



Creep damage initiation in high-temperature steel: a comparative study of ferritic, martensitic and austenitic microstructures



Eirini C. Galliopoulou¹✉, Siqi He¹, Michael Salvini², Nicolò Grilli², Gerardo T. Martinez³, Hao Shang¹, Alan Cocks⁴, John Siefert⁵, Jonathan D. Parker⁵ & Tomas L. Martin¹✉

Premature failure of critical energy infrastructure caused by creep damage, threatens productivity and safety, emphasizing the need for robust methods to monitor and predict material performance. Creep damage nucleation in metals is driven by nano- and micro-structural features, but linking these early evolutions to later failure is complex. This study presents a correlative methodology integrating SEM, FIB, EBSD, EDX, TEM, image recognition, data analysis and crystal plasticity simulations to investigate creep cavity formation in ferritic Grade 91, martensitic Grade 91 and austenitic 316H steels. Results show that grain boundaries and localized deformation strongly influence creep cavity initiation. MnS inclusions drive cavity nucleation in Grade 91 steel, while grain boundary ferrite and $M_{23}C_6$ carbides dominate in 316H steel. Calibrating a crystal-plasticity framework with experimentally identified precursors reveals that deformation, inclusions and precipitates translate into damage-sensitive indicators such as slip rate hotspots and volumetric strain, enabling prediction of early creep cavity nucleation mechanisms.

One of the most effective strategies to enhance the efficiency of electricity generation in both conventional and nuclear power plants is to increase the temperature and pressure of the steam driving the turbines. Transitioning from subcritical to ultra-supercritical (USC) conditions in coal-fired power plants and natural gas plants can raise thermal efficiency from approximately 38–47%, thereby reducing both fuel consumption and CO₂ emissions¹. USC steam turbines are typically designed to operate at temperatures of 650 °C or higher². Ongoing research and development efforts in the USA, EU and Japan are focused on Advanced Ultra-Supercritical (A-USC) steam turbines, which aim to achieve superheater temperatures exceeding 700 °C and steam pressures around 35 MPa³. From a nuclear perspective, Generation IV reactors are being designed with core outlet temperatures that can exceed 900 °C, enabling higher thermal efficiencies and advanced fuel cycles⁴.

To meet these extreme operating conditions, both current and next-generation power plants require advanced structural materials capable of withstanding high temperatures and stresses over long service lives. One of the primary degradation mechanisms under such conditions is creep, a

time-dependent deformation that occurs when materials are subjected to sustained stress at elevated temperatures. Creep typically involves an initial strain-hardening phase due to dislocation generation, followed by dynamic recovery and dislocation substructure formation and at later stages the nucleation, growth and linking of cavities that ultimately lead to component failure⁵. Such failures, especially if premature and occurring within the intended service life, can compromise not only the efficiency but also the safety of power plant operations.

Creep damage originates at the microstructural level, initiating through mechanisms such as cavity formation and strain localization, but evolves into macroscopic failure as the material progresses through its service life. To thoroughly understand and model these damage mechanisms, it is essential to employ advanced characterization tools capable of detecting early-stage creep phenomena. At the same time, it is equally important to scale these observations to the engineering level, which requires statistically representative data and macro-scale analysis to ensure that localized findings are relevant to the behaviour of full-scale components.

¹Interface Analysis Centre, University of Bristol, Bristol, England, UK. ²School of Electrical, Electronic and Mechanical Engineering, University of Bristol, Bristol, England, UK. ³Department of Materials, University of Oxford, Oxford, England, UK. ⁴Department of Engineering Science, University of Oxford, Oxford, England, UK.

⁵Electric Power Research Institute (EPRI), Charlotte, NC, USA. ✉e-mail: eirini.galliopoulou@bristol.ac.uk; tomas.martin@bristol.ac.uk

While advanced microscopy techniques such as Scanning Electron Microscopy (SEM), Electron Back-scatter Diffraction (EBSD), Focused Ion Beam (FIB) microscopy, Transmission Electron Microscopy (TEM) and Atomic Force Microscopy (AFM) offer exceptional resolution for examining microstructural features like grain boundaries, voids and precipitates at the micro- and nano-scale, their scope is inherently limited when it comes to representing the behaviour of full-scale engineering components. These components, which often span from centimeters to meters, contain a significant number of such features, making it infeasible to extrapolate bulk material behaviour from isolated microscopic observations. An additional challenge is that the microstructure within real components is often heterogeneous, so local measurements may not reflect the behaviour of the material elsewhere. This mismatch in scale, together with spatial variability, presents a major challenge for integrating microstructural data into engineering-level simulations, such as crystal plasticity finite element (CPFE) models, which require statistically representative volumes encompassing thousands of grains. To tackle this challenge, researchers need to adopt multiscale and correlative strategies that integrate data from diverse imaging techniques and length scales, aiming to construct a more complete picture of the material's structural response under service conditions. Previous CPFE-based damage models used prescribed damage laws to predict creep cavity formation, but they did not compare the predictions on a one-to-one basis with microscopy images. Statistical measures have been successfully compared with experiments⁶ but detailed classification approaches to identify damage indicators among the CPFE state variables have not been previously explored.

Deng et al.⁷ combined microscopy techniques such as EBSD, TEM, X-ray Diffraction (XRD), Atom Probe Tomography (APT), Electron Channeling Contrast Imaging (ECCI) and digital image correlation (DIC) to examine a mechanically-induced twinned deformed microstructure. To correlate macroscopic creep behaviour with microscopic defect structures and solute segregation, Sharma et al.⁸ adapted a correlative microscopy approach, combining TEM, APT and EBSD as well as quantitative data analysis. A multiscale correlative tomography method was implemented by Slater et al.⁹ where X-ray computed tomography (micro- and nano-CT), FIB-SEM with EBSD and scanning transmission electron microscopy - energy dispersive X-ray (STEM-EDX) imaging was combined to investigate the correlation of grain boundary orientation and precipitation on creep cavitation in Type 316 stainless steel. However, their analysis was limited to examination of a volume containing only three grain boundaries, thus lacking the statistical confidence needed for extrapolation to engineering-scale models. Many earlier studies are restricted to 2D characterization, even though creep damage and microstructural evolution develop in 3D. Statistically meaningful insights into degradation mechanisms can be achieved by combining large-volume 3D tomography with targeted high-resolution observations. Galliopoulou et al.¹⁰ applied 3D PFIB serial sectioning coupled with image segmentation and cavity quantification and complemented this with high-resolution 2D SEM, EBSD, and EDX analyses. This integrated methodology revealed the spatial distribution of cavities and precipitates and supported interpretations of the microstructural creep mechanisms in Grade 91 steel.

Correlative microscopy techniques produce multiple complementary datasets from the same material, making it essential to use data analysis methods capable of integrating and interpreting them collectively. A major advantage is having an analysis workflow that is versatile enough to be applied across different materials, enabling direct comparison when the same imaging techniques are used. In the present study, three microstructures commonly found in industrial energy systems, ferritic Grade 91, martensitic Grade 91 and austenitic 316H steel, were selected. The same data analysis approach was applied to their correlative electron microscopy datasets following creep exposure, allowing for consistent evaluation of microstructural damage and cavity evolution across different material systems.

Grade 91 steel has gained widespread use in the energy sector for critical pressure components such as tubing, piping and forgings in boilers, superheaters and headers¹¹. It is extensively applied in supercritical and ultra-supercritical thermal power plants operating at 500–600 °C¹² and is also considered a strong candidate for components in advanced ultra-supercritical (AUSC) plants and Generation IV reactors due to its excellent high-temperature performance¹³. 316H austenitic stainless steel has similarly seen broad application in structural and boiler components of Advanced Gas-cooled Reactors (AGRs), which operate at temperatures up to 750 °C^{14,15}. It is also a leading candidate for reactor vessels in sodium-cooled fast reactors (SFRs), one of the Generation IV nuclear reactor designs¹⁶.

Although Grade 91 and 316H are currently among the most promising candidates for Gen IV reactors, their performance under these conditions is not yet fully understood, particularly in terms of creep cavitation and damage. This knowledge gap was one of the key reasons these materials were selected for applying the correlative electron microscopy and data analysis workflow developed in this study. Cavity nucleation typically initiates during the early stages of creep, usually the primary and secondary stages¹⁷. This phenomenon is often attributed to weakened bonding between second-phase particles and the matrix, as well as grain boundary sliding^{18,19,20}. Although grain boundary sliding has historically been described as a relative motion between neighboring grains, real 3D constraints limit such behaviour. Nevertheless, previous work has shown that sliding-induced stress redistribution can still increase the normal stress on inclined and transverse boundaries, thereby promoting cavitation^{5,21,22}. Cavity nucleation is generally linked with the simultaneous presence of discontinuities and impurities at grain boundaries. Inclusions such as manganese sulphides (MnS) and Al₂O₃ have been consistently associated with void formation in 9%Cr steels^{23,24}. In Grade 91 steel, creep cavities have also been linked to intergranular precipitates like M₂₃C₆ carbides and Laves phase²⁵. A recent investigation comparing ferritic and martensitic grain boundaries in P91 steel²⁶ revealed that fine ferritic grains degrade more rapidly under creep conditions. This degradation was marked by a sharp decline in dislocation density and hardness, making ferritic grain boundaries more prone to cavity formation and coalescence than tempered martensitic boundaries. In tempered martensitic Grade 91 steels, lath boundaries have been proven more resistant to cavity nucleation while creep damage is mainly located at prior austenite grain boundaries (PAGBs)²⁷. In austenitic stainless steels such as 316H, creep cavitation is closely tied to the grain boundary network and the presence of intergranular precipitates²⁸. The M₂₃C₆ carbide is the most prevalent and is strongly associated with cavitation²⁹. Also, secondary phases like ferrite have been associated with cavity initiation in 316H steels³⁰. It has been suggested that local stress may induce cavity nucleation through decohesion at interfaces such as austenite–ferrite, ferrite–M₂₃C₆ or at triple junctions involving all three phases, due to strain incompatibilities³¹.

Ferrite, martensite and austenite are thermally and structurally related through phase transformations³². Since austenite often serves as the parent phase, its characteristics can influence the evolution and damage behaviour of the resulting microstructures, offering valuable insights into creep mechanisms across different phase types. Ex-service ferritic Grade 91, martensitic Grade 91 and austenitic 316H steels were selected not only for their industrial relevance and future potential in high-temperature applications, but also because their similarities and differences provide a valuable basis for understanding creep from multiple perspectives. This study presents a correlative workflow combining SEM, TEM, EBSD, EDX, FIB, image segmentation, advanced data analysis and CPFE damage simulations to investigate creep cavity nucleation in all three microstructures. It is demonstrated that the methodology is broadly applicable across different materials while producing consistent and comparable results. This highlights its potential to improve reproducibility, support standardized characterization workflows and enable more efficient comparison of creep-damage behaviour across different materials and studies.

Results and Discussion

Ferritic grade 91 steel

Two ferritic uniaxial samples were creep tested: one interrupted at 4% strain (G91-F-4%) while the other was tested to failure at 35.6% strain (G91-F-35.6%). The failed sample was selected to serve as the host for the correlative SEM-EBSD technique. Due to the significant necking and resulting reduction in cross-sectional area near the fracture (52.2%), local stresses in that region are elevated compared to areas farther from the failure zone. Since the objective of this study is to investigate cavity precursors during the early stages of creep, regions with lower stress levels were targeted to capture cavities in their initial formation phase and to avoid areas near the failure

edge dominated by pronounced damage such as cavity interlinkage and crack propagation. Thus the region which was selected was located 28 mm far from the failure edge. On the other hand, the interrupted sample was chosen for high resolution investigations comprising of high magnification SEM imaging on targeted early stage cavities and TEM-EDS imaging of similar nano-scale regions, which validated the micro-and macro-scale observations and provided deeper insight into the underlying damage mechanisms.

