found that only the grain-boundary cavities are able to grow into larger sizes. A combination of three parameters, i.e. flatness, elongation and complexity, was proposed to quantitatively classify the particle shapes. Based on the shape classification, we found that the particles formed at different locations have different shapes: particles dispersively distributed on the grain boundaries and within the matrix are mainly spherical or equiaxed, particles on the inclined grain boundaries are more elongated or flattened, while particles located on the grain junctions have a relatively complicated shape. From this study the self-healing character of added Au in Fe-based alloys is fully supported and the degree of filling for each cavity is determined quantitatively [2].

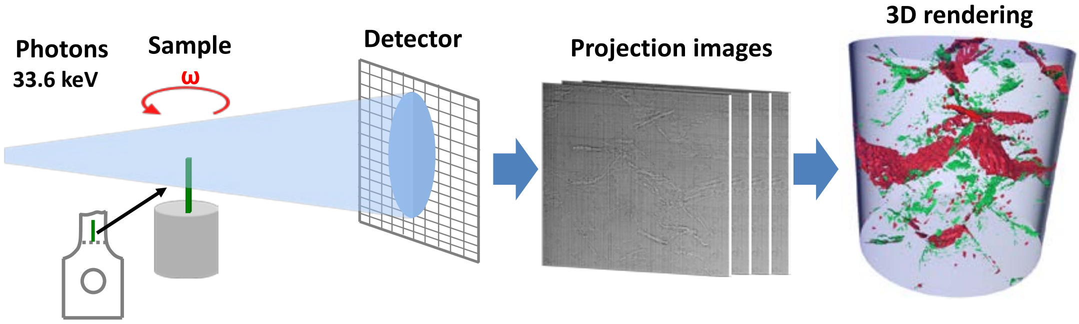

Fig. 1. Schematic diagram of the synchrotron X-ray holotomography imaging process. In the 3D rendering the precipitates and cavities are labelled in green and red, respectively, while the iron matrix is light grey.

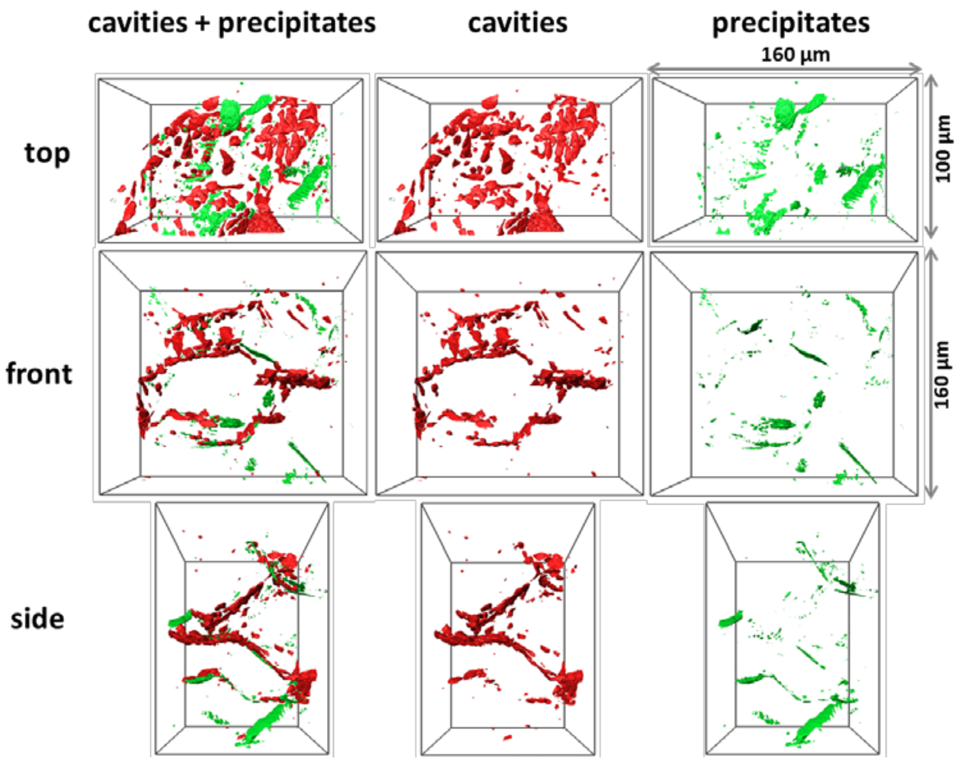

Fig. 2. (a) Spatial distribution of cavities and precipitates in a region of interest of the Fe-Au sample after creep at 550 °C and 60 MPa. The applied stress is normal to the top view.

References

[1] S. Zhang, C. Kwakernaak, W. G. Sloof, E. Brück, S. van der Zwaag, N.H. van Dijk, *Self healing of creep damage by gold precipitation in iron alloys*, Advanced Engineering Materials 17 (2015) 598-603.

[2] H. Fang, C.D. Versteylen, S. Zhang, Y. Yang, P. Cloetens, D. Ngan-Tillard, E. Brück, S. van der Zwaag, N.H. van Dijk, *Autonomous filling of creep cavities in Fe-Au alloys studied by synchrotron X-ray nano-tomography*, submitted to Acta Materialia.