Correlative microscopy of creep cavitation in steels using image processing of SEM, FIB-XeF2 and EBSD

Dr Tomas Martin[1], Dr Siqi He[1], Ms Eirini Gallioupolou[1], Mr Peter Thomas[1], Mr Edward Horton[1], Mr Michael Salvini[1], Dr Nicolo Grilli[1], Professor David Knowles[1], Professor Mahmoud Mostafavi[1], Professor Peter Flewitt[1]
[1]University of Bristol, Bristol, United Kingdom

Background incl. aims

Creep cavitation is an important degradation mechanism in metal alloys at high temperatures, such as in aerospace engines, gas turbines and nuclear fission and fusion reactors. During operation, metal components such as 316H stainless steel boiler headers are subjected to elevated temperatures and stresses, particularly close to weldments and geometrical features [1]. Over extended service lifetimes, these temperatures and stresses result in vacancy and dislocation movement that can gradually accumulate at microstructural features in the alloy, resulting in the formation of creep cavities that can develop into cracks and eventual failure of the component. This mechanism starts at the microstructural level, and to understand the process, it is important that the evolution of creep cavitation from nucleation to growth is characterized using advanced microscopy.

Previous work [1] has shown that in ex-service material after 65,000 hours in service in an advanced gas-cooled nuclear reactor (AGR) at 490°C–530°C, the heat-affected zone close to a weld in a 316H boiler header became extensively cavitated as a result of creep cavitation. Detailed microstructural analysis using scanning electron microscopy (SEM), electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM) showed that cavities in this material were correlated with precipitation on the grain boundaries of the steel, most significantly at the interface between $M_{23}C_6$ carbides and ferrite which had formed during thermal ageing. This detailed understanding of the interaction between microstructure and creep cavitation is essential to determine component lifetimes and design new creep-resistant materials.

However, to fully understand the interactions between creep damage and microstructure, it is beneficial to characterize this interaction over larger regions of an affected material, whilst retaining the high resolution structural, crystallographic and elemental information of individual microscopy images. In this work, we present a new methodology that combines these techniques with image processing to provide spatially-identified datasets of creep cavities, precipitation and grain structure with nanometre resolution, but across millimeter length scales of a component.

Methods

Type 316H austenitic stainless steel material was removed from a boiler component, initially service-exposed for 65,000 hours at 490°C–530°C in an AGR. Material was extracted away from high stress regions of the component and was characterized to ensure it had thermally-aged microstructure but no existing creep cavitation. The material was subsequently machined into a notched creep specimen with a gauge length of 40mm, a notch radius of 6mm and a notch acuity of 5. The creep specimen was subjected to 25 cycles of tensile stress relaxation at an initial net section stress of 390 MPa and a temperature of 550°C, and then allowed to relax in pure strain control. After testing, the crept specimen was sectioned axially, ground and polished using diamond slurry and vibropolishing to an EBSD-quality mirror finish.

An area of 3.2x1.2 mm across the centre of the sectioned specimen around the notch was imaged using 162 backscattered electron (BSE) images with a 30% overlap between neighbouring images using a Zeiss SigmaHD FEG-SEM, as well as EBSD and FIB-XeF_2 imaging

 https://doi.org/10.1051/bioconf/202412933004

across the same region. The FIB-XeF_2 imaging approach used a ThermoFisher Scios2 focused ion beam instrument, and utilized a XeF_2 gas injection system to enhance contrast between the metal matrix and precipitates, as described in [2]. The software package Dragonfly (Object Research Systems Inc, Montreal, Canada) was used for post-image processing, segmenting the BSE images to identify cavities and the FIB-XeF_2 images to identify precipitates using an Otsu threshold selection methodology [3]. EBSD data was corrected using an in-house Matlab script and all three datasets were then overlaid to enable correlation between the cavity, precipitate and grain boundary information across the entire 3.2x1.2mm region to be characterized statistically. Further detail on the methodology can be found in [4].