Figure 1a shows the stitched SEM map covering a $0.41 \text{ mm} \times 0.35 \text{ mm}$ area. The cavity density found upon segmentation was $> 2000 \text{ cavities/mm}^2$. Figure 1b displays the EBSD map of the same exact region, while Fig. 1c

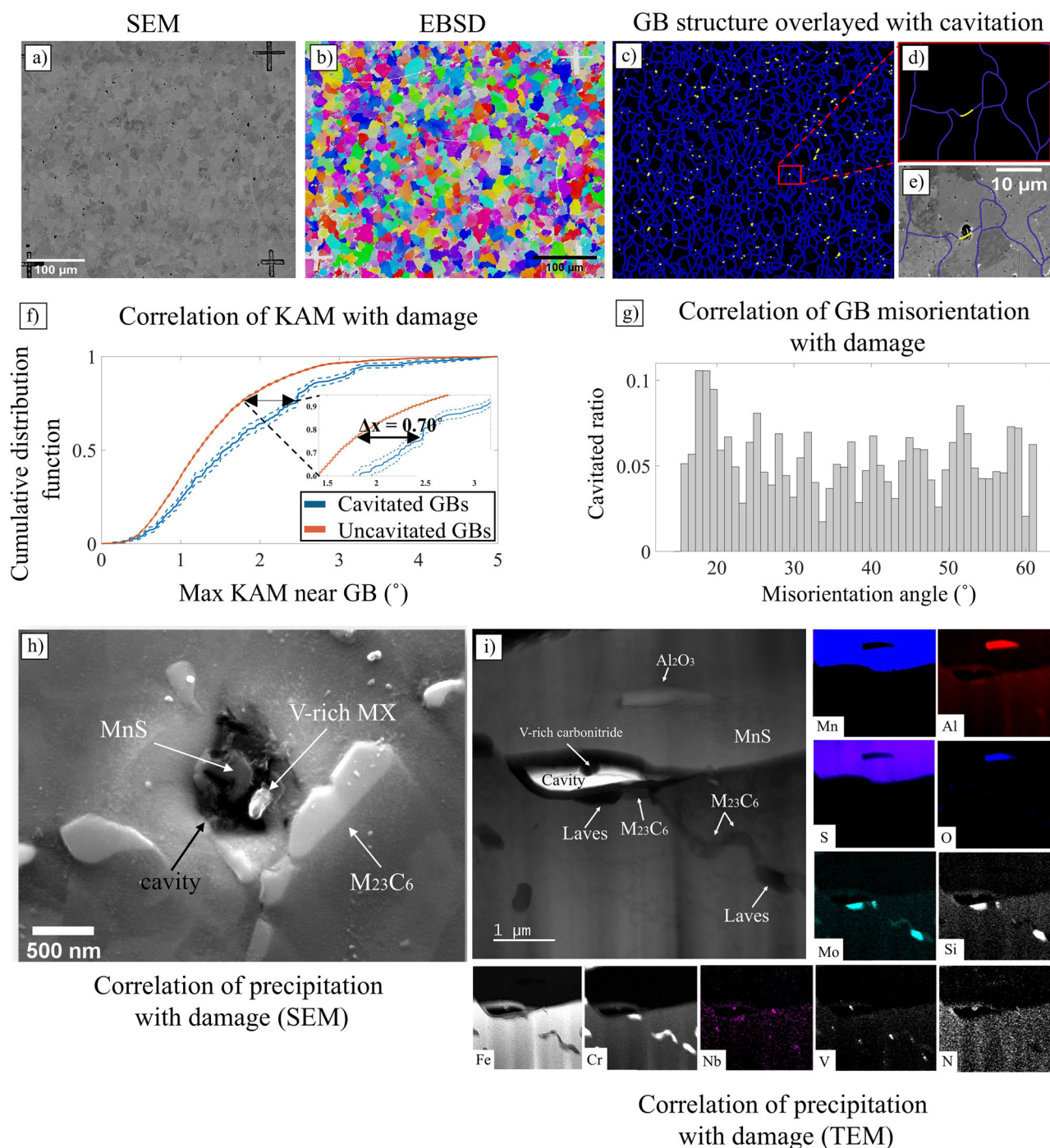


Fig. 1 | Correlative analysis results of ferritic Grade 91 steel. a Stitched SEM map of examined region, (b) EBSD map, (c) cavities (yellow) overlaid on grain boundaries (blue), (d) magnified view of (c), (e) magnified view of (c) overlaid on SEM map, (f)

maximum KAM values near GBs for the examined region, (g) Cavitation ratio of GBs across all misorientation angles, (h) high magnification SEM map exhibiting cavitation related to MnS inclusion and smaller precipitates and (i) STEM-EDS map.

illustrates the extracted grain boundary network with the cavitated grain boundary segments highlighted in yellow color, while undamaged grain boundaries are annotated with blue color. The analysis reveals that the vast majority of cavities were intergranular and predominantly located at grain boundary junctions. A representative example of such intergranular cavitation is shown on the high magnification images in Fig. 1d, e. Figure 1d is a magnified view of the region in Fig. 1c, while Fig. 1e shows the corresponding area from the SEM map overlaid with the grain boundary network. It is evident that the damaged grain boundary segment (in yellow color) aligns precisely with the cavity observed in the SEM map, indicating a high degree of accuracy in the segmentation and analysis process.

By combining Fig. 1b, c, the misorientation of each grain boundary can be individually identified. Since cavitation was predominantly observed along grain boundaries, it became important to investigate whether certain types of boundaries are more susceptible to damage. In the examined region, 5.02% of the total grain boundary length exhibited cavitation, regardless of misorientation. One common way to classify grain boundaries is by their misorientation angle, which is obtained through EBSD mapping. To explore this, the misorientation angles of all grain boundaries were plotted against their cavitation ratio in Fig. 1g, covering the range from 15° to 62.8°. The cavitation ratio is defined as the fraction of the total length of grain boundaries at a given misorientation that is decorated with cavities. For example, a ratio of 0.1 at 15° indicates that 10% of the total length of 15° boundaries is cavitated. As shown in Fig. 1g, the highest cavitation ratios occur at misorientation angles between 17° and 20°, exceeding 0.1. However, these boundaries are among the least frequent in the microstructure, while boundaries between 45° and 55° are the most common³³. In previous study on ferritic Grade 91 material³³, grain boundaries with misorientation angles between 15° and 20° also exhibited the highest cavitation ratios, reaching up to 0.15, but the lowest carbide precipitation. Notably, the region examined in that study was located 19 mm from the fracture surface, lying much closer to the necking zone and thus subjected to higher stress levels than the region analyzed in the present work (28 mm from fracture). This increase of cavitation ratio from ~ 0.1 to ~ 0.15 suggests that the cavitation susceptibility of 15°–20° grain boundaries increases with applied stress. Since precipitates can pin grain boundaries and help stabilize them during deformation^{34,35}, their absence may lead to increased grain boundary sliding. In such cases, sliding is accommodated by the motion of intrinsic dislocations within the grain boundary zone through glide and climb, making boundary displacement easier when precipitate obstacles are not present. This increased grain boundary sliding can lead to localized strain mismatches, which facilitate the initiation of cavities. Although these boundaries have lower misorientation and are less prone to dislocation pile-up, their lack of precipitate-based reinforcement makes them vulnerable to creep damage through a mechanism distinct from that of higher-angle boundaries, where precipitates can either pin the boundaries or their coarsening can cause cavitation³⁶. Overall, although 15°–20° boundaries are relatively rare in the ferritic microstructure, they appear particularly vulnerable to cavitation.

To investigate if localized strain is associated with cavity nucleation, Kernel Average Misorientation (KAM) was analyzed along grain boundaries in the same region of the failed sample, located 28 mm from the fracture surface. Figure 1f presents the cumulative distribution functions of maximum KAM values near cavitated (blue) and uncavitated (orange) grain boundaries. Cavitated boundaries exhibit consistently higher maximum KAM values. This suggests a strong correlation between localized plastic deformation and intergranular damage.

To investigate creep cavities at an early stage of formation and identify the factors contributing to their initiation, the sample interrupted at 4% strain was selected for targeted analysis. The results are presented in Fig. 1h, i. Initially, the sample was screened over a large area using stitched SEM mapping. However, cavitation was minimal both in number and in size, with cavities observed at the nanoscale. Due to the limitations of accurately segmenting such small features in large-area maps, a more focused imaging approach was adopted to examine individual cavities in detail in this sample.

All randomly selected cavities were found to be associated with MnS inclusions. Figure 1h, i show high-resolution SEM and STEM-EDS maps respectively, of cavities linked to MnS. The SEM image reveals a cavity associated with an MnS inclusion, accompanied by smaller precipitates such as MX-type carbonitrides and $M_{23}C_6$ carbides. The STEM image provides a clearer view of the cavity's position relative to the MnS inclusion. The cavity is located at the interface between the MnS and the ferritic matrix, with smaller precipitates decorating the cavity's perimeter. MnS inclusions have a higher thermal expansion coefficient ($18 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$)³⁷ compared to the ferritic P91 matrix ($12 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$). This mismatch can generate local stresses at the inclusion-matrix interface during exposure to creep temperatures. The presence of additional precipitates, such as carbides or Laves phase, can further amplify these local stresses. These findings in Fig. 1f, h, i are most likely all interrelated. Since strain and stress are closely linked, the elevated KAM values observed near cavitated grain boundaries in Fig. 1f may result from MnS inclusions located on those boundaries, which induce local deformation and promote cavitation. This interpretation aligns with previous studies that examined the role of MnS inclusions in steel deformation. For example, Wang et al.³⁸ reported high KAM values surrounding MnS inclusions, while Liu et al.³⁹ observed elevated KAM across grain boundaries intersected by or aligning with the location of MnS inclusions, both linking these inclusions to damage initiation.

Martensitic Grade 91 steel

Three martensitic samples were evaluated under creep conditions: a uniaxial specimen interrupted at 1% strain (G91-M-1%), a uniaxial specimen that failed at 3.7% strain (G91-M-3.7%), and a double-notched specimen tested to failure (G91-M-DN), with fracture occurring near the second notch. The notched configuration (G91-M-DN) was used to introduce a triaxial, elevated stress state and a stress gradient where the plain-bar section carries roughly half the stress of the notch throat, allowing assessment of stress-dependent damage evolution and it was selected for correlative SEM-EBSD analysis. To minimize the influence of elevated local stresses near the notches, the region chosen for examination was located approximately 20 mm from the first notch. The uniaxial failed martensitic specimen (G91-M-3.7%) was included to enable a direct comparison of cavity density and average cavity size evolution with the uniaxial failed ferritic specimen (G91-F-35.6%), ensuring a consistent basis for evaluating microstructural damage progression across the two materials. In contrast, the uniaxial interrupted martensitic sample (G91-M-1%) was used for high-resolution investigations, including high-magnification SEM imaging of early-stage cavities and TEM-EDS analysis of comparable nanoscale regions.

