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COMPONENT RELEVANT CREEP DAMAGE IN TEMPERED MARTENSITIC 9 TO 12 %CR STEELS

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ABSTRACT

Creep brittle behaviour in tempered martensitic, creep strength enhanced ferritic (CSEF) steels is linked to the formation of micro voids. Details of the number of voids formed, and the tendency for reductions in creep strain to fracture are different for the different CSEF steels. However, it appears that, the susceptibility for void nucleation is related to the presence of trace elements and hard non-metallic inclusions in the base steel. A key factor in determining whether the inclusions present will nucleate voids is the particle size. Thus, only inclusions of a sufficient size (the critical inclusion size is directly linked to the creep stress) will act directly as nucleation sites. This paper compares results from traditional uniaxial laboratory creep testing with data obtained under multiaxial conditions. The need to understand and quantify how metallurgical and structural factors interact to influence creep damage and cracking is discussed and the significant benefits available through the use of high quality steel making and fabrication procedures are highlighted. Details of component behaviour are considered as part of well-engineered, Damage Tolerant, design methods.

1. INTRODUCTION

For over 20 years, EPRI has facilitated research related to the performance of CSEF steels, in general, and Grade 91, in particular. In-service problems continue to be documented [1,2], see for examples damage developed in a stub tube to header weld. Review of service experience supported by detailed root cause analysis has identified that problems have been primarily concerned with:

- Poor component designs,
- Metallurgical Risk - Composition sometimes linked to incorrectly heat treated steel, and
- Weld performance and HAZ cracking.

A simple take away from EPRI research on CSEF steels is that these steels are metallurgically more complicated than the traditional low alloy steels used in boilers and piping. However, the necessary understanding of how the specific metallurgical complexities influence component behaviour has been lacking. Indeed, one major challenge faced by designers and users alike is that component behaviour is influence by stress state effects yet the vast majority of research is carried out under simple loading conditions using small smooth bar laboratory sized specimens. The fact then that there are both metallurgical challenges and influences of multiaxial stress states means that only by following comprehensive research which integrates careful planning with controlled execution can real progress be achieved. EPRI has been facilitating integrated research studies examining these effects and an extensive document base is available [3].

Technology transfer and training in key aspects related to the use of CSEF steels in general and Grade 91 steel in particular is not easy. It is inevitable that in some situations lessons will be learned through difficult experiences involving either undetected damage resulting in failures or through inspections which lead to false positives. Both of these situations are costly and can be avoided with the correct knowledge and planning. The present paper seeks to illustrate key elements which influence the creep behaviour of CSEF steel components by considering specific representation information. The information is by necessity concise and the results selective but, in each case, the controlling influences are supported by specific evidence and appropriate references.

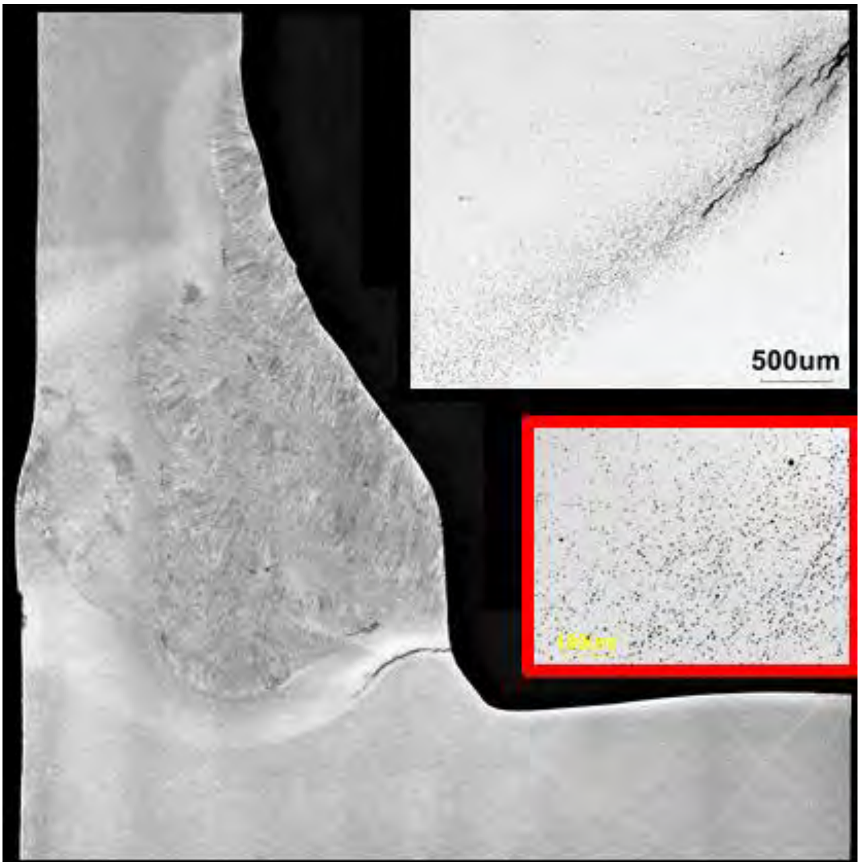

Figure 1. Metallographic section showing creep damage that developed during service in the HAZ of a tube to header weld. The insets show detail of the microdamage that was present ahead of the primary crack. As is typical with creep damage the most susceptible microstructural region shows the highest damage levels. In this specific case susceptibility for creep damage was linked to both local metallurgy and stress state.

2. ESTABLISHING VALID TECHNIQUES FOR CHARACTERIZATION OF CREEP VOIDS

It is apparent that some of the variation in reported creep void densities from the experimental studies is a consequence of differences in the experimental methods used. Differences in specimen preparation, polishing, etching, the type and magnification of examination can all influence the experimental results. Indeed, details of the software packages used for data capture and analysis will also affect the outcomes. Thus, it is important that specific details of the methods used to achieve representative results are fully established and validated. EPRI has taken the initiative in these efforts and several publications detailing approaches for preparation and characterization have been reported [4, 5]. The following summary is extracted from this research, however for complete background and details it is recommended that the original documents should be reviewed.

