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New Correlative Microscopy Approaches to Understand the Microstructural Origins of Creep Cavitation in Austenitic Steels

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Creep is an important degradation mechanism in high temperature, high stress environments in industries such as aerospace and nuclear reactors. Metallic components gradually accumulate creep damage over extended periods of time that can eventually build to cracking. Understanding the microstructural mechanisms behind such processes in failed components or using laboratory-based uniaxial creep tests is complicated by the large ductile deformation that occurs in the final period prior to fracture, which can obscure many of the subtler features that caused it. Typically an interrupted test gives better representative data of the early stages of cavitation, but this presents challenges to understand how nanometer-scale cavities are distributed at larger length scales [1]. Often only by combining multiple characterization techniques across different length scales can the full picture of creep damage be fully characterized [2].

In this study we will explore new ways to understand how creep cavitation forms in AISI 316H austenitic stainless steel used for advanced gas-cooled nuclear reactors (AGRs), through both ex-service material from a boiler header and laboratory uniaxial creep test specimens. We present new correlative microscopy approaches to understand the early stage nucleation of creep cavities prior to failure using a combination of scanning electron microscopy (SEM), electron backscatter diffraction (EBSD), focused ion beam (FIB) imaging assisted by xenon difluoride (XeF_2) gas and transmission electron microscopy (TEM), together with machine-learning assisted image recognition processing.

In ex-service material from a 316H boiler header run for after 65,000 hours at 490-520°C, we show how the initiation of creep cavitation is fundamentally linked to the changes in microstructure as a result of thermal ageing for extended time periods [3]. Figure 1 shows EBSD and FIB-XeF_2 images of the microstructure in the HAZ of a weld in this ex-service boiler header, showing extensive creep cavitation on grain boundaries, located next to new $M_{23}C_6$ carbide precipitates and BCC ferrite formed as a result of thermal ageing. The ferrite phase is expected to form as a result of depletion of austenite stabilizing elements as new carbide forms over time at high temperature. The EBSD grain orientation of these ferrite precipitates shows a clear relationship with the neighboring austenite grains even when a cavity exists between them, presenting strong evidence that the ferrite forms before the cavity nucleates. This relationship between austenite, carbide and ferrite is crucial to the trapping of vacancies and dislocations at the grain boundary leading to localized nucleation of creep cavities.

Figure 2 shows a novel approach to characterize creep cavitation in a similar 316H material that has been creep tested in a notched laboratory test at 390MPa and 550°C for 10,789 hours. Again, cavitation is observed on grain boundaries, but the more controlled stress direction allows better characterization about the interactions between stress and microstructure in the formation of creep cavitation. A combination of stitched high resolution SEM-backscatter images and EBSD grain orientation has been taken over the same 1 x 1 mm region of the creep-tested specimen close to the notch. Using a combination of image recognition of the SEM images to identify cavities, image correction of the EBSD using a custom script, we are able to directly analyze the cavitation behavior with grain orientation both on individual boundaries as well as statistically over the entire area of the image. These new methodologies for microstructural characterisation can provide significant new insights into the underlying mechanisms of creep cavity nucleation and can be used to create advanced computational modelling to better understand this key degradation behaviour [4].

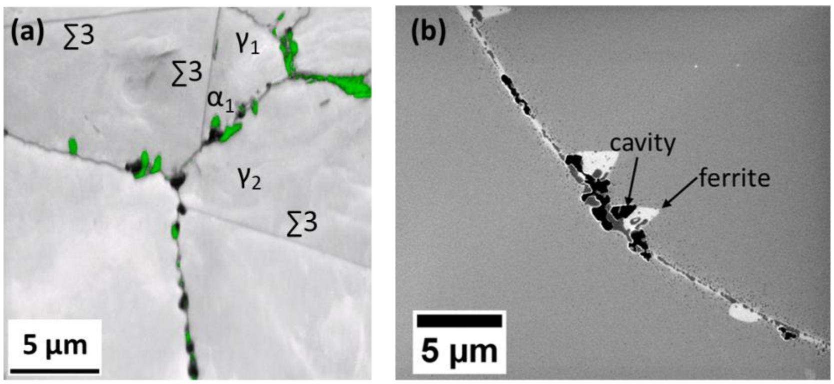

Figure 1. Grain boundary cavitation in the heat-affected zone near a weld of an ex-service 316H steel boiler header after 65,000 hours at 490-520°C. (a) shows SEM in grayscale with EBSD phase maps of ferrite precipitates in green (b) shows FIB-XeF2 image of a grain boundary showing contrast differences between cavities (black), carbides (dark grey) and ferrite (white).

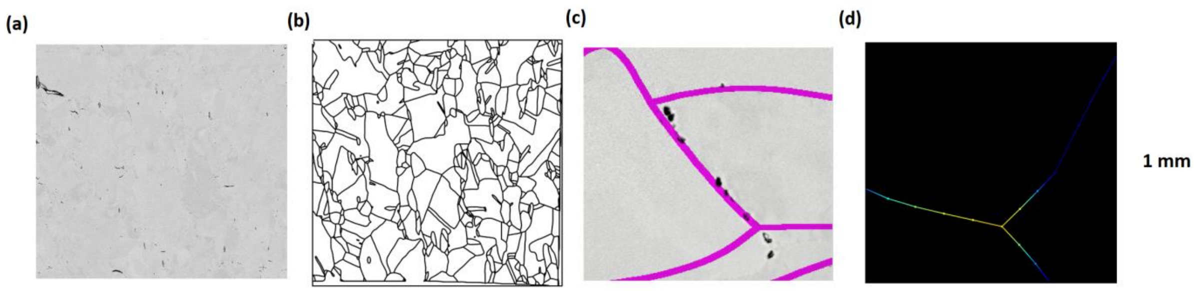

Figure 2. Correlative microscopy of a laboratory uniaxial notched creep specimen after testing at 550°C and 390MPa for 10789 hours. (a) shows SEM BSE images of a region of the specimen close to the notch, (b) shows EBSD of the same region. (c) and (d) show examples of the correlative approach to link damage to microstructure.

References:

[1] S He, PEJ Flewitt, DM Knowles and TL Martin, Proceedings of the 15th International Conference on Engineering Structural Integrity Assessment and the 2019 International Symposium on Structural Integrity, pp. 194-203 (2021)

[2] TL Martin, S He, AD Warren, H Shang, DM Knowles and PEJ Flewitt, Proceedings of the Pressure Vessels and Piping Conference 2020 (2020) doi:10.1115/PVP2020-21791

[3] S. He, H. Shang, A Fernandez-Caballero, A.D. Warren, D.M. Knowles, PEJ Flewitt and TL Martin, Materials Science and Engineering: A **807** (2021), 140859. doi:10.1016/j.msea.2021.140859

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