Figure 2 presents the correlative SEM-EBSD data from the martensitic double-notched failed sample (G91-M-DN), displayed in a format consistent with the ferritic material analysis shown in Fig. 1. Fig. 2a shows the stitched SEM map covering an area of 0.72 mm × 0.6 mm. Segmentation revealed a cavity density of 372 cavities/mm², which is significantly lower than that observed in the ferritic sample, highlighting martensite's greater resistance to cavity nucleation. Martensite forms through a diffusionless shear transformation of austenite (FCC) during rapid cooling, trapping carbon atoms within the distorted lattice. Although the original austenitic microstructure is not directly visible post-transformation, PAGBs remain critical, as intergranular damage in martensitic steels predominantly occurs along these boundaries rather than at martensitic lath boundaries⁴⁰. Therefore, reconstructing the parent austenite microstructure is essential for accurately identifying damage-prone regions. To achieve this, child-to-parent reconstruction was performed using the MTEX toolbox in MATLAB, and the correlative SEM-EBSD technique was applied to the reconstructed parent microstructure, as shown in Fig. 2b. Figure 2c displays the extracted PAGB network, with cavitated boundaries highlighted in yellow and undamaged boundaries in blue. Most cavities were located along PAGBs and particularly at PAGB junctions. A representative case of such intergranular cavitation is shown in Fig. 2d, e. Figure 2d provides a magnified view of the region highlighted in Fig. 2c, while Fig. 2e overlays the SEM map with the corresponding parent grain boundary network.

Fig. 2 | Correlative analysis results of martensitic Grade 91 steel. a Stitched SEM map of examined region, **(b)** EBSD map, **(c)** cavities (yellow) overlaid on grain boundaries (blue), **(d)** magnified view of **(c)**, **(e)** magnified view of **(c)** overlaid on SEM map, **(f)** maximum KAM values near GBs for the examined region, **(g)** Cavitation ratio of GBs across all misorientation angles, **(h)** high magnification SEM map exhibiting cavitation related to MnS inclusion and **(i)** STEM-EDS map.

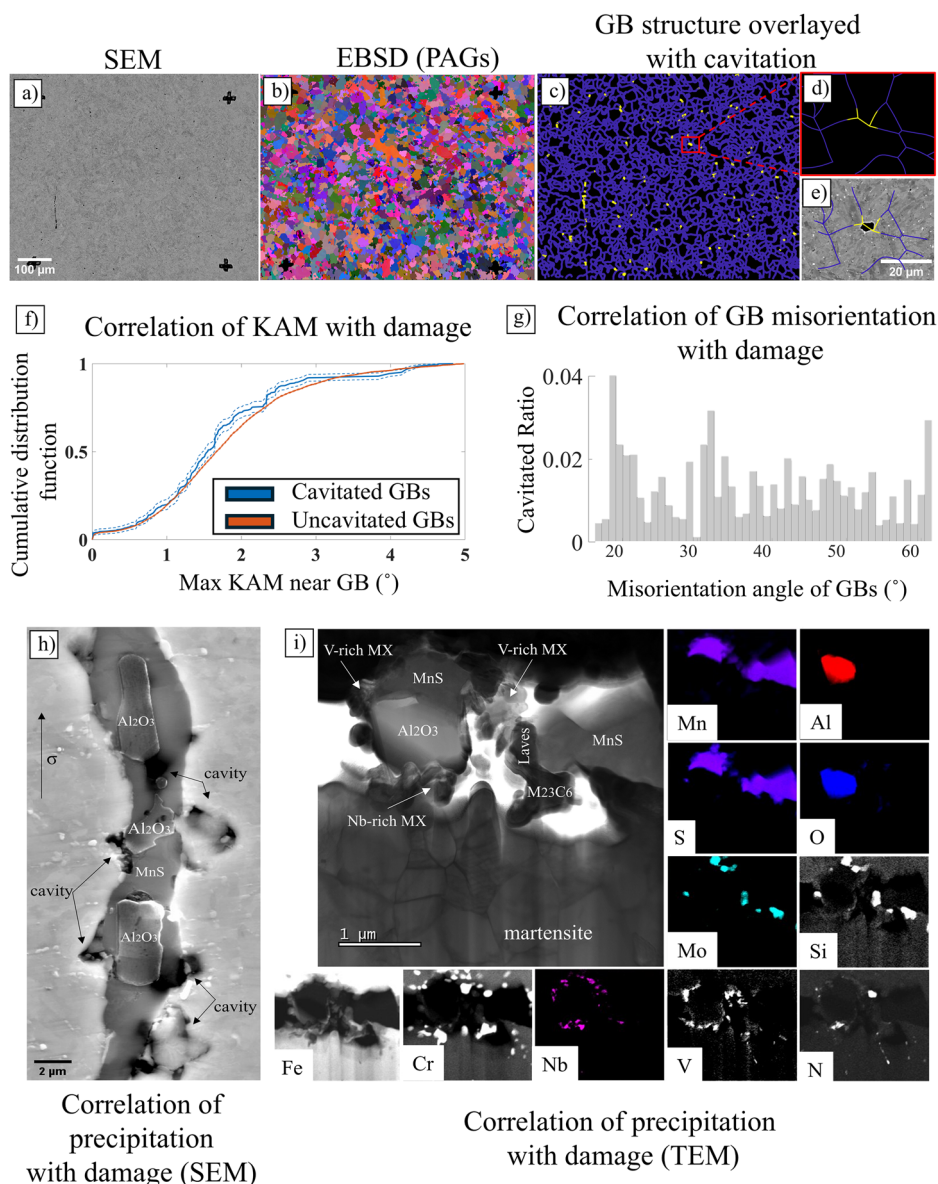


Figure 2g reveals distinct peaks in cavitation ratios corresponding to PAGB misorientations near 19° , 32° , and 60° , with the most pronounced peak occurring at 19° . This suggests that, even in the lowest stress regions of the sample, low-angle PAGBs, particularly those around 19° , are the first to undergo damage. While such boundaries typically exhibit lower lattice mismatch, their involvement in GB junctions where extended boundary length and multiaxial stress concentrations enhance diffusion and promote cavity nucleation, can potentially justify their susceptibility to cavitation. The peak observed at 32° may be attributed to the preferential segregation of MnS inclusions during solidification. The majority of these inclusions were observed along GBs with misorientations in the $30\text{--}50^{\circ}$ range, making boundaries near 32° more prone to cavitation due to the presence of these second-phase particles. Finally, the elevated cavitation tendency at 60° , corresponding to $\Sigma 3$ coincidence site lattice (CSL) boundaries, is unexpected given their typically high resistance to damage. This anomaly could be explained by several factors: cavitation occurring on adjacent random boundaries, the participation of $\Sigma 3$ boundaries in triple junctions, or the presence of incoherent $\Sigma 3$ twin facets (such as $\{112\}$ or $\{110\}$), which are energetically less stable and more diffusively active than coherent twins^{40,41}.

Although Fig. 2f does not show a significant difference in the maximum KAM between cavitated and uncavitated GBs, previous work using

the same correlative technique⁴⁰, demonstrated that high-magnification EBSD is necessary to detect localized deformation near early-stage cavities - deformation that remains undetected in large-area SEM-EBSD scans. In those highly deformed regions, EDS mapping revealed strong manganese peaks, likely indicating the presence of MnS inclusions that were not clearly visible in the imaging plane. This implies that the observed localized deformation may not be driven by the martensitic grain structure itself, but rather by the presence of inclusions. The fact that both cavities and MnS inclusions predominantly form along PAGBs supports the hypothesis that inclusion-induced triaxiality is the primary driver of localized deformation and cavity nucleation. In contrast, the role of martensitic laths may be secondary or even inhibitory to cavitation. This interpretation is further supported by the relative scarcity and smaller size of intragranular cavities along block or lath boundaries. Martensitic laths, characterized by high dislocation density, act as vacancy sinks, reducing the vacancy flux necessary for cavity growth. Therefore, the localized deformation observed near PAGBs is more likely associated with the stress concentration around MnS inclusions, rather than the intrinsic structure of the martensitic laths or blocks. An important point is that the choice of characterization method is critical. The large-scale EBSD used in Fig. 2b failed to reveal localized deformation, which only became apparent with higher-resolution scans.

The influence of MnS inclusions on the microstructure is further illustrated in Fig. 2h, which shows a high-magnification SEM image of an elongated MnS inclusion with an entrapped Al_2O_3 particle in the interrupted 1% strain sample. In the martensitic material examined, MnS inclusions were frequently observed in close proximity to aluminum oxides Al_2O_3 , forming complex inclusions. While similar associations were occasionally seen in the ferritic material, they occurred with much lower frequency. In most ferritic regions, MnS appeared without accompanying large Al_2O_3 particles. This observation aligns with earlier findings by Wakoh et al.⁴², who proposed that oxide particles can act as nucleation sites for MnS when sulfur levels are sufficient. During casting, MnS tends to segregate around pre-existing solid impurities such as Al_2O_3 , which originate from the refractory lining, crucible materials, or slag⁴³. These solid surfaces lower the energy barrier for MnS nucleation, promoting the formation of composite inclusions.

The ferritic and martensitic samples in this study share the same chemical composition and service history with their only difference originating from their post-service heat treatments, as described in the Methods section. The faster cooling in the martensitic heat treatment appears to have preserved the as-cast MnS- Al_2O_3 associations, trapping them in their original form. In contrast, the slower cooling of the ferritic treatment may have allowed for equilibrium transformations and longer diffusion times, enabling MnS to precipitate independently, often away from Al_2O_3 . This difference in thermal history provides a plausible explanation for the higher frequency of complex MnS- Al_2O_3 inclusions observed in the martensitic samples compared to the ferritic ones.

Figure 2h reveals three distinct types of early-stage, nanoscale cavities associated with MnS inclusions. The first type appears at the MnS-martensitic matrix interface, while the second forms at the MnS- Al_2O_3 interface. Adjacent grains appear highly deformed and are encircled by nano-sized cavities. This represents the third type of MnS-associated cavity and further supports the hypothesis that MnS inclusions induce localized deformation in the matrix. This deformation is likely driven by thermal expansion mismatch between the MnS and the surrounding martensitic matrix, which can lead either to direct decohesion at the inclusion-matrix interface or strain accumulation in neighboring grains, ultimately resulting in cavitation.

The TEM-EDS map in Fig. 2i, also taken from the interrupted 1% strain sample, provides additional insight into inclusion-related damage. Cavities are again observed at both MnS-martensite and MnS- Al_2O_3 interfaces. Smaller precipitates such as MX-type carbonitrides, M_{23}C_6 carbides and Laves phase are present at these interfaces, potentially acting as secondary nucleation sites for cavities. As a general observation, secondary precipitates were more frequently found within cavities at the MnS-martensite interface, whereas cavities at the MnS- Al_2O_3 interface often formed without the involvement of smaller precipitates contributing to damage. Differences in the thermal expansion coefficients ($8.4 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ for Al_2O_3 compared to $18 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ for MnS)³⁷, may lead to damage at the interface. The thermal expansion coefficient of martensite is $14.1 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ ⁴⁴, indicating a smaller mismatch between MnS-martensite than between MnS- Al_2O_3 . This discrepancy may explain why cavity nucleation at the MnS-martensite matrix interface is often accompanied by smaller precipitates, which promote local stress concentrations and assist in initiating damage.

The role of MnS inclusions in damage formation is similar in both ferritic and martensitic Grade 91 steels. In ferrite, MnS particles are mostly present as isolated sulphides or in simpler combinations, whereas in martensite, MnS inclusions are more often associated with Al_2O_3 . Thus, although cavities form at MnS-matrix interfaces in both microstructures, the martensitic heat treatment preserves the as-cast MnS- Al_2O_3 associations, introducing an additional damage-prone interface that is less frequent in ferrite. Despite the presence of this extra cavity initiation site in martensite, the overall cavity density and grain boundary cavitation ratio are significantly higher in the ferritic steel. This can be attributed to the strengthening effect of the martensitic lath structure, which inhibits cavity

growth and interlinking. The formation of this lath structure arises from the diffusionless austenite-to-martensite transformation during rapid cooling, producing a hierarchical arrangement of laths, blocks and packets with a fine dislocation substructure and small effective grain size. Such a microstructure resists creep deformation and confines damage primarily to the prior-austenite grain boundaries, which act as the dominant weak interfaces. In contrast, the ferritic heat treatment allows carbon to redistribute by diffusion, leading to a more equiaxed and comparatively softer grain structure that offers less resistance to boundary sliding, vacancy transport and cavity coalescence. As a result, the softer ferritic matrix allows cavities to expand and coalesce more easily, following diffusion paths along grain boundaries with less resistance from the surrounding material. This behaviour is statistically illustrated in Fig. 3. However, although MnS inclusions are a common feature across Grade 91 steels, different heats can exhibit marked variability in creep strength and ductility and the dominant cavitation mechanisms may not manifest with the same intensity in all heats.