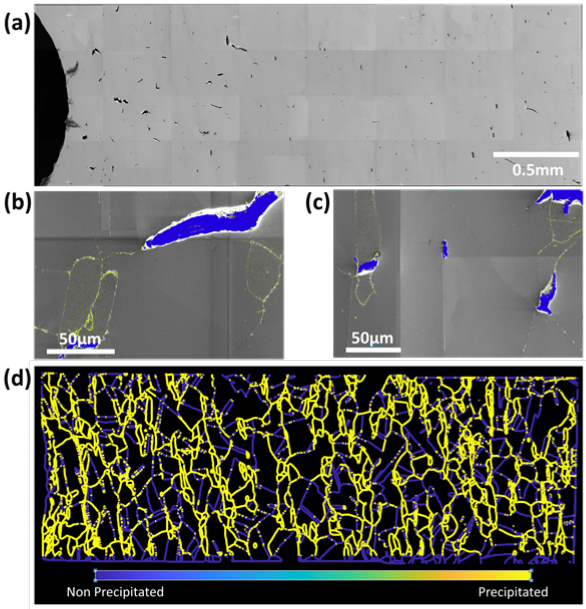
Results

The graphic shows an example of this correlative workflow. Figure (a) shows a stitched FIB-XeF_2 image of a 3.2 mm x 1.2 mm region of the creep specimen, with the notch at the left of the image. The stiched image comprises 32 individual FIB-XeF_2 images, which along with the 162 BSE images were image processed to identify creep cavities, cracks and precipiates by grayscale contrast and morphology. Two example frames with image processing are shown in (b) and (c), where yellow represents identifed $M_{23}C_6$ carbide precipitates (verified by transmission electron microscopy diffraction) and blue represens creep damage. (d) shows the statistical overlay of the identified precipitates in yellow with the grain boundary locations obtained by EBSD in blue, across the entire sampled region. Similar maps of damage were also obtained. The statistical analysis of these data described in full in [4] showed that damage showed very little correlation with grain-to-grain differences in Schmid factor (which many computational models of creep assume to be a key factor in damage location), but in this material precipitation, grain boundary angle and localised strain (as indicated by the kernal average misorientation in the EBSD data) showed much clearer correlation with damage, indicating that in this material the trapping sites at grain boundary carbide precipitates are the primary initation sites for creep damage.

Conclusion

Creep cavitation is a complex degradation mechanism, which starts at the atomic or nanometre scale and over extended times can build to failure of entire components. The methodology presented here shows the effectiveness of advanced microscopy combined with image processing to statistically correlate microstructural features with damage locations in crept material, enabling much better understanding over large areas of the contributing factors to this process.

Graphic:



Keywords:

Creep, steel, correlative microscopy

Reference:

[1] S. He, H. Shang, A.F.- Caballero, A.D. Warren, D.M. Knowles, P.D. Flewitt and T.L. Martin, The role of grain boundary ferrite evolution and thermal aging on creep cavitation of Type 316H austenitic stainless steel, Materials Science and Engineering: A, 807, 140859 (2021) DOI:10.1016/j.msea.2021.140859

[2] P.J. Thomas, M. Zimina, A. Shin, J. Pearson, P.E.J. Flewitt, T.L. Martin, Microstructural Evolution due to CO2 Oxidation and Carburisation in Fe9Cr1Mo Steel, in: Transactions of the SMiRT 27, (2024).

[3] N. Otsu, A Threshold Selection Method from Gray-Level Histograms, IEEE Transactions on Systems, Man, and Cybernetics 9 (1979) 62–66. DOI:10.1109/TSMC.1979.4310076.

[4] S. He, E. Horton, S. Moore, E. Galliopoulou, P.J. Thomas, A. Fernandez-Caballero, E. Elmukashfi, M. Salvini, M. Mostafavi, D.M. Knowles, P.E.J. Flewitt, T.L. Martin, A correlative approach to evaluating the links between local microstructural parameters and creep initiated cavities, Materials & Design (2024) 112905. DOI:10.1016/j.matdes.2024.112905.