2.1 Sample Preparation issues

Previous research on cross weld creep testing has shown that it is important that posttest examination is performed on this mid thickness plane [4] , as damage at the specimen surface is not properly representative of true damage levels present.

Metallographic preparation was performed using a sequential, automated polishing technique. The following abrasive wet/dry grits were utilized to produce samples acceptable for examination: 120 grit silicon carbide, 320 grit silicon carbide, 600 grit silicon carbide, 3 μm high performance diamond, 1 μm high performance diamond and 0.04 μm colloidal silica. Etching was performed after polishing using Villella's reagent to reveal the macro structure of the weldment for reporting. However, the sample is then re-polished for examination using laser microscopy so as to limit the potential interference of "false" creep cavities that may develop from the etching process. Thus, although a macro image in the etched condition is provided in the results condition, this does not represent the as-analyzed condition.

2.2 Documentation of Creep Voids

Polished macro samples are examined using a Keyence VK-X105 Confocal Laser Microscope. At EPRI this microscope is mounted to a precision stage with a maximum travel distance of 100 mm in both the X and Y orientations. The Keyence microscope utilizes two pieces of software which are important to the analysis of as-obtained images: The VK Image Stitching Software and the VK Image Analyzer Software. The VK Image Stitching Software merges the collected images into a single compiled image of selected magnification. In general, the series of images are overlapped by ~12 to 15% to ensure proper merging into a larger, compiled image.

A 20X objective was utilized for the analysis of creep cavitation in the Grade 91 HAZ This magnitude of objective provides a magnification of ~400X on a 15 inch monitor [20]. As discussed previously [5], the magnification is not the only critical variable in the assessment of creep cavitation since the number of pixels in the obtained image can also be altered. Both of these factors influence the minimum void size detected.

3. FACTORS AFFECTING THE TENDENCY FOR CREEP VOID DEVELOPMENT

The validated procedures for sample preparation and creep void characterization underpin an extensive library of damage which has been characterized from:

- Laboratory tests on typical small specimens,

- Feature tests performed under controlled conditions, and
- Assessment of damage generated in-service by components.

The results obtained have been fundamental to separating the contributions to damage susceptibility of different factors. Thus, selected samples have been evaluated where different creep stress and temperature were used on the same steels and geometries. These tests thus permit an assessment of exposure conditions on behaviour. Analysis of these results clearly establish that stress and temperature conditions must be selected to ensure that the results obtained are relevant to the damage processes expected in plant. These influences are discussed more fully later in this paper.

3.1. What are the Metallurgical Effects

The results of creep tests from different steels tested using similar geometries and conditions have been examined. Variations in creep cavitation behaviour in these comparisons are clear evidence of the influence of inherent steel metallurgy on behaviour. These effects are illustrated with reference to the micrographs shown in Figure 2. These laser microscope images show cavitation in the HAZ of stub tube welds from the same steam header. This component had operated for about 80,000 hours at temperatures around 580^{o}C [6]. The number density and size of the voids revealed at the same magnification is different for these two samples. Thus, for example, in Figure 2A the number density of voids is over 2000 per mm^2. In contrast the voids density in Figure 2B is around 500 per mm^2. These differences are consistent with variations in the density of inclusions and the level of trace elements in the base steels. Details of local concentrations of trace elements at AlN inclusions are shown in Figure 3.

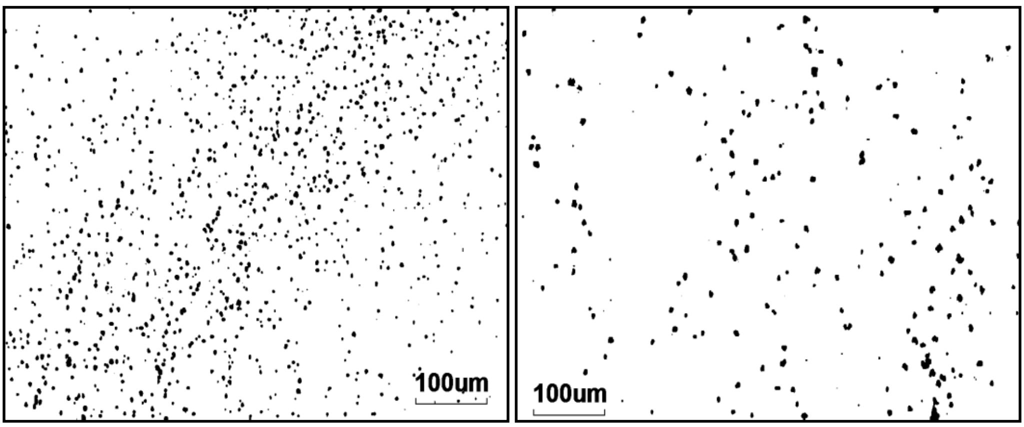

Figure 2. Laser micrographics taken from the HAZ of stub tube to header welds from the same component. In these micrographs the images were recorded using EPRI validated preparation and characterization techniques [5]. The base steel in 2A is identified as B2 with the base metal in 2B from the forged tee piece.