To investigate damage evolution in both materials, cavity density and average cavity size were measured along the stress axis of the uniaxially failed ferritic (G91-F-35.6%) and uniaxially failed martensitic (G91-M-3.7%) samples. Figure 3a, b present high-magnification SEM images of the ferritic and martensitic microstructures, respectively, highlighting the distinct grain morphologies of the two materials, with the ferritic microstructure exhibiting predominantly equiaxed grains and the martensitic microstructure characterized by lath structures. Eleven regions were scanned in the ferritic sample and nine in the martensitic. The average cavity size per region, relative to total cavity count along the gauge length, is shown in Fig. 3c (ferritic) and Fig. 3d (martensitic). A decreasing average cavity size with increasing cavity count near the fracture edge suggests dominant continuous nucleation, as newly formed cavities are nanoscale. Conversely, an increasing average size indicates cavity interlinkage, since coalesced cavities are significantly larger than individual ones.

In the ferritic material (Fig. 3c) the average cavity size (black distribution) exhibits similar values along the gauge length until reaching the area $Y = 25 \text{ mm}$, forming what could be described as a size plateau, although the total cavity number (blue distribution) increases substantially after $Y = 20 \text{ mm}$. This suggests that, within this segment of the sample, both continuous nucleation and interlinkage occur at a balanced rate, preventing a significant decrease or increase in the average cavity size. On the other hand, in the region $Y = 28 \text{ mm}$, a notable escalation of the overall cavity size is observed. This sharp increase indicates the occurrence of intense interlinkage in that particular area, consequently elevating the overall cavity size. The different cavity types (individually nucleated cavities and interlinked cavity clusters) are shown in the characteristic SEM maps which were chosen as representative of each of the cavity populations. In summary, both phenomena of continuous cavity nucleation and cavity interlinkage, manifest concurrently in the material in areas of lower stress levels. However, as stress intensifies closer to the fracture edge of the sample, cavity interlinkage becomes more pronounced, increasing the overall cavity size.

In the martensitic material (Fig. 3d), moving towards the fracture edge of the sample, the average cavity size reduces significantly, notably starting from area $Y = 6 \text{ mm}$, with the lowest average cavity size observed in area $Y = 22 \text{ mm}$, which is the closest mapped area to the fracture. Despite the considerable rise in cavity counts in high-stress regions, the average cavity size decreased, affirming the formation of new, smaller-sized cavities due to heightened stress. Hence, continuous cavity nucleation emerges as the dominant mechanism in the martensitic microstructure within high-stress areas. The opposite trend was observed for the ferritic microstructure (Fig. 3c) where an increase in stress corresponded to an increase in the average cavity size, meaning that cavity growth dominated and resulted in cavity coalescence. This highlights an important distinction between the ferritic and tempered martensitic microstructures, where the higher strength of martensite relative to the ferrite limits cavity growth, while in ferrite, cavities grow and eventually coalesce more easily.

It should be noted that the cavity measurements were performed on polished cross-sectional surfaces, and therefore the observed cavities

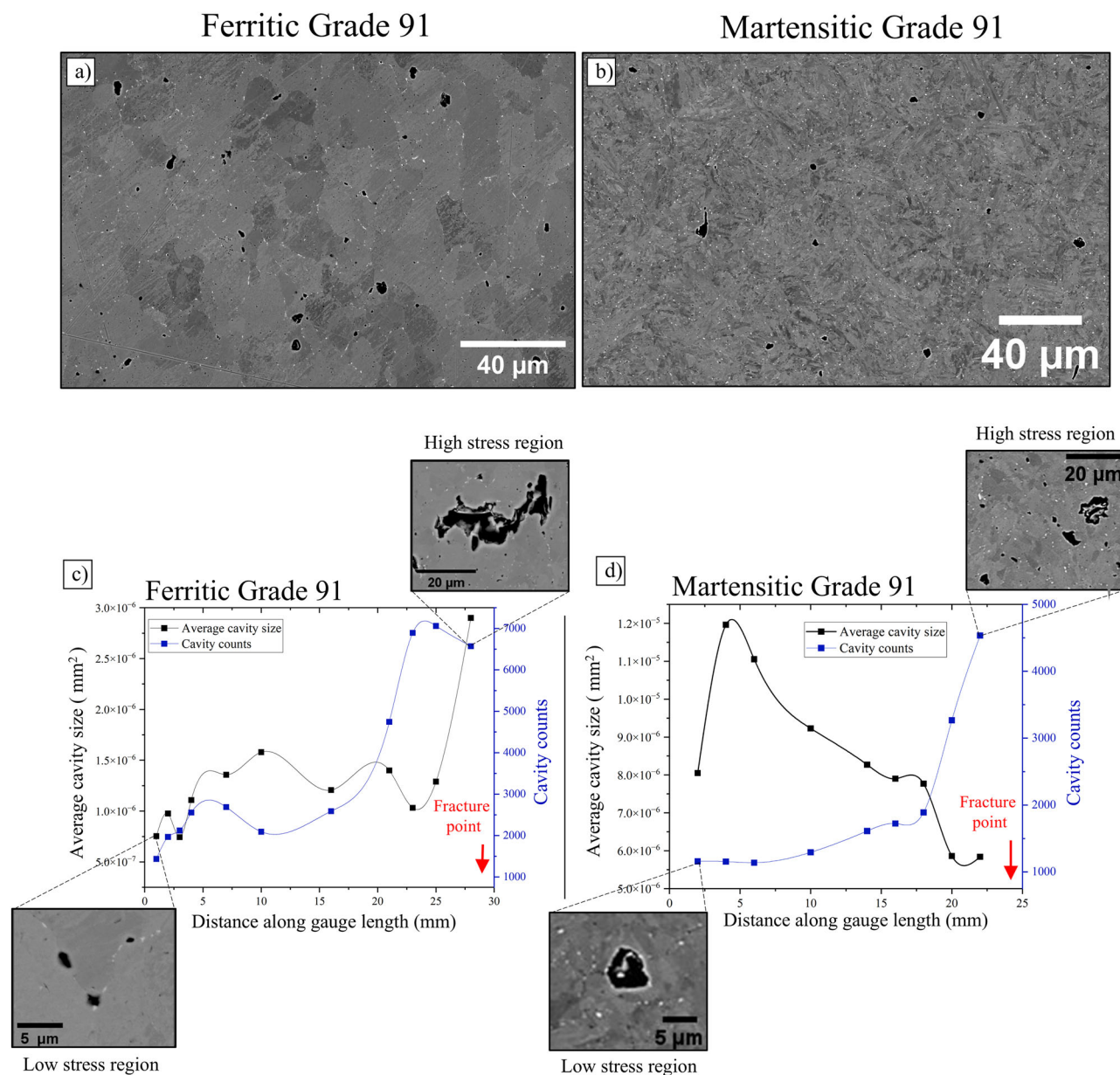


Fig. 3 | Comparison of microstructure and cavity evolution in ferritic and martensitic G91 materials. SEM map of (a) ferritic sample G91-F-35.6% and (b) martensitic sample G91-M-3.7%. Average cavity size along distance of gauge length

of the uniaxial failed creep tested (c) ferritic sample G91-F-35.6% and (d) martensitic sample G91-M-3.7%.

represent a two-dimensional projection of the three-dimensional damage state. Although surface preparation may introduce minor effects on the exposed cavity morphology, identical metallographic procedures were applied to all specimens. Therefore, the measurements remain suitable for comparative analysis between the ferritic and martensitic materials, enabling statistically meaningful evaluation of relative differences in cavity nucleation, growth and interlinkage behavior.

Austenitic 316H steel

For the austenitic stainless steel, the creep test was performed on an ex-service 316H component subjected to long-term thermal exposure in an AGR environment, followed by a notched cyclic stress-relaxation at 550 °C (Table 3). To reduce the influence of the notch-induced stress gradient and the plasticity immediately adjacent to the notch root, the correlative SEM-EBSD approach was applied to a 1.1 mm × 1.1 mm region positioned approximately 1 mm away from the notch surface. Figure 4a shows the stitched SEM map and Fig. 4b the corresponding EBSD map of the same

region of interest (RoI). The overlay of cavity masks with the grain boundary network (Fig. 4c) demonstrates that damage is predominantly intergranular, forming extended, partially coalesced cavity strings along boundary segments. The magnified views (Fig. 4d, e) highlight that, in contrast to the ferritic and martensitic Grade 91 specimens where cavities are comparatively isolated and often inclusion-associated, the 316H microstructure exhibits a more continuous intergranular damage topology. This difference is consistent with a boundary-controlled damage process in which a large fraction of boundaries become active damage sites once microstructural heterogeneity at boundaries (e.g., precipitation, secondary phases) creates local stress concentrations.

A key outcome of the correlative analysis is the strong association between cavitation and heterogeneous plasticity at the grain boundary scale. KAM, used here as an indicator of local lattice curvature and strain accumulation, shows a clear shift towards higher maximum values near cavitated boundaries (Fig. 4f). This result supports a mechanism where cavitation is promoted in grain boundary zones that experience elevated local

