ESRF	**Experiment title:** Self healing of creep damage by nanoscale precipitation in ternary Fe-Au-W alloys studied by nano X-ray tomography	**Experiment number**: MA-4629
Beamline: ID16A	**Date of experiment**: from: 01-04-2021 to: 04-04-2021	**Date of report**: 09-09-2022
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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. We recently demonstrated that efficient healing of creep damage can be achieved in binary Fe-Au [1] and Fe-W [2] alloys by precipitation of Au and Fe_2W inside creep cavities. In order to reach a superior performance ternary Fe-Au-W alloys are investigated, where both healing mechanisms operate simultaneously. Nano-tomography experiments were performed to reveal the 3D morphology of cavities (damage) together with the synergy or competition between both types of precipitation (healing). The 3D characterization of the nanoscale cavities and the precipitates forming inside the cavities was shown to provide a unique insight in the autonomous repair mechanism in complex alloy systems.

For the present nano-tomography experiment on instrument ID16A [3] we studied a ternary Fe-Au-W alloy (1 at.% Au and 1 at.% W), where the Au and W were initially fully dissoved in the matrix. The alloy was subjected to creep for a fixed stress level at a temperature of 550 ºC prior to the tomography experiments. The samples were interrupted at different stages of their creep life time. The creep-damaged samples contain four phases: (i) Fe-Au-W matrix, (ii) unfilled creep cavities (about 1 μm in size) with nanosized (iii) Au-rich precipitations and (iv) W-rich 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.

A series of 2D X-ray projections (in total 1800 projections for each scan) were recorded as the sample rotated around an axis over 180 degrees at 4 focus-to-sample distances for the phase retrieval. Combining the information at the 4 distances, the phase map for each angle was obtained. The obtained phase maps were reconstructed to 3D digital images and visualized with Avizo software.

A region of interest shown in **Fig. 1** is extracted from the creep-failed sample subjected to creep at a stress of 145 MPa and a temperature of 550 ºC. The figure shows 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 yellow for the Au-rich precipitates and in blue for the W-rich precipitates. Often more than one precipitate nucleate on the creep cavity surface of a single creep cavity. This study clearly demonstates how creep damage in ternary Fe-Au-W alloys is healed by the formation of nanoscale Au-rich and W-rich 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 [3]. It is found that the supersaturated Au solute diffuses significantly faster than the supersaturated W. This difference in healing kinetics indicates that supersaturated Au and W provide two healing agents that operate on different time scales. Combining both can significantly extend the time scale over which self healing of creep damage can potentially be achieved.

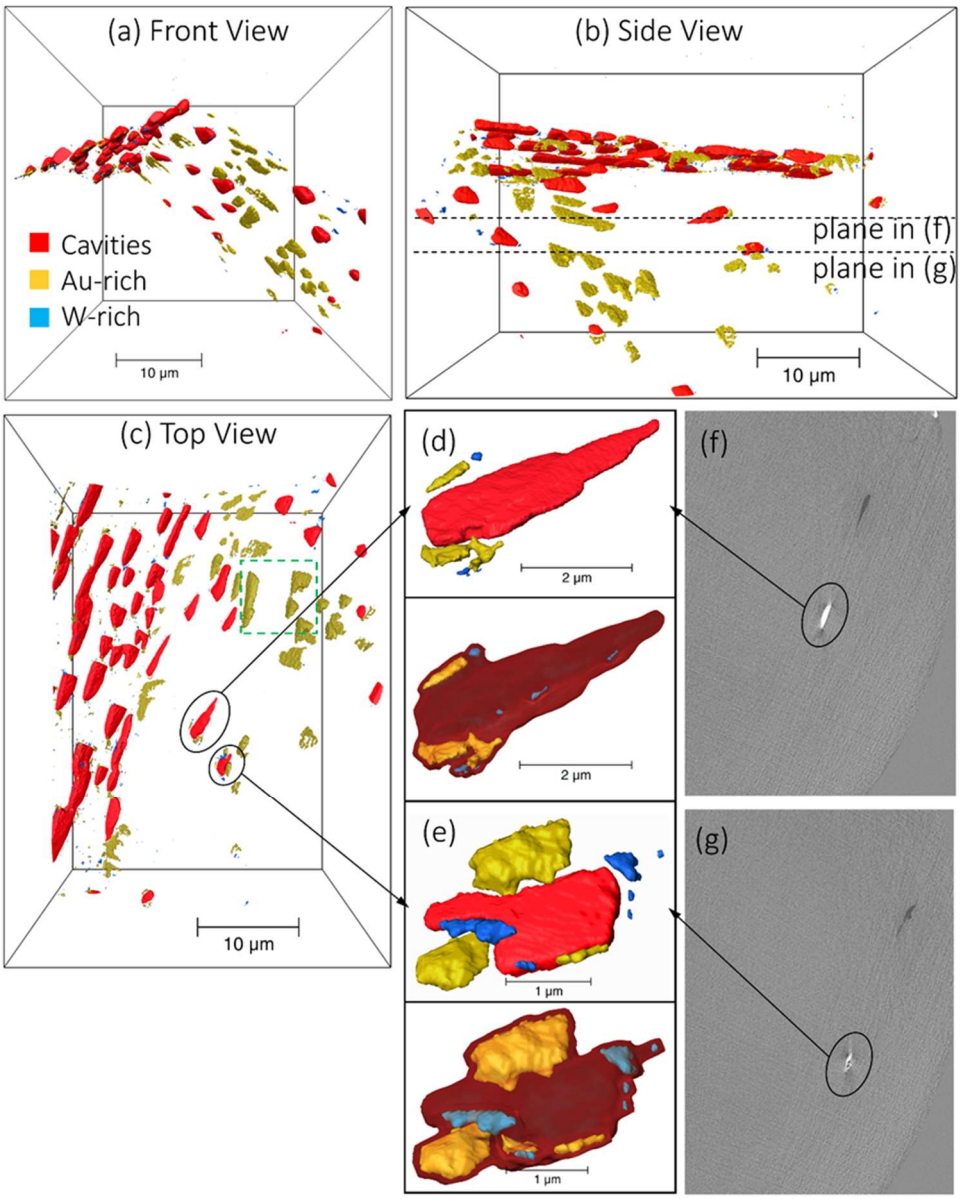

Fig. 1. Segmentation of Au-rich and W-rich precipitates after 223 h creep at 550 °C. (a) - (c) Front view, side view and top view for a region of interest (ROI) with a volume of 30 × 30 × 40 µm3. (d) - (e) Two examples of cavities partly filled by both Au-rich and W-rich precipitates. The precipitates and open volumes are shown separately on the left, while for better visualisation, larger transparent contours are presented on the right to indicate the original cavity in the absence of healing. (f) - (g) Two tomography slices normal to the stress direction. The circles in (f) and (g) correspond to the partially-filled cavities in (d) and (e), respectively. The green box in (c) shows examples of fully filled cavities, some after linkage with their neighbours. The dashed lines in (b) indicate the position of the slices in (f) and (g).

References

[1] H. Fang et al., *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 et al., *Direct view on the autonomous repair of creep damage in iron-tungsten alloys*, Acta Materialia 166 (2019) 531-542.

[3] Y. Fu et al., *Self healing of creep-induced damage in Fe-3Au-4W by multiple healing agents studied by synchrotron X-ray nano-tomography*, Acta Materialia 239 (2022) 118266.