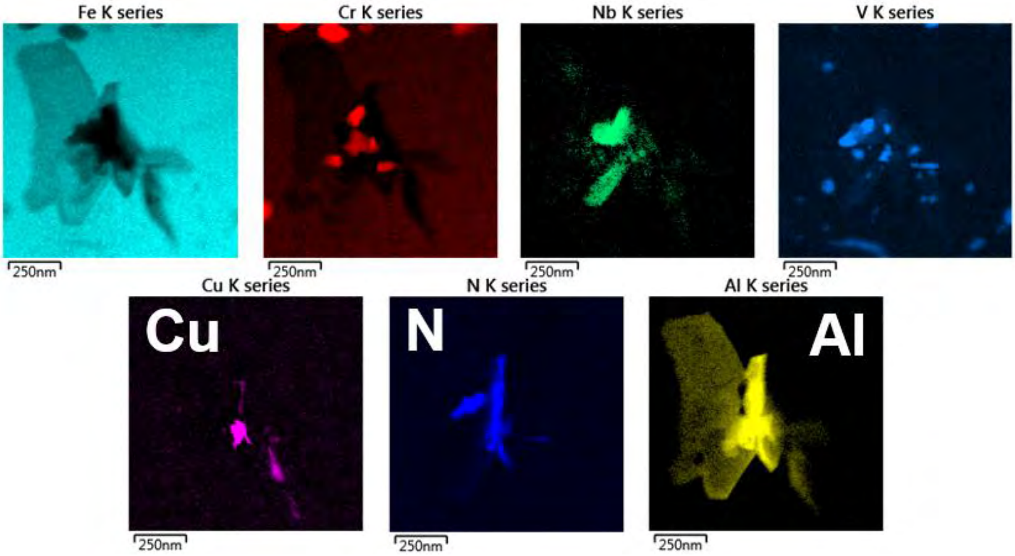

Figure 3. Detailed micrograph showing inclusions associated with an individual creep cavity followed by compositional maps showing the distribution of the elements Cr, Nb, V, Cu, N and Al associated with this location [7].

An independent 'blind' metallographic assessment was also undertaken to document the inclusion content in selected Grade 91 steels. The approach involved scanning as polished sections using an automated SEM-EDS system. This system ensured that over 1,000 inclusions (and up to 4,000) were analyzed and classified for each of the materials labeled B1, B2, B3 and B4 in Figure 4. Detailed recording showed that over 12 different types of inclusions were identified. Importantly, analysis of steel TP1 showed that it contained the lowest number fraction of inclusions <250, i.e. nearly 4X lower than the next lowest number fraction heat of Grade 91 steel. The results in the form of ternary phase diagrams are shown in Figure 4. In each diagram, the inclusions were classified into three general types as follows: Mn-bearing, Al+Si-bearing or Ca-bearing.

It is apparent that even for the steels supplied at the same time from the same fabricator, differences in inclusion type were identified. For example, the lowest ductility heats B2 (26.5% ROA) and B4 (35.5% ROA), exhibited a higher concentration of Mn-bearing (i.e. MnS) and Al+Si-bearing (i.e. alumina or silica) inclusions. The base steel with good creep ductility, B1, showed a distribution of inclusions evenly balanced between Ca-bearing (i.e. complex inclusions from the Ca-refinement step) and Al+Si-bearing. Most notably, B1 shows few Mn-bearing inclusions. For B3 (67% ROA) showed a distribution of inclusions that is between that of the lowest ductility heats and that of the high ductility heat B1 (76.5% ROA).

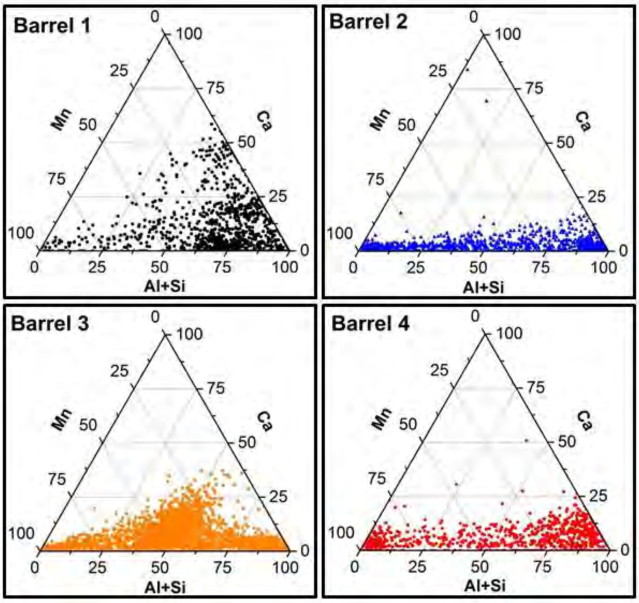

Figure 4. Inclusion Analysis data shown using a Ternary Phase Diagram Approach for Barrel 1 (B1), Barrel 2 (B2), Barrel 3 (B3) and Barrel 4 (B4). Results for Tee Piece 1 (TP1) are not shown as this steel contained a very low number fraction of inclusions.

The inclusion analysis provides evidence that specific types of inclusions could be responsible for creep damage susceptibility. Indeed, MnS has been shown to be harmful with respect to high temperature ductility for low alloy CrMo and CrMoV type steels [8]. Inclusions can nucleate creep voids directly but can also act as trap sites for tramp elements such as Sn, Sb, As, Pb or Cu. Thus, these sites can locally increase the concentration of these deleterious elements, Figure 3. High densities of inclusions, in combination with trace elements, can then act as a pre-mature cavitation site in components operating under high temperature creep applications. Similar work on Grade 92 steel has also identified that inclusions (Mainly BN, MnS and Al3O3) were associated with creep void formation, Figure 5 [9].

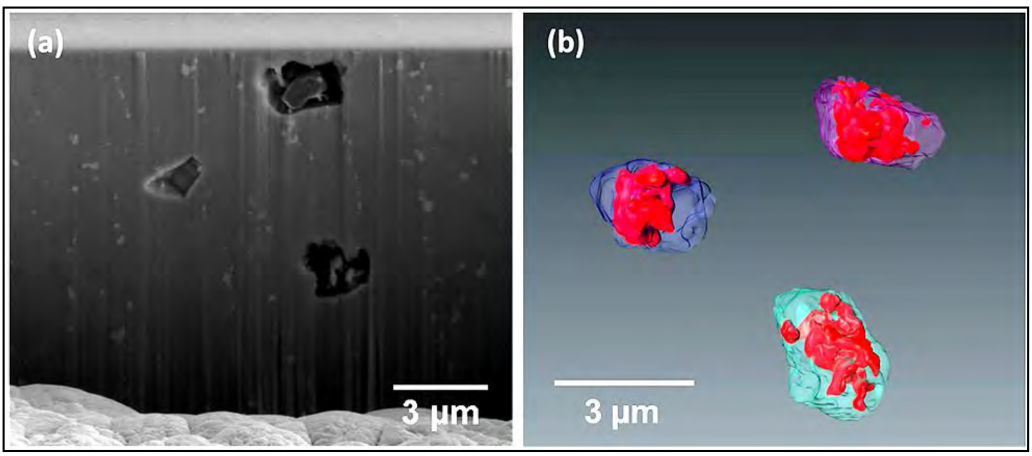

Figure 5. A process involving ion beam sectioning, data capture and image reconstruction allowed details of the creep voids size shape and relation to inclusions to be established [9].