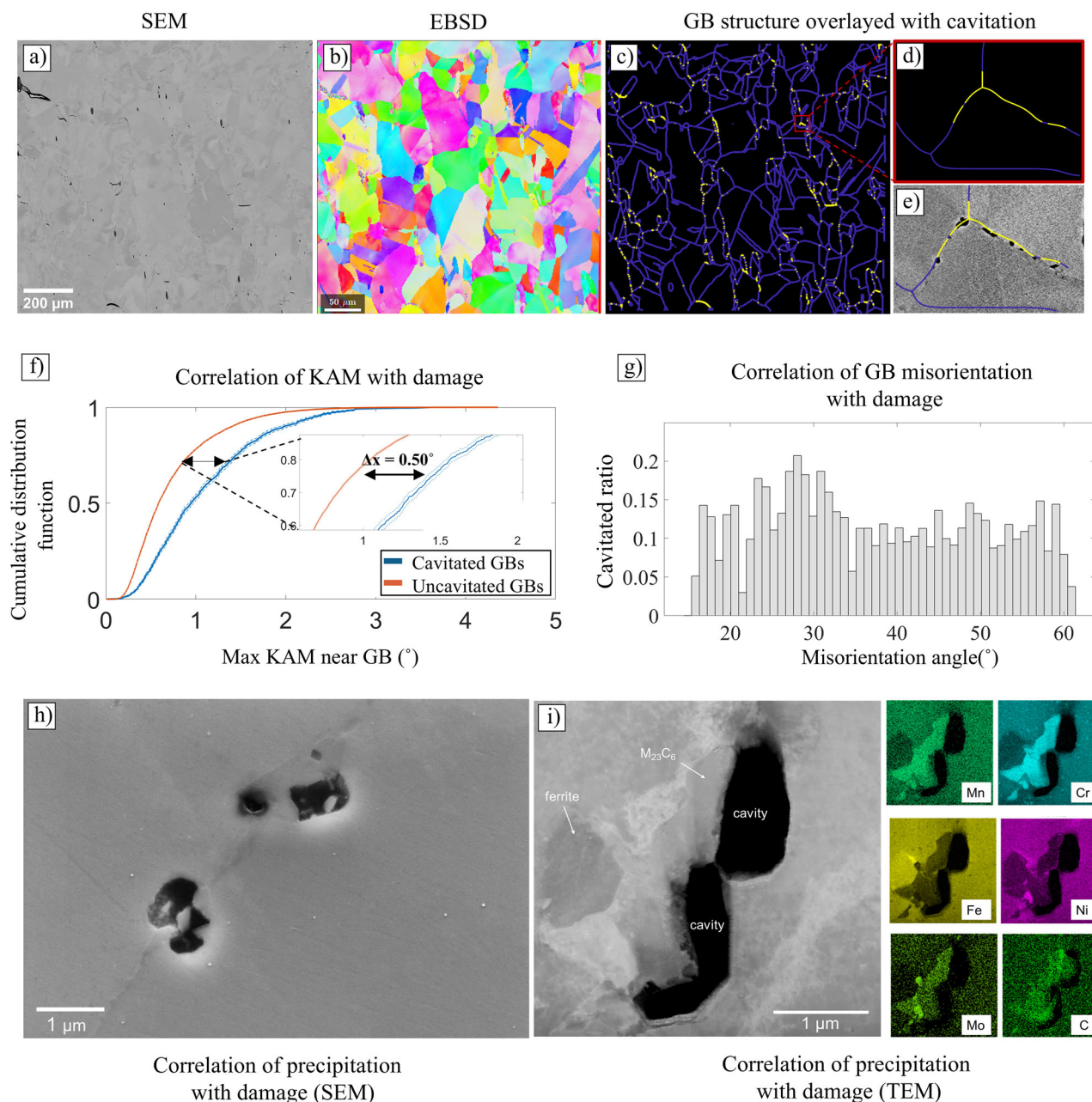


Fig. 4 | Correlative analysis results of austenitic 316H steel. **a** Stitched SEM map of examined region, **(b)** EBSD map, **(c)** cavities (yellow) overlaid on grain boundaries (blue), **(d)** magnified view of **(c)**, **(e)** magnified view of **(c)** overlaid on SEM map, **(f)**

maximum KAM values near GBs for the examined region, **(g)** Cavitation ratio of GBs across all misorientation angles, **(h)** high magnification SEM map exhibiting cavitation related to GBs and precipitation and **(i)** STEM-EDS map.

deformation, rather than being uniformly distributed across the boundary network. From a micromechanical perspective, this behaviour can be interpreted as a consequence of strain incompatibility between adjacent grains and the associated need to accommodate slip transfer and grain boundary sliding. In this sense, KAM is not only a correlator of damage, but also an experimentally accessible indicator for where the microstructure is failing to accommodate deformation compatibly⁴⁵.

In addition to strain localization, grain boundary character and boundary decoration both appear to govern where cavities initiate and how they evolve. The cavitation ratio as a function of boundary misorientation (Fig. 4g) indicates that mid-angle boundaries, particularly in the ~ 20 – 30° range, are disproportionately susceptible to cavitation relative to their frequency in the boundary population. The misorientation influences the boundary structure, diffusion/segregation behaviour and the propensity for precipitate nucleation and continuity along the boundary. If mid-angle

boundaries preferentially accumulate a denser or more continuous precipitation coverage, they would experience stronger local constraint mismatches and more frequent stress concentrators, therefore increasing both nucleation probability and the chance of cavity alignment and coalescence along the same segment⁴⁵.

The microstructural linkage between cavities and grain boundary second phases is directly supported by the high-magnification SEM image (Fig. 4h) and confirmed by TEM/EDS (Fig. 4i). The dominant features associated with cavity initiation are $M_{23}C_6$ carbides co-located with grain boundary ferrite. Cavities commonly nucleate by interfacial decohesion at austenite–carbide, austenite–ferrite or ferrite–carbide interfaces and at multiphase junctions where stress triaxiality is elevated. The strong mismatch in mechanical response between the austenitic matrix, the comparatively ductile ferrite and the brittle carbide phase promotes localized stress development under creep, making these interfaces particularly

vulnerable to separation and vacancy condensation. Importantly, this provides a mechanistic route by which precipitation becomes not merely a microstructural descriptor but an active driver of damage, i.e. precipitates can (i) pin or harden boundary segments, promoting stress concentration in adjacent softer regions, (ii) act as stress raisers due to elastic/plastic incompatibility and (iii) create fast diffusion paths and interfacial sites where vacancies can condense and stabilize a cavity nucleus^{45–47}.

The observed interlinked cavity morphology in 316H can be rationalized as the combined outcome of boundary sliding/strain incompatibility and a precipitate-decorated boundary network that provides repeated nucleation sites and assists cavity growth along connected boundary segments. This behaviour contrasts with Grade 91, where inclusion-induced nucleation dominates early-stage cavitation and where microstructural strengthening features (e.g., martensitic laths) can suppress cavity growth and interlinkage. In 316H, once precipitation establishes a persistent population of interfacial weak points at boundaries, damage progression can transition from discrete nucleation to “stringing” and linkage along susceptible boundaries, consistent with the more continuous intergranular damage seen in Fig. 4d, e.

Overall, the 316H results demonstrate a damage scenario in which (i) intergranular strain localization, (ii) boundary misorientation (as an indicator for boundary structure/segregation propensity) and (iii) extensive grain boundary precipitation jointly control the spatial distribution of cavitation. Conversely, descriptors such as grain size and intragranular orientation-based metrics (e.g., Schmid factor) are expected to be secondary at the scale resolved here, because boundary precipitation and multiphase interfaces dominate the local driving forces for decohesion and vacancy condensation. These observations motivate the inclusion of boundary-resolved indicators (strain localization measures, boundary character and precipitate/secondary-phase decoration state) in microstructure-sensitive cavity nucleation models for austenitic steels, particularly when transferring models across microstructures where the dominant initiation sites differ.

Crystal plasticity simulation results

Figure 5 presents the observed creep damage across the austenitic, ferritic and martensitic steel microstructures, together with the predicted damage sites obtained from both the microstructure-informed classification model outlined in the Methods section and the Dyson–McLean model, which is a physics-based approach describing creep damage as the nucleation, growth and coalescence of grain boundary cavities driven by stress-assisted diffusion and constrained grain boundary sliding, linking cavity evolution to macroscopic creep strain and eventual intergranular fracture⁴⁸. For all materials analysed using the crystal plasticity framework, the microstructure-informed approach shows a substantial improvement in correctly identifying experimentally observed creep damage locations, as shown in Fig. 5a–c. This improvement is consistent with the experimentally observed localization of damage at sites associated with boundary character, second-phase particles and strain incompatibility in each material. Figure 5d shows the normalised F-scores, indicating the relative importance of the crystal plasticity model outputs used within the classification algorithm. The results demonstrate consistent F-scores across materials for each damage indicator except for volumetric strain. In the austenitic stainless steel, volumetric strain contributes significantly more to the classification performance than in the ferritic or martensitic steels. This aligns with the experimental observation that cavity strings in 316H form by boundary opening along $M_{23}C_6$ -decorated interfaces, where triaxial stresses and dilatational separation dominate. The highest-ranked damage indicators across each material were the sum of slip rates and the maximum slip rate. In austenitic stainless steel, these indicators had equal ranking. In ferritic and martensitic steel, the max slip rate contributed more to damage than the sum of slip rates, consistent with experiments showing that MnS-associated cavities in these materials originate from strong local slip mismatch and shear-driven incompatibility along grain boundaries or PAGBs. This suggests that individual slip system activity is more important than interactions between different slip systems when considering damage formation.

The results for austenitic stainless steel show that the microstructure-informed model predicted damage at 15 of 35 locations observed as being damaged experimentally, an improvement on the 4 predicted using the Dyson–McLean model. From the ferritic steel simulations, using the microstructure-informed model over the Dyson–McLean model improved predictions from 3 of 22 to 8 of 22. Finally, the martensitic steel simulations showed similar improvement, increasing from 0 of 8 to 6 of 8 locations correctly predicted. The microstructure-informed model shows greater performance when considering the false positive rate, with far fewer instances of incorrectly predicting damage at locations that did not exhibit damage within the test specimens. It also shows more accuracy in the total cavity count. Furthermore, the microstructure-informed model captured regions with significant damage far better than the Dyson–McLean model.

Certain indicators describe creep damage more effectively than others because they are more directly linked to the physical mechanisms governing cavity nucleation at grain boundaries. Variables associated with tensile normal stress and accumulated plastic deviatoric strain capture the local stress concentrations and deformation modes that promote grain boundary separation and void formation, and therefore correlate more strongly with observed damage locations. In contrast, indicators that reflect more general deformation behaviour and are less closely aligned with the dominant damage processes, exhibit reduced predictive capability. This is evidenced by the lower F-score of the volumetric stress and strain compared to other damage indicators and it is linked to the role of stress and strain triaxiality in creep cavity nucleation.

As discussed earlier in the current section, as well as in the wider scientific literature, creep damage localisation is influenced by microstructural features including carbide precipitates, grain boundary misorientation and second phase inclusions such as MnS. Crystal plasticity finite element (CPFE) frameworks naturally capture the mechanical incompatibility of anisotropic plastic deformation between neighbouring grains and at PAGBs, leading to heterogeneous stress and strain fields even in the absence of explicitly modelled particles. These heterogeneous fields therefore act as mechanical analogues of the experimentally observed damage-initiation sites: high slip activity in regions affected by MnS inclusions in ferritic and martensitic steels and elevated volumetric strain where carbide/ferrite-decorated boundaries dominate in austenitic 316H. When carbide precipitates are introduced explicitly, they act as local stiffness and strength enhancers within this already heterogeneous field, promoting additional stress concentrations and amplifying plastic strain incompatibilities at particle–matrix interfaces. Metallic carbide precipitates have been shown to affect fracture behaviour in crystal plasticity simulations over small numbers of grains⁴⁹; however, in large polycrystal simulations over representative volumes, the mesh resolution is typically insufficient to include such features explicitly. Hence, there remains a lack of models capable of explicitly incorporating the mechanical role of inclusions and precipitates, particularly within large representative polycrystals. In these cases, future developments may incorporate experimentally informed measures of local carbide density or morphology into constitutive descriptions to account for their influence without full geometric resolution. Similarly, the effect of grain misorientation has been investigated through local variations of the critical energy release rate within phase field damage models, improving statistical correlation of damage initiation with experimental observations⁵⁰.

