ESRF	**Experiment title:** Self healing of creep damage by nanoscale precipitation in Fe-W alloys studied by nano X-ray tomography	**Experiment number**: MA3497
Beamline: ID16A	**Date of experiment**: from: 14-07-2017 to: 17-07-2017	**Date of report**: 12-02-2020
Shifts: 9	**Local contact(s)**: 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 precipitation in Fe-Au, Fe-Mo and Fe-W alloys. Nano-imaging tomography was previously performed on Fe-Au alloys subjected to high-temperature creep at varying applied loads to monitor the cavities (damage) and Au precipitates (healing) [1]. A unique insight in the site-specific autonomous repair mechanism was achieved by a direct 3D characterization of the nano- and micro-scale cavities and precipitates.

For the present nano-tomography experiment on instrument ID16A [2] we studied the technologically more relevant binary Fe-W alloy (1 at.% W) system, which was subjected to creep for two different stress levels at a temperature of 550 ^{o}C prior to the tomography experiments. The samples were interrupted at different stages of their creep life time (25%, 50%, 75% and 100%). The creep-damaged samples contain three phases: (i) Fe-W matrix, (ii) unfilled creep cavities (about 1 μm in size) and (iii) nanosized Fe_2W precipitation inside the creep cavities. 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 30 nm.

The samples were 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 1.0 s, for resolutions of 100 and 30 nm. Tomography data 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. 1** is extracted from the creep-failed sample subjected to creep at a stress of 160 MPa and a temperature of 550 ºC. The figure shows 4 different partially fille cavities of distinctly different sizes for 3 different projections. The unfilled cavity is shown in red and the precipitation inside the creep cavity is shown in green. Often more than one precipitate nucleate on the creep cavity surface of a single creep cavity. This study clearly demonstates how creep damage in binary Fe-W alloys is healed by the formation of nanoscale Fe_2W precipitates inside the creep cavities. The number of precipitates within one cavity and the degree of filling was obtained for a large collection of individual creep cavities and analysed quantitatively [2]. Two clearly different types of behaviour are observed for isolated and linked cavities, where the isolated cavities can be filled completely, while the linked cavities continue to grow. The demonstrated selfhealing potential of tungsten in iron-based metal alloys provides a new perspective on the role of W in high-temperature creep-resistant steels.

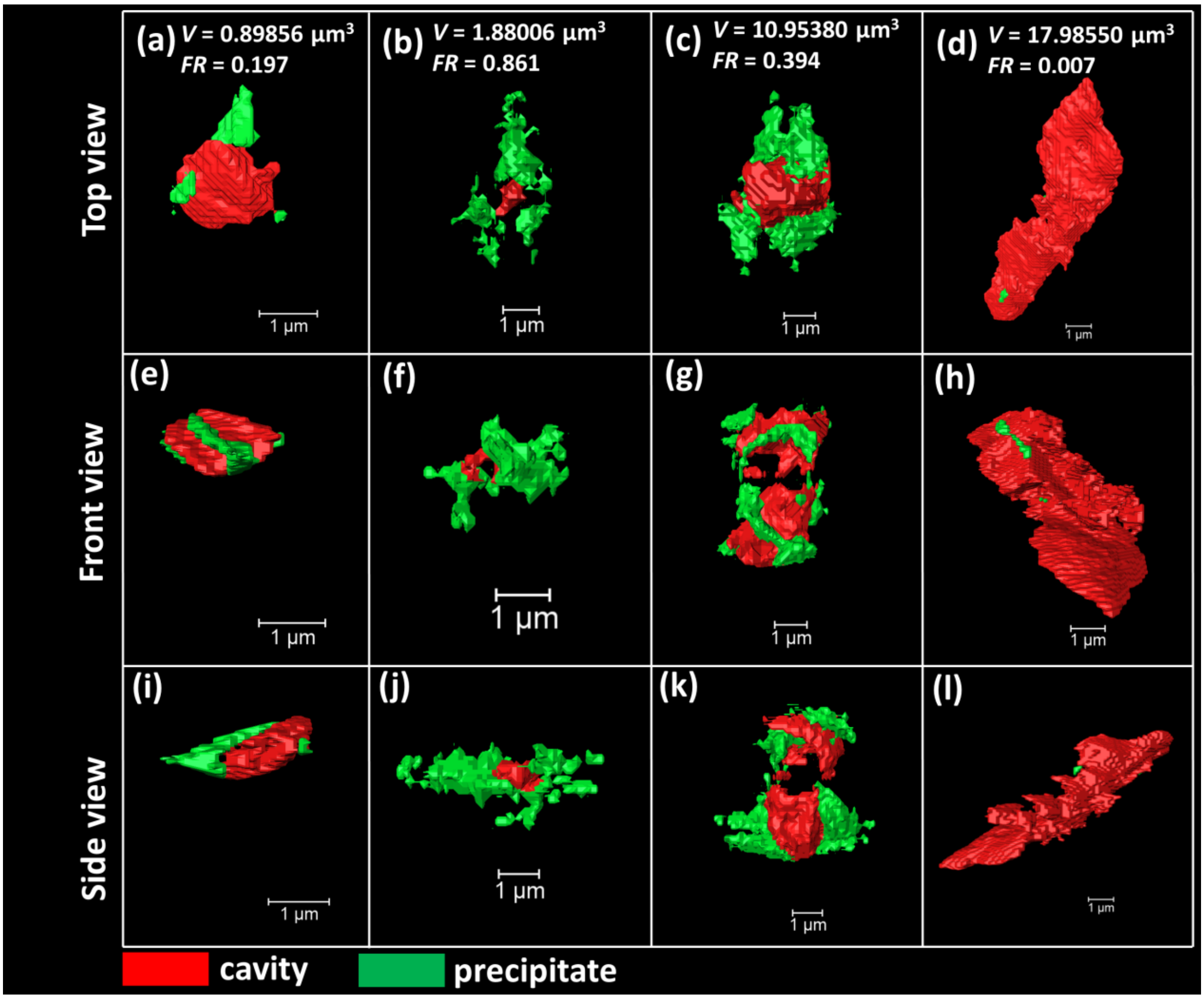

Fig. 1. Partially filled cavities in the Fe-W alloy sample after creep at a temperature of 550 ºC and a stress of 160 MPa up at rupture viewed from different angles and obtained for a voxel size of 30 nm [2]. The applied stress was normal to the top view. The unfilled cavity is in red and the precipitate filling of the cavity in green. Indicated are the volume of the partially filled cavity and the filling ration (FR).

References

[1] 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*, Acta Materialia 121 (2016) 352-364.

[2] H. Fang, N. Szymanski, C.D. Versteylen, P. Cloetens, C. Kwakernaak, W.G. Sloof, F.D. Tichelaar, S. Balachandran, M. Herbig, E. Brück, S. van der Zwaag, N.H. van Dijk, *Self healing of creep damage in iron-based alloys by supersaturated tungsten*, Acta Materialia 166 (2019) 531-542.