The factors affecting creep void development in Grade 92 steel established by the EPRI research are consistent with results published from independent studies, Figure 6. Compilation of the results from smooth bar specimens showed that:

- Samples tested at 650°C with creep rupture lives of less than 10,000 hours generally showed a visible neck, i.e. reduction in area above 50%. In contrast, tests of longer duration fractured with low values of reduction of area,
- Samples tested at 550oC invariably showed reduction of area above 50%, and
- The behaviour recorded at 600°C was more varied. Thus, some very long term tests at 600°C exhibited ductility >50%.

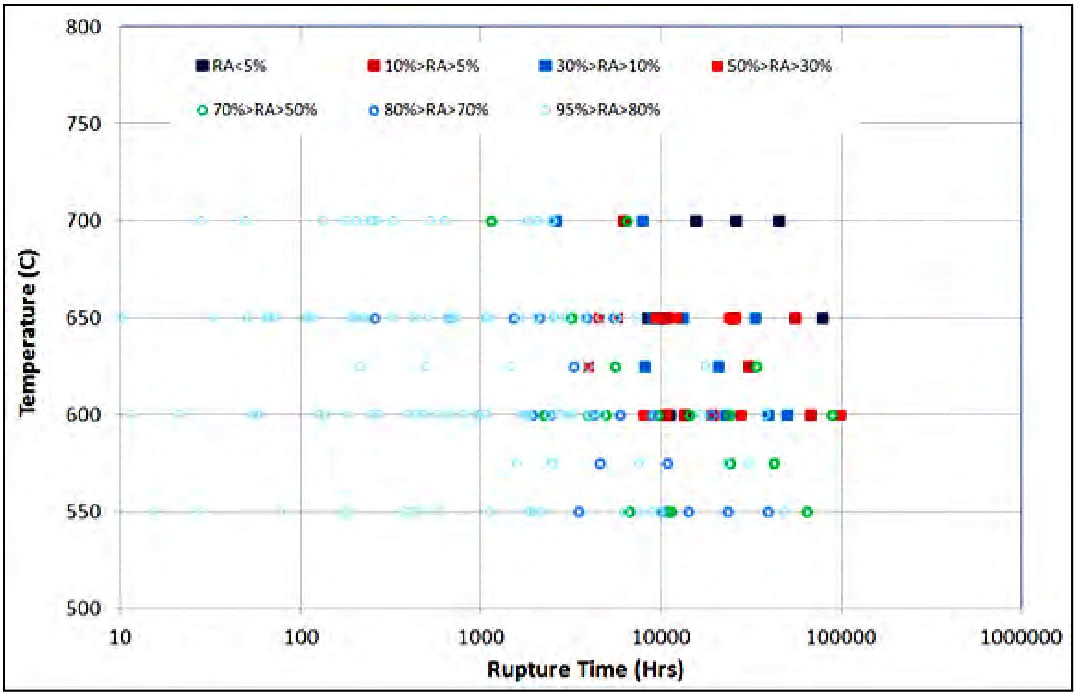

Figure 6. Trend in reduction of area measured following creep tests for different ranges of temperature and rupture time. Open symbols are data for tests where the RoA was greater than 50 % with closed symbols showing tests where the RoA was less than 50%.

The number of cavities at fracture generally increased with creep life for creep tests at both 600° C and 650° C It has been suggested that the tendency for creep cavity nucleation maybe linked to the formation of Laves Phase. Thus, pre-aging to form Laves Phase prior to creep testing might be expected to enhance cavitation and reduce life (and ductility). Experiments at 650° C showed no significant change in the amount of creep damage (number of cavities, size of cavities assessed using back scattered electron microscopy) in thermally aged specimens compared to as-received Grade 92 steel creep tested for similar durations [10].

3.2. When during the creep life do creep voids form?

There has been some conflicting evidence reported regarding when creep voids form prior during creep. Several studies have reported that voids can only be identified relatively late in creep life.In contrast EPRI research has shown unambiguously that creep voids can nucleate relatively early in the creep life. There are several reasons which can be used to rationalize these apparent conflicting observations. These are summarized as:

- Under conditions where the stress state is uniform across the section, it is apparent that the creep voids are initiated at locations below the component surface [4]. Thus, even a properly performed examination at the surface of a weld will often under estimate the extent of the creep damage present.
- For the selected creep testing conditions the steels used may not develop creep voids, however this cannot be taken to represent that these steels will not develop cavities under all conditions nor not all steels will show the same susceptibility to when cavities form,
- The preparation and / or the evaluation methods used may be unable to properly identify early stages of creep void development.

The results in Figure 7 show that when inclusion particles are present in the steel these act as cavity nucleation sites [11]. It is apparent that the tendency for nucleation will depend on factors such are the size and shape of the inclusion, the interface between the inclusion and the steel and the local creep conditions. Thus, the stability of a specific nucleation site will be inversely proportional to the local stress. The progression in Figure 7 shows decohesion at the inclusion interface even in Grade 92 steel prior to creep. These cavities grew as creep increased.