Overview of results

The correlative methodology that was employed in this study, integrating SEM, EBSD, TEM, EDX and advanced data analytics with crystal plasticity simulations, provides a powerful framework for understanding creep damage initiation at the microstructural level. Unlike traditional approaches, this workflow enables direct mapping of cavities onto grain boundary character and correlates damage with inclusions, precipitates and strain localization, offering statistically meaningful insights across different materials. Its versatility and reproducibility make it a valuable tool for

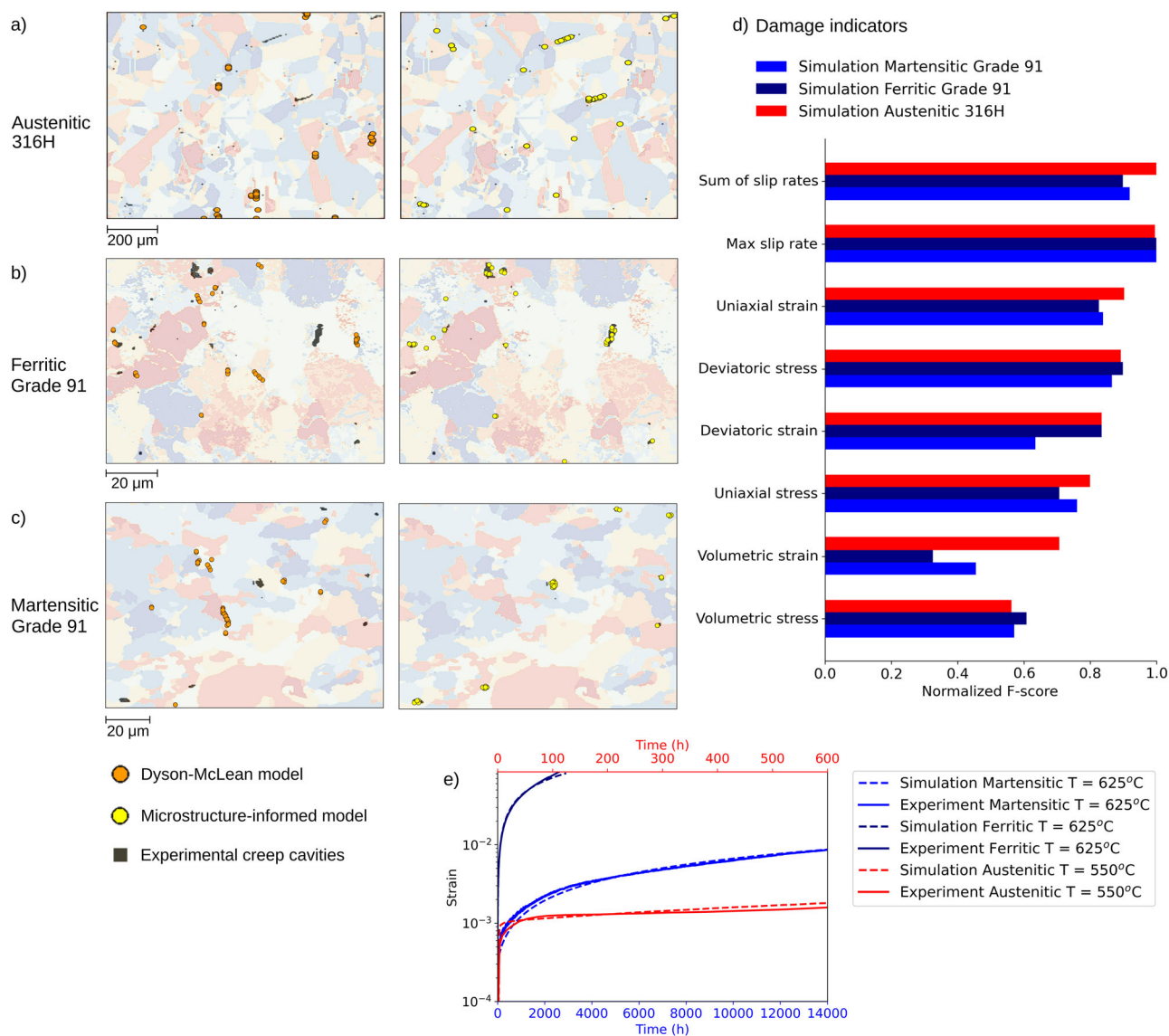


Fig. 5 | Crystal-plasticity assessment of creep damage in austenitic, ferritic and martensitic steels. Results from the crystal plasticity simulations comparing locations of creep damage predictions across (a) austenitic, (b) ferritic and (c) martensitic steels using the microstructure-informed (right) and Dyson-McLean models

(left), (d) the calculated F-scores ranking the relative importance of a number of damage indicators for the three steels simulated are also shown and (e) the creep strain rate is shown for each material tested and simulated using crystal plasticity.

developing microstructure-sensitive predictive models, which are critical for accurate life assessment of high-temperature components.

Ferritic, martensitic and austenitic steels all exhibit creep damage that initiates predominantly at grain boundaries, yet the mechanisms and susceptibility differ markedly. While intergranular cavitation is common to all three, ferritic steel shows high cavity density and rapid growth and interlinkage due to its softer matrix. Low-angle boundaries (15–20°) are particularly vulnerable because they lack stabilizing carbide precipitation and cavities often nucleate at MnS-matrix interfaces where smaller precipitates such as M_{23}C_6 carbides and MX-type precipitates amplify local stress concentrations. In contrast, martensitic steel demonstrates greater resistance, as its lath structure suppresses cavity growth and linkage. Damage occurs mainly along prior austenite grain boundaries, especially low-angle and $\Sigma 3$ boundaries that frequently participate at junctions, where diffusion paths and stress triaxiality are increased. Cavities are strongly associated with MnS and complex $\text{MnS-Al}_2\text{O}_3$ inclusions, but their growth rate remains lower than the rate of continuous nucleation. This results in predominantly isolated cavities and significantly less interlinkage compared to ferritic steel. Austenitic 316H steel differs further by forming extended cavity

strings along grain boundaries decorated with M_{23}C_6 carbides and ferrite. Here, interfacial decohesion at austenite-carbide, austenite-ferrite, ferrite-carbide or multiphase junctions under high triaxial stress drives strain incompatibility, making linkage easier than in martensite and broadly similar in severity to ferrite, although the underlying cavity growth and linkage mechanism differ from that in ferrite. In austenite's case, damage is driven less by matrix softness, as in ferrite, and more by interfacial decohesion and strain incompatibility at austenite-carbide and multiphase junctions. These contrasts underscore how crystal structure and boundary chemistry govern creep resistance across these steels.

The Dyson-McLean model demonstrated better agreement with the experiments when trained using the measured cavity locations. In the crystal-plasticity framework, the strain fields surrounding MnS-associated regions were translated predominantly into increased slip-rate activity, while the strain fields near M_{23}C_6 -decorated boundaries appeared mainly as elevated volumetric strain; this distinction may reflect the comparatively softer response of MnS inclusions, which promotes shear-driven incompatibility, versus the stiffer carbide phases, which tend to amplify normal opening stresses along grain boundaries.

Table 1 | Chemical composition of the studied P91 material

Element	C	Cr	Mn	Mo	Ni	Si	Al	P	S	N
wt%	0.100	8.770	0.410	0.940	0.120	0.410	0.040	0.009	0.010	0.045
Element	Nb	V	Cu	As	Sn	Sb	Zr	Pb	B	O
wt%	0.071	0.210	0.190	0.013	0.008	0.002	< 0.002	0.00075	< 0.0003	< 0.0003

From an industrial perspective, these insights can guide alloy design such as grain boundary engineering, heat treatment optimization and inclusion control strategies to enhance creep performance. By combining experimental observations with physics-based simulations and machine learning classification, this approach bridges the gap between microstructural characterization and engineering-scale predictions, enabling more reliable creep life modeling and supporting the development of digital twins for structural components.

Methods

Materials

The Grade 91 material studied was sourced from the final superheat outlet header of Aberthaw Power Station Unit 8, installed in 1992 and operated for 79,000 hours at 570 °C and 16.5 MPa. After 58,000 h, Type IV cracking was observed, leading to its replacement in 2008. To restore its properties, the material underwent renormalization (1065 °C for 30 min, air cooled) and tempering (765 °C for 2 h, air cooled). For the ferritic Grade 91, an additional heat treatment was applied, involving annealing at 950 °C followed by slow cooling to 760 °C, held for 3 h to transform BCT martensite into softer BCC ferrite. This allows for the evaluation of Grade 91 in both its martensitic and ferritic conditions in the current study. Table 1 lists the chemical composition of the Grade 91 material.

It is important to highlight that the aim of this study was to investigate the microstructural mechanisms responsible for cavitation initiation. To achieve this, materials were examined at both early and advanced creep stages to understand where, when and why cavities nucleate, as well as how they evolve. Accordingly, both interrupted and failed creep tests were performed, resulting in five Grade 91 specimens: one ferritic interrupted at 4% strain, one ferritic that failed at 35.6% strain, one Table. 2 martensitic interrupted at 1% strain, one martensitic that failed at 3.7% strain and one double-notched martensitic that also failed. All specimens, except the failed double-notched martensitic, were uniaxial. The uniaxial tests were conducted at 625 °C under 60 MPa, while the martensitic double-notched specimen was tested at 625 °C and 70 MPa net section stress. The dimensions of the ferritic, martensitic and austenitic specimens are shown in Fig. 6, while the specimens and their corresponding testing details are summarized in Table 3.

The 316H steel studied in this work was extracted from an ex-service Type 316H austenitic stainless steel component in AGR plant, which operated at a temperature between 490 °C and 530 °C for 65,000 hours, provided by EDF Energy Ltd. A notched specimen was machined from the ex-service material for the creep test. The specimen was subjected to 25 cycles of tensile stress relaxation at an initial net-section stress of 390 MPa (calculated using the initial cross-sectional area at the notch) and a temperature of 550 °C. The specimen was first loaded to a net-section stress of 390 MPa and then allowed to relax under pure strain control for 500 h. The procedure is repeated 25 times, leading to a total dwell time of 25 × 500 h.

Techniques

All specimens followed the same preparation method in order to ensure surface finishes that would preserve the surface microstructure and avoid introducing defects on it. A Struers Accutom cutting saw was used to section the specimens and then they were embedded in conductive carbon resin. The initial stage of surface preparation involved grinding the specimens using a Buehler EcoMet 30 Semi-Automatic Grinder-Polisher, progressing through a series of silicon carbide abrasive papers with grit sizes of 400, 600,

1200, 2500 and 4000. Polishing was then carried out using diamond suspensions, beginning with a 3 µm particle size and gradually refining the surface down to 0.25 µm. The final polishing step involved using colloidal silica on polishing cloths for 15 min in the automatic polisher. After polishing, all specimens were thoroughly cleaned with ethanol and isopropanol to eliminate any remaining debris or drying residues.

SEM imaging was implemented to capture large-area images without compromising the high resolution required for accurate feature characterization. To achieve this, for the martensitic material high-resolution sequential images with a field of view of approximately 110 µm × 82.5 µm were acquired at a magnification of 1000×, with 30% overlap between them on a Zeiss SigmaHD FEG-SEM. These images were then stitched together in ImageJ Fiji⁵¹. The final stitched map covered an area of approximately 0.72 mm × 0.43 mm, which, considering the nano- to micro-scale size of the cavities, was sufficiently large to provide statistically meaningful information about the damage present. At the same time, the high resolution of the individual images allowed for clear visualization of cavity shapes and sizes. The average cavity size in the martensitic material was 3.03 µm. The EBSD reconstruction resulted in an average grain size of 10.24 µm, and the correlative analysis was performed using this reconstructed grain structure. Although the martensitic lath structure was significantly finer than the reconstructed grain size, the reconstructed grain structure was selected for the correlative analysis, because the vast majority of cavities were observed on PAGBs. For the ferritic material, due to the presence of many nano-sized cavities, a higher magnification of 2000× was selected and the field of view of the individual images was 55 µm × 41.25 µm. The sequential imaging covered a total area of 0.41 mm × 0.35 mm. This mapped area was selected considering the average ferritic grain size of 11.51 µm and the average cavity size of 2.63 µm, providing sufficient statistical representation of the damage while maintaining the resolution required for accurate cavity characterization. Due to the larger grain size of the austenite grains in the 316H sample, with an average grain size of approximately 50 µm and an average cavity size of 1.50 µm, a total area of 1.2 mm × 1 mm was mapped under a 1000× magnification, to capture more grain boundary segments, as the creep damage was predominantly intergranular. The larger mapped area was therefore selected to ensure adequate sampling of grain boundary damage in the austenitic microstructure.

TEM lamellae were extracted using a ThermoFisher Scios 2 DualBeam FIB system. FIB-SE imaging was performed under a 30 kV voltage. Standard trench milling using the FIB was conducted under a 30 kV voltage and a 65 nA beam current, followed by cleaning/thinning steps by gradually lowering the beam current from 7 nA down to 0.3 nA. STEM imaging and STEM-EDS mapping were carried out with an aberration corrected 200 kV JEOL ARM-200F microscope working at 200 kV acceleration voltage, equipped with a 100 mm² Centurion EDX detector, housed at the David Cockayne Centre for Electron Microscopy, University of Oxford.

For the Grade 91 samples, a Symmetry S3 EBSD detector by Oxford Instruments on a Zeiss SigmaHD FEG-SEM microscope was utilized for EBSD imaging under an accelerating voltage of 30 kV. The Aztec Oxford software assisted in data collection and further processing was executed with the AZtec Crystal software. For the 316H sample, a DigiView 3 EBSD detector by EDAX, was utilized on the same microscope. To improve the signal-to-noise ratio and optimize the pattern quality, the largest available aperture size in the microscope was used (120 µm) for imaging all samples. Further EBSD processing, including parent grain reconstruction and grain

Table 2 | Chemical composition of the studied 316H material

Element	C	Si	Mn	P	S	Cr	Mo	Ni	B	Co	Fe
wt%	0.06	0.40	1.98	0.02	0.014	17.17	2.19	11.83	0.005	0.10	66.23

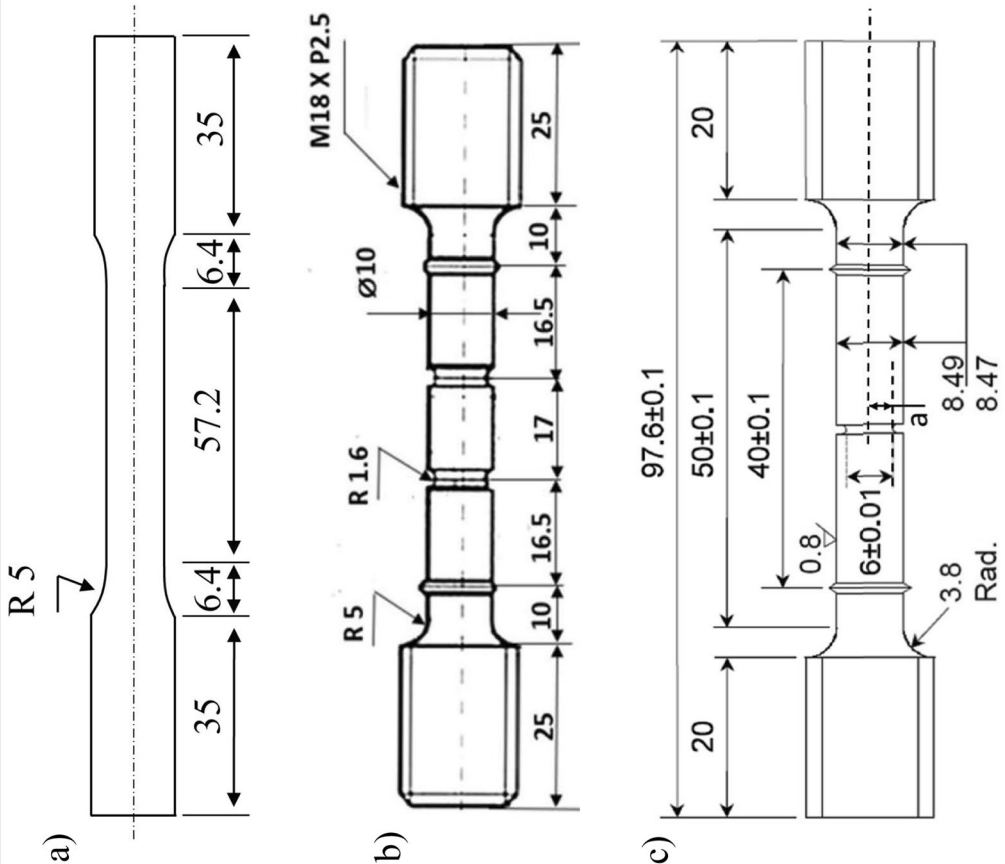


Fig. 6 | Specimen dimensions. Dimensions in mm of the (a) uniaxial ferritic and martensitic, (b) double-notched martensitic and (c) notched austenitic specimens.

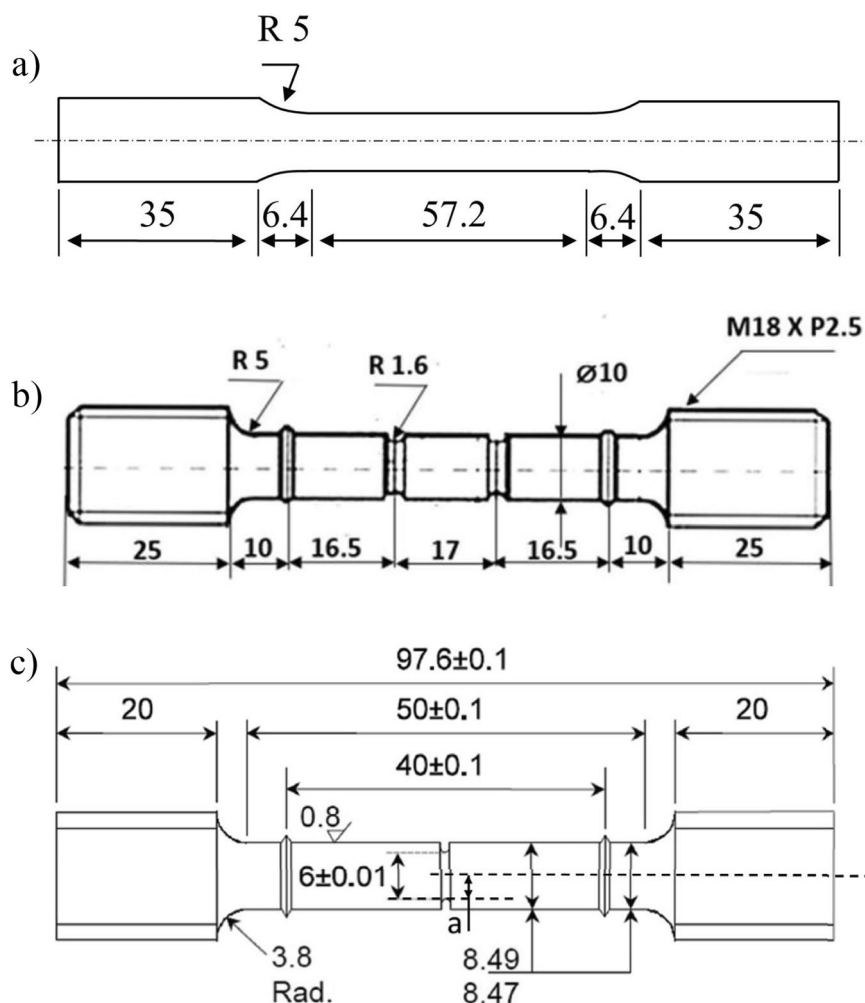


Table 3 | Summary of Grade 91 and 316H samples

Test ID	Crystal structure	Test status	Temperature (°C)	Stress (MPa)	Time (h)	RoA (%)	Strain (%)
G91-F-4%	Ferritic	Interrupted	625	60	3000	–	4.0
G91-F-35.6%	Ferritic	Failed	625	60	5369	52.2	35.6
G91-M-1%	Martensitic	Interrupted	625	60	9003	–	1.0
G91-M-3.7%	Martensitic	Failed	625	60	37,167	3.0	3.7
G91-M-DN	Martensitic	Failed	625	70*	10,355	2.0	–
316H	Austenitic	Interrupted	550	390	10,789	–	–

*Net section stress.

boundary segmentation, was carried out with MTEX, which is a MATLAB® toolbox designed to visualize, process and filter crystallographic data^{52,53}.

Although SEM provides valuable insight into cavity morphology and distribution and EBSD offers detailed crystallographic and grain boundary information of the examined region, using them separately limits the depth of interpretation. When these two datasets are correlated, creep cavities can be projected and visualized on the EBSD maps, yielding a more comprehensive understanding of cavity positioning in relation to the underlying crystallographic context. This integrated view enables the identification of whether damage is located along grain boundaries, whether strain accumulation is present in those regions and what the local slip activity might be. By combining SEM and EBSD data, it becomes possible to explore the microstructural mechanisms associated with cavitation in greater detail.

This approach becomes even more powerful when complemented by high-resolution targeted techniques such as TEM and EDX, which provide additional perspectives on damage at finer scales. The integration of multiple techniques enhances the reliability and accuracy of the conclusions drawn. For this reason, a correlative methodology was employed combining SEM and EBSD datasets using the MTEX toolbox in MATLAB®. The term 'correlative' refers to the integration of complementary characterization techniques across multiple length scales. Correlated SEM, EBSD, and cavity segmentation datasets provide a macroscopic overview of the damage distribution, while high-magnification SEM and SEM-EDS analyses reveal microstructural details, and TEM characterization provides nanoscale insight into the underlying damage mechanisms. This multi-scale approach enables the simultaneous interpretation of damage evolution from the

macro- to the micro- and nanoscale. The SEM–EBSD technique has been described in recent publications^{33,45}.

EBSD is usually performed with the stage tilted around 70°, to maximize the interaction volume of the electron beam and therefore, the number of back-scattered electrons escaping the sample and being detected⁵⁴. This high-angle tilt introduces some distortion in the EBSD maps relative to the SEM maps that are acquired without tilting⁵⁵. To accurately correlate these two datasets, a nonlinear transformation method, known as thin-plate spline (TPS)⁵⁶ was employed. This approach corrected for EBSD distortions by aligning reference crosses that were FIB-milled at the corners of the RoI while the SEM image that was acquired without tilt, served as the distortion-free reference for this point-to-point alignment.

Figure 7 illustrates the SEM–EBSD methodology. Some of the individual high-magnification SEM images (which were stitched together to cover a large area) were randomly selected to manually annotate features that the user confidently recognized as creep cavities. These annotations were used to train a deep-learning segmentation model within the DragonFly software by Object Research Systems [ORS]⁵⁷. Once trained, the model was applied to the full stitched SEM image (Fig. 7a) to segment the cavity masks, quantify them and to extract statistics on cavity density and size¹⁰. The output is a binary map (Fig. 7b) matching the dimensions of the original SEM image, where bright pixels represent cavity locations, effectively filtering out all other microstructural features. This is important for overlaying only the cavity data on the EBSD map. Figure 7c shows the EBSD map acquired with the fiducial markers intentionally included, ensuring alignment with the same region previously imaged using SEM. The EBSD map was processed using the MTEX toolbox to segment the grain boundary structure within the RoI and to calculate the misorientation angles of each grain boundary segment (Fig. 7d). By combining the binary cavity segmentation map (Fig. 7b) with the grain boundary misorientation data (Fig. 7d), it was possible to identify the precise locations of intergranular damage and associate each damaged segment with its corresponding misorientation angle. In the resulting overlay (Fig. 7e), yellow color indicates cavitated grain boundary segments, while blue color represents the uncavitated ones. This method enables the extraction of statistical data regarding the prevalence of specific grain boundary types across the three materials studied, as well as their susceptibility to creep cavitation. This data can be valuable information for grain boundary engineering models. To achieve this, the misorientation angle range between 15° and 62.8° of grain boundaries was separated into distinct misorientation angle bins to evaluate which misorientation angles are more frequently associated with cavitation. Low-angle boundaries with misorientation angles below 15° were not included in the analysis, as no damage was found on them, which was expected. The grain boundary misorientations have maximum values of 62.8° and the mathematical derivation and explanation for this upper bound was first provided by Mackenzie⁵⁸. In crystallography, misorientation refers to the rotation required to align the crystal lattices of two grains and in cubic systems, due to their 24 distinct rotational symmetries, many orientations are equivalent, resulting in a symmetry-reduced space for defining unique misorientations. Within this reduced space, 62.8° has been analytically established as the maximum possible misorientation angle, representing the largest distinct rotation between two grains before symmetry operations map it back into the fundamental region.