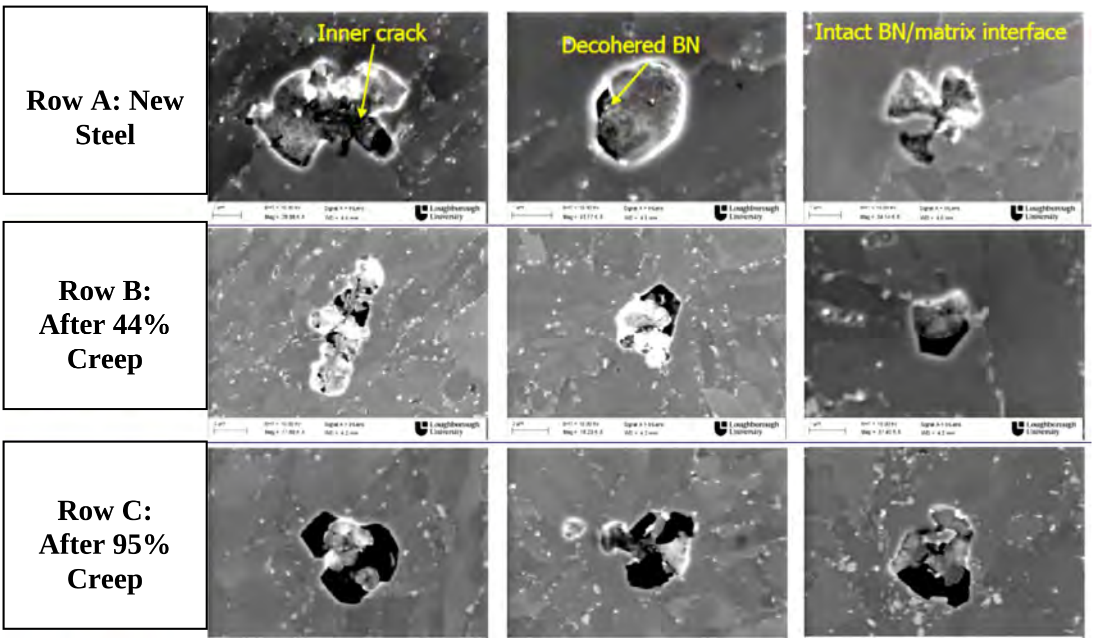

Figure 7. Examples of creep voids forming at inclusions in Grade 92 steel. Row A are examples of steel prior to creep, row B is an example of creep cavities present at about 44% life fraction and row C shows examples of creep cavities at a life fraction of about 95% [11].

It has been shown that there is a general trend for the number density of creep voids present in martensitic steels to increase with creep life fraction, Figure 8 [12]. As shown in Figure 7, it appears that in most cases void nucleation is facilitated by the presence of 'hard' particles such as inclusions within the steel. The mechanism of cavity growth is linked to local creep strain, with the growth or dilation rate of individual voids considered to be constrained by deformation of the local matrix. Under these conditions the following relationship provides a reasonable description of the development of creep damage. In this expression the life fraction is related to the number fraction of voids as:

$$\frac{t}{t_r} = \left[1 - \left(1 - \frac{N}{N_f}\right)^{\lambda}\right]$$

where N is the number of voids per mm^2, N_f is the number of voids per mm^2 at fracture and λ is a function of tertiary creep strain. Thus, a higher value of λ is calculated for steels and conditions which exhibit greater tertiary creep.

The values of these parameters influence the form of the relationship between the number of voids and creep life. The way in which the value of key parameters relates to damage rate is illustrated in Figure 8. Both of the lines drawn in this Figure have been based on the same number of voids at fracture, namely an N_f of 1200 voids mm-2. However, the calculations of the relationship between number of creep voids and life fraction have been made for different values of λ. As λ increases from 2 to 3, the curve defining the rate of change in void density becomes noticeably steeper; this is particularly true as the life fraction increases. Thus, it is interesting to note that for a void density of ~600 voids/mm2, the calculated life fraction range is 70 to 90%, Figure 8. This range approximately bounds the experimental data. At or very close to 100% of life fraction, the void density values vary from 500 to >1150 voids/mm^2. This variability may then be a consequence of differences in the techniques used or as a result of different tendencies for creep void nucleation in the steels tested. Often it is impossible to judge these influences since details of the experimental methods used and/ or information regarding the steel pedigree is not provided. What is apparent is that meaningful assessment of component integrity using observations of creep void development can only be carried out when knowledge of how steel composition and microstructure change N_f and λ have been established.

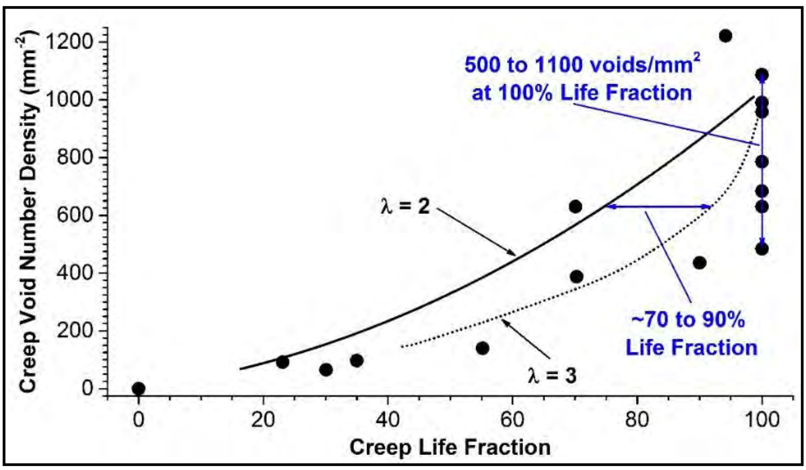

Figure 8. Relationship showing how the density of creep voids present in tempered martensitic steels increases with life fraction. The individual data points show results obtained from specific tests. It is apparent that even at or very close to fracture the number density of creep voids varies between about 500 and 1100 voids per mm^2.

3.3. *How does stress state effect damage development?*

It is apparent that multiaxial stress states can modify creep deformation and fracture behaviour. Thus, behaviour in components, which in the majority of cases takes place under multiaxial conditions, can be very different to that recorded in the laboratory for small, smooth bar samples loaded in uniaxial tension. It is thus very important to understand the influence of different stress states on creep behaviour. For many creep resistant steels, enhanced maximum principal or increased triaxial stress states will reduce ductility and promote cavity development. Thus, creep cavitation can develop earlier when high triaxial stress states are imposed compared to simple uniaxial loading. This behaviour is clearly shown with reference to comparison of smooth bar and notch bar testing of Grade 92 steel at 600°C, Figure 9.