The intermediate images in Fig. 7 depict the same process but on a higher magnification, offering an insight into the accuracy and precision of the SEM–EBSD correlation, that the DragonFly segmentation and MTEX analysis is able to provide. The dataset shown in Fig. 7 was acquired solely for illustrative purposes, to demonstrate the correlative SEM–EBSD methodology and does not represent the primary data used for analysis. The regions selected for detailed analysis were chosen to be away from notches and the specimen's failure edge, ensuring that the local stress remained moderate. This allowed the investigation to focus on early-stage cavitation rather than advanced cavity interlinkage or crack propagation and these regions will be discussed in detail in the following section.

Crystal plasticity and classification algorithm framework

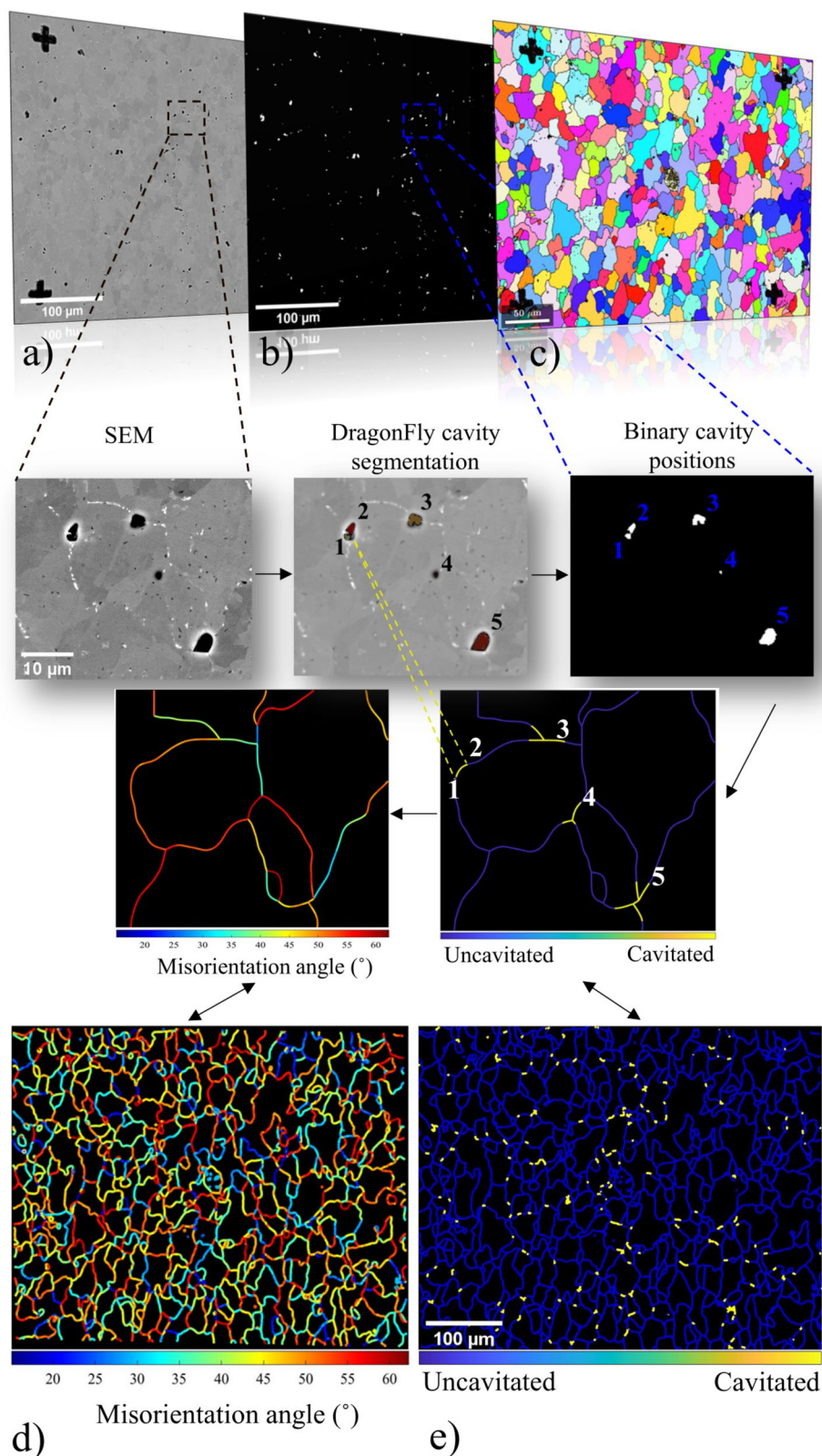
Crystal plasticity finite element simulations have been carried out using a Kalidindi-type finite strain return mapping algorithm to determine the elastic and plastic deformation gradient⁵⁹. Dislocation-based constitutive models, used to calculate the slip rate in each slip system, are used for both austenite and ferrite. These models include 12 and 24 slip systems, respectively, together with specific dislocation multiplication and annihilation laws^{60,61}. The mean free path and annihilation distance for the dislocations are the main parameters determining the hardening behaviour. The critical resolved shear stress of each slip system is determined using a Taylor-type dislocation-based hardening law with an interaction matrix between slip systems provided by previous studies using discrete dislocation dynamics and single crystal experiments^{62,63}. The slip rate in each slip system is determined using a combined constitutive relation comprising two stress-dependent power-law terms, which respectively represent the contributions of plastic slip and creep deformation mechanisms. The dislocation-based model additionally includes slip gradients and the resulting geometrically necessary dislocation densities, contributing to hardening⁶⁴. This feature is essential to accurately capture stress concentrations near grain boundaries, which is particularly important in the present study, as the main focus is on modeling formation and evolution of intergranular creep cavities.

The representative volumes for the simulations are reconstructed starting from the EBSD data one-to-one, i.e., each pixel of the experimental EBSD map is translated into a single hexahedral element of the simulation mesh, which is in turn assigned the corresponding three Euler angles. The dimensions of the simulation domain are determined by balancing the computational feasibility of the calibration procedure with the required level of accuracy, ensuring that a statistically sufficient number of grains is included. The characteristic length of the representative volume is 1000 µm for the austenitic phase, 120 µm for the ferritic phase and 140 µm for the martensitic phase, due to the different resolutions by which the EBSD measurements were acquired, as shown in the Results and Discussion section. The martensitic microstructure is simulated using the reconstructed prior austenitic grains instead of including the small-scale laths directly in the simulations. This is motivated by the experimental observation of creep cavities at the prior austenite grain boundaries. 3D simulations reproducing the EBSD region in plane stress conditions are performed with pressure boundary conditions and temperatures that are representative of the experimental setup described in Table 3. Details about the calibrated parameters of the constitutive model for austenite are available in⁵⁰, while in the current study, the focus is on extending the calibration methodology to austenitic, ferritic and martensitic microstructure. Crystal plasticity simulations have been executed using the MOOSE Framework, developed at Idaho National Laboratory⁶⁵.

Crystal plasticity model parameters governing plastic yield, hardening behaviour and creep strain rate were calibrated using a SciPy-based Nelder–Mead optimization algorithm in two stages. In the first stage, the instantaneous load-up response was calibrated by comparing the simulated stress–strain curve from the crystal plasticity model with macroscopic test data for 316H stainless steel and P91 ferritic–martensitic steel at 550 °C and 625 °C respectively. Elastic constants were taken from published literature at the corresponding temperatures, while parameters such as the statistically stored dislocation density and lattice friction were adjusted to reproduce the experimental yield stress and ultimate tensile strength. Once the load-up behaviour was satisfactorily captured, the second stage focused on calibrating the time-dependent response. The creep strain rate was tuned by comparing the simulated creep strain evolution with experimental creep data. The Nelder–Mead algorithm iteratively minimized the error between experimental and simulated responses by evaluating successive parameter sets with a gradient-based search method until a specified convergence criterion was met.

A classification algorithm based on gradient boosted decision trees, implemented using the XGBoost library, is used to rank the crystal plasticity model state variables according to their relative contribution to the experimentally observed damage. State variables, such as slip rates on

Fig. 7 | Schematic illustration of the correlative MTEX SEM-EBSD technique. **a** Large scale SEM stitched map, **(b)** binary map indicating cavity locations upon DragonFly segmentation, **(c)** EBSD map, **(d)** grain boundary misorientation angle map and **(e)** grain boundary map indicating the uncavitated grain boundary lengths (blue) and cavitated grain boundary segments (yellow) upon MTEX correlation between the SEM and EBSD datasets. The intermediate images depict the same technique, but on a higher magnification.



individual slip systems, stress and strain tensor components are extracted from the final simulation time step, in each element of the mesh, corresponding to the same deformation stage as the microstructure observed by microscopy. Significant quantities and invariants are calculated, such as the sum of the slip rates on each slip system, the maximum slip rate among the slip systems, deviatoric, volumetric and uniaxial stress and strain invariants.

These quantities are available for each element in the mesh and a corresponding data structure is built that contains binary information from experiments indicating whether each element corresponds to a damaged region. The corresponding dataset is divided into training data, comprising approximately 70% of the elements, and test data, comprising the remaining 30%. Care is taken to ensure that both the training and test datasets contain a

large enough representative number of damaged and undamaged regions. After XGBoost decision tree is fully trained, the normalized F-score can be plotted for each of the analyzed simulated state variables: the normalized F-score is a measure used to evaluate the performance of a classification algorithm and it combines precision and recall into a single metric. Precision indicates the proportion of true positive predictions, i.e., of all the elements predicted as creep cavities, how many appears as cavities in the experiment; while recall indicates the proportion of true positive predictions out of all actual positive instances, i.e., total number of damaged elements in the experiment. The F-score is the harmonic mean of precision and recall. The application of such a classification algorithm is important for identifying the origin of creep cavities in different microstructures. A higher F-score for a given simulated state variable indicates that this variable is a better predictor of damage, thus providing insight into the underlying damage mechanism, for example, whether damage is stress-controlled, controlled by plastic deformation on the slip systems or controlled by the total strain. Moreover, this approach enables a meaningful comparison between creep cavity formation in austenitic, ferritic and martensitic phases.

Data availability

The datasets generated and/or analyzed during the current study are not publicly available due to their large size and the requirement for specialized software to access and process them, but are available from the corresponding author on reasonable request.

Code availability

The crystal plasticity software is published as open-source code and it is available in GitHub⁶⁶.

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Author contributions

E.C. Galliopolou: Writing– review & editing, Writing– original draft, Methodology, Investigation, Formal analysis, Conceptualization. S. He: Writing– original draft, Resources, Methodology, Investigation. M. Salvini: Writing– original draft, Resources, Modelling. N. Grilli: Writing– original draft, Resources, Modelling. G.T. Martinez: Resources, Investigation. H. Shang: Resources, Methodology. A. Cocks: Supervision, Methodology. J. Siefert: Supervision, Methodology, Funding. J.D. Parker: Supervision, Methodology, Funding. T.L. Martin: Writing– review & editing, Writing– original draft, Supervision, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization.

Competing interests

The authors declare no competing interests.

Additional information

Correspondence and requests for materials should be addressed to Eirini C. Galliopolou or Tomas L. Martin.

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