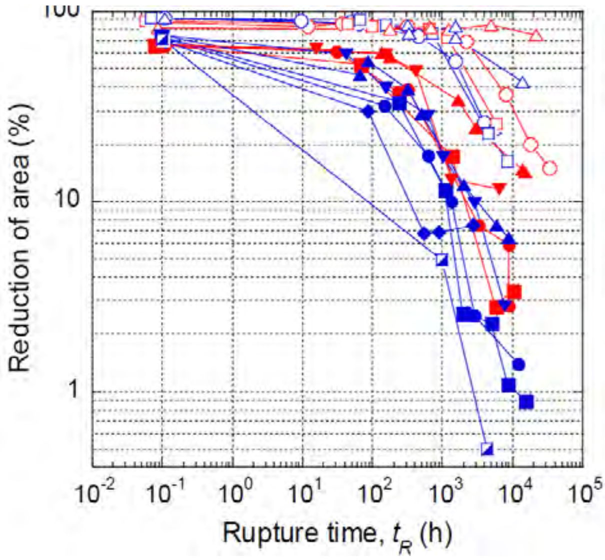

Figure 9. Comparison of the reduction of area for notch bar and smooth bar tests on the same Grade 92 steels at 600°C. It is apparent that the trend to low ductility creep fracture is recorded in the notch bar tests before the smooth bar tests [13].

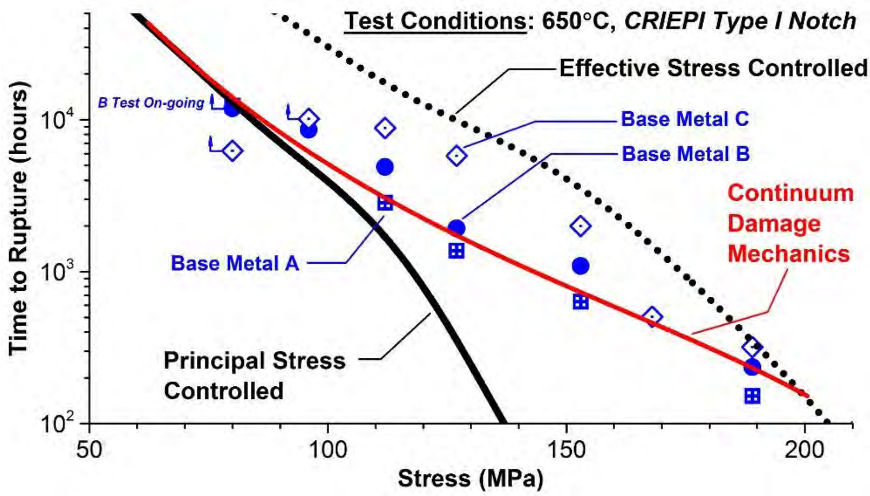

Figure 10. Analysis which supports the observations that for tests at 650°C using the same geometry of notched specimens, short term failure is caused by deformation and long term fracture is controlled by cavity nucleation and growth [14].

Detailed consideration of Figure 9 shows that for the same Grade 92 steels in short term tests both plain bar and notched bar samples fail with high ductility. In both cases the short term damage is a consequence of excessive deformation. Posttest metallographic evaluation of specimens in this region showed very limited creep cavity development. For increased creep test durations the ductility drops. Significant reductions in creep ductility of smooth bar tests at 600 and 650°C are observed for durations above about 10,000 hours, Figures 6 and 9. In contrast, the data at 600°C show trends to lower ductility even after only test durations of about 1,000 hours, Figure 9. Thus, although notch conditions can promote creep brittle behaviour, use of such geometries does not guarantee that test behaviour is relevant to long term component performance. Careful planning and execution of creep test programs must always carried out to ensure that meaningful results are obtained.

The observed trend in the behaviour is directly compatible with the fact that the multiaxial stress controlling behaviour changes from Equivalent Stress to Triaxial or maximum Principal Stress, Figure 10. As shown, this transition in behaviour can be described using appropriate continuum damage approaches. It is important to note that if the Equivalent Stress is used to estimate behaviour for all conditions, a significant over prediction of creep life will occur. The use of non – conservative approaches for the prediction of long term behaviour clearly increases the risk of in-service problems up to and including component fracture. Further insight into stress state issues can be obtained by properly document creep damage developed during testing. EPRI strategies for all characterization of this type is to use the validated approaches outlined earlier [5]. An example of the information obtained from posttest assessment of relatively long term notch bar tests is illustrated in Figure 11. The highest number density of creep voids recorded in the cross section at the root of the notch is identified slightly away from the surface, Figure 11. A greater number of voids is recorded across the section at the root of the notch then was detected away from the notch region. Clearly since the samples are made from the same steel the variations in creep void density are a direct consequence of variations in stress state. The posttest characterization offers an excellent approach to assessing both the general validity of the experimental test program and as key information to compare with predictions from continuum damage mechanics.

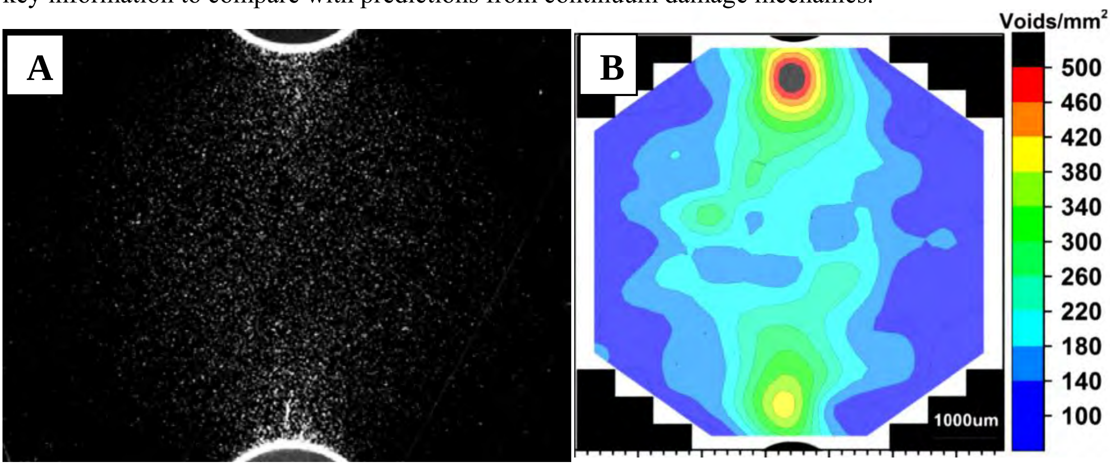

Figure 11. The distribution of creep voids at the sample center line in the notch region of a Grade 92 laboratory test (A) with a number distribution map obtained using an image analysis package (B). The highest number of voids has developed slightly ahead of the root of the notch.

4. DISCUSSION

It is apparent then that metallurgical factors are linked to both deformation resistance and fracture resistance. Several recent papers have considered background and detail of how composition, heat treatment and fabrication details influence creep behaviour [15, 16]. In summary, the key factors are as follows:

Deformation Resistance. It is necessary to Control **All** Heat treatment to provide the desired:

- Dislocation Density and Arrangement
- Type , size and distribution of MX ; provides resistance to dislocation movement etc
- Size and Distribution of M_{23} C_6; provides stabilization of the dislocation substructure.

It is apparent that deformation behavior is primarily controlled by ensuring that correct heat treatment and composition of (C + N) and carbide formers Mo, V, Nb etc are exercised. Long term changes linked to Ni. The overall aim of fabrication process to ensure at least Code minimum deformation resistance is achieved is thus to achieve a homogeneous, uniform tempered martensitic microstructure which is stable during service at high temperatures.

Resistance to Creep Fracture (Cavitation): Control **ALL** Fabrication and Heat treatment to:

- Ensure that undesirable inclusions are not introduced during steel making,
- Prevent the formation of deleterious phases as a result of metallurgical stability of alloy (e.g. delta ferrite, intermetallics etc), and
- Control the presence and segregation of tramp elements.

Creep fracture resistance is primarily linked to size, shape, distribution of inclusions e.g. Al_2O_3, MnS etc); hard intermetallics e.g. AlN and Cu, Sn, As, Sb etc. The overall aim of fabrication processes to ensure that components do not exhibit unacceptable variations in fracture resistance and thus damage tolerance is to minimize the presence of metallurgical factors which promote the formation and/ or accelerate the growth of creep cavities. These factors once present in the steel are not simply mitigated by heat treatment and can affect the creep damage susceptibility. Experience has shown that the metallurgical factors which are deleterious to creep ductility of the base metal will be an influence in weld HAZs.

It appears that when welds are made in CSEF steels which show an increased susceptibility to form creep voids, extensive creep damage will be found in the HAZ. Posttest characterization has shown that indeed for many situations creep will lead to significant nucleation and growth of creep voids. Details of the methods used to make these measurements have been published previously [5]. The example shown in Figure 12 illustrates the posttest damage present in a Grade 91 steel cross weld feature test near to creep fracture. Image analysis has been applied to determine the distribution of creep voids detected across the weld HAZ. The results obtained are should as a number – distance histogram. It is apparent for this specific test the highest density of density of creep voids was measured about 2 mm from the fusion line.

It should be emphasized that research has shown that the tendency for creep cavity development and the type and number of voids developed are affected by the inherent metallurgy of the steel, the difference in creep resistance between the weld metal and the parent and the details of the weldment geometry. The metallurgical risk factor influences the

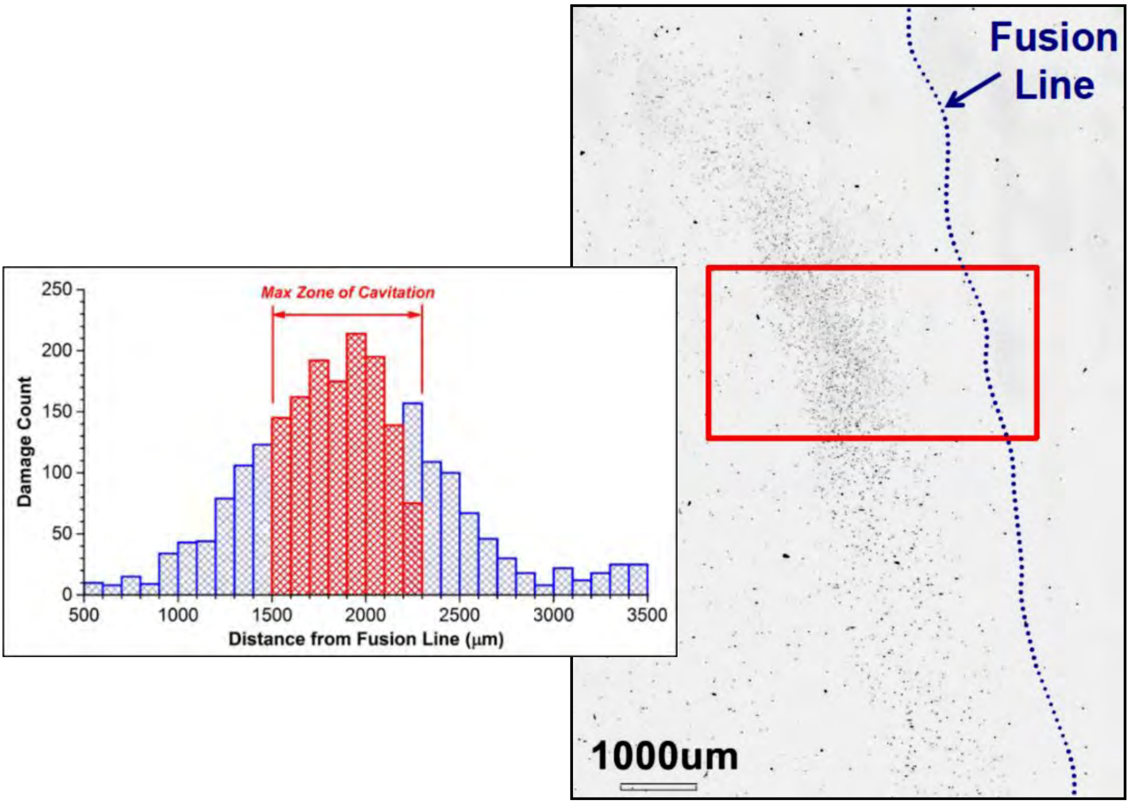

Figure 12. Typical creep cavitation damage revealed by posttest metallographic characterization of a Grade 91 steel feature test sample. The inset shows a histogram of the number of voids detected with distance from the fusion boundary. Details of the preparation and characterization process have been provided previously [5].

susceptability for voids to form. Differences in creep resistance and geometry then appear to influence the local stress state developed during creep within the HAZ. As a general rule the location of greatest damage is observed in the fine grained HAZ. This region is linked to weld thermal cycles close to 950°C. This type of thermal cycle detroys the normal martensitic structure and leads to a local band of very low strength to be present. Since this transformation will be present in all CSEF steel welds, the widely rteported variability in HAZ creep behaviour is predominantly a consequence of different metallurgical damage resistance and stress state (geometry) effects.

The overall behaviour is illustrated with reference to the schematic diagram shown in Figure 13. This diagram shows that the ‘inherent’ performance can be described by resistance to creep deformation and cavitation. Figure 13 shows these different levels represented by a 3 x 3 matrix with each axis having levels of low, medium and high. There is thus the potential that steel made to the same current specifications can be show variations in behavour from low / low (the top left hand box in the matrix) to high / high (the bottom right hand box in the matrix). It is apparent that even for steel which is ‘properly’ heat treated the creep life can vary by over an order of magnetude for uniaxial smooth bar tests at the same stress and temperature. Reducing this variability has been a mojor motivation behind EPRI recommendation for improved Guidelines for the fabrication of Grade 91 steel [17].

It is important to note that available information shows that cross weld creep life for Grade 91 steel can vary by an even greater amount, Figure 13, than noted for base metal. Thus, for feature tests performed under the same creep conditions the lives observed varied by a factor of 30. Improvements to reduce the metallurgical risk factor together with a 'well engineered' approach to weld design and manufacture [18] are key aspects of EPRI's strategy for improving the in – service performance and asset management of CSEF steels.

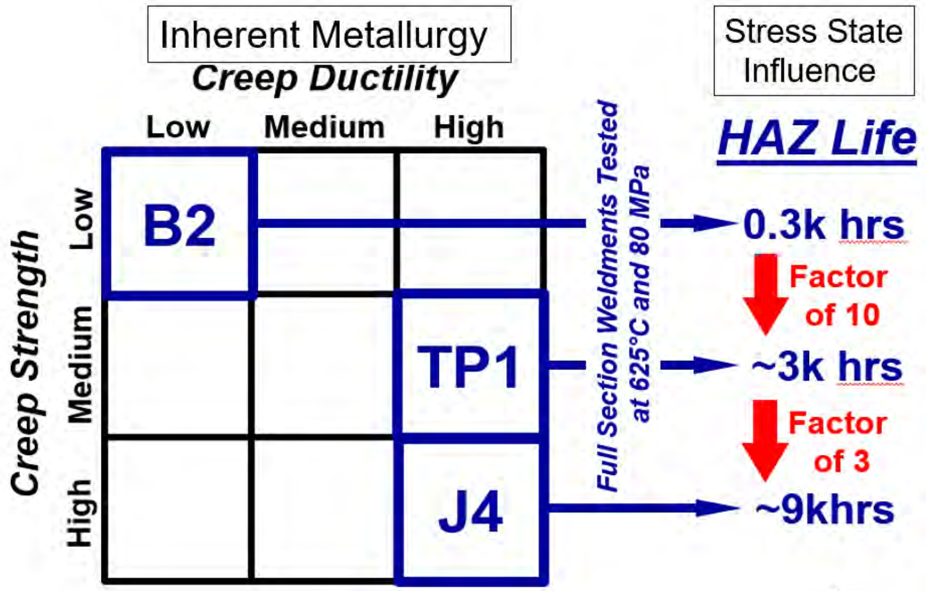

Figure 13. Schematic Illustration of how the variation of inherent metallurgical factors effect resistance to creep deformation and ductility. For a given tempered martensitic base metal (same metallurgical factors) the multiaxial creep behaviour is also influenced by stress state

Hardness testing has been widely used as a means of assessing the material quality of components made from tempered martensitic steels. The primary benefits of hardness testing are that these methods are relatively inexpensive and should produce data quickly. It is well established from previous EPRI research that, even when added as a guide to assessing as-fabricated quality, it is critical that staff making hardness measurements are properly trained and use validated equipment, methods, and procedures [19]. Despite the well-known limitations of hardness testing, several attempts have been made to develop relationships between hardness data and component remaining life, creep damage, fracture resistance, and other engineering parameters, for a range of steels. The ease and low cost of making hardness measurements has resulted in the substitution of hardness testing for more reliable nondestructive evaluation or assessment methods. Simplifications of this type can cause significant problems, and it is clear that hardness does not properly measure creep properties or fracture toughness in complex materials such as the creep-strength-enhanced ferritic Grade 91 steel.

5. CONCLUSIONS

Tempered martensitic steels exhibit a trend to relatively low ductility as creep rupture times increase. The tendency for brittle behaviour is in all cases due to the formation of creep voids.
Details of the number of voids formed, and the tendency for reductions in strain to fracture, are different for different steels. However, it appears that in all cases, void nucleation is related to:

– The presence of trace elements and hard non-metallic inclusions which are related to steel making, composition, normalizing temperature & cooling rate.
– The stress state which is a function of geometry, loading and local creep behaviour

It should thus be emphasized that trends in creep performance under Multiaxial Loading conditions are different to behaviour in simple smooth bar homogeneous specimens. Creep continuum damage mechanics simulations can be used to evaluate geometries effects and real component behaviour that cannot be tested in a laboratory. A Practical Life Management strategy for CSEF steels thus cannot be achieved by a singular solution. The EPRI recommended strategy integrates control of key factors affecting performance such a better steel specifications, design, fabrication and control of operation.

6. ACKNOWLEDGEMENTS

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