

DEVELOPMENT OF THE R5 VOLUME 2/3 PROCEDURE TO ENABLE THE CREEP-FATIGUE CRACK INITIATION ASSESSMENT OF CARBURISED STAINLESS STEEL COMPONENTS

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ABSTRACT

In a high temperature (>480°C) carbon dioxide rich gas environment, 300 series stainless steels typically develop a duplex oxide of magnetite and a nickel-chromium rich spinel. Whilst the oxidation behaviour is well understood, it has only recently been considered that this duplex oxide growth also results in carbon injection into the metal, resulting in a surface layer of carburised material up to approximately 500µm deep.

This carburisation results in the lamellar formation of carbides along grain boundaries. These brittle phases covering the grain boundaries are expected to result in a reduction in the affected material's creep ductility and fatigue endurance. At the same time, due to changes in local chemistry and microstructure, other material properties are affected in the carburised layer, such as creep deformation behaviour, tensile properties and cyclic stress-strain properties.

Existing R5 methodology used to predict creep-fatigue crack initiation does not account for the presence of carburisation. However as the R5 Volume 2/3 assessment methodology aims to predict initiation of cracks of sizes similar to the depth of the carburised layer, there is likely to be a significant impact on the time to initiate creep-fatigue cracking. This paper briefly discusses the nature of carburisation and then focuses on the materials data and methodology changes required to obtain a more accurate and conservative estimate of creep-fatigue initiation times for carburised components.

INTRODUCTION

Advanced Gas-cooled Reactor (AGR) power plants, operated in the UK, use a gaseous primary coolant typically made from made up of CO (~1%), CH₄ (~230vpm), H₂O (~400vpm), H₂ (~260vpm) and the balance being CO₂. The superheater and reheater sections of the boilers are made from Type 316 austenitic stainless steel operating at typical temperature between 460°C and 620°C. In this gaseous environment it is expected that at temperatures of 480°C and above Type 316 will develop a duplex oxide of magnetite and a nickel-chromium rich spinel, Chen *et al* (2015). Metal loss due to oxidation is generally considered to be benign on its own and the degradation mechanism of concern in the austenitic boiler sections tends to be creep-fatigue crack initiation from a defect free structure in areas of stress concentration, followed by creep-fatigue crack growth.

It was historically understood that the oxidation mechanism of austenitic stainless steels resulted in carbon ingress into the material, as shown in Figure 1. However it was perceived that this was limited by the formation of a healing layer at the base of duplex oxide and the structural integrity consequences of carbon injection were not considered further. However, examination of ex-service components has shown that in certain cases when a duplex oxide develops, a protective healing layer does not always form subsequently. The result of this is continuous duplex oxide growth throughout life, resulting in continuous

carbon ingress which creates a substantial carburised region after long exposure times up to a typical depth of 0.5mm, however this has been seen to extend up to 1mm in some austenitic steels.

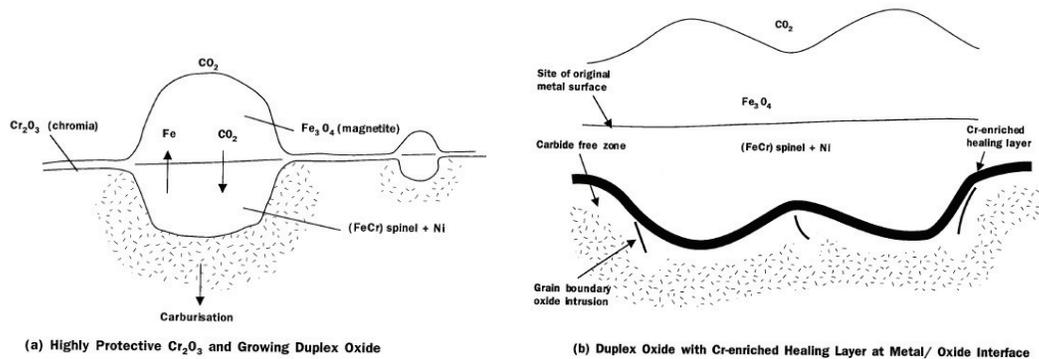


Figure 1: Oxidation behaviour of austenitic stainless steels in an AGR gaseous environment

The structural integrity of austenitic boiler components is typically demonstrated through the application of the R5 assessment procedure, Dean (2014), to determine a components predicted lifetime. When considering creep-fatigue crack initiation, the R5 procedure considers the creep and fatigue damage accumulation at the initiation location, typically at the surface. Currently the R5 assessment procedure tells the user environmental effects must be considered but provides no methodological advice on how to do this. Carburisation has a marked effect on a number of material properties relevant to creep-fatigue degradation, most notably creep ductility and fatigue endurance. Therefore a programme of work has been established to characterise the mechanism of carburisation, quantify the changes in material properties and suggest how the existing assessment methodology can be updated to allow conservative creep-fatigue structural integrity assessments to be carried out on carburised components.

Such effects have been considered previously in the UK by the Central Electricity Generation Board (CEGB) in the 1970's and 1980's when considering the potential of carburisation in fast reactor design, where contamination of liquid salt with carbon (such as oil) could result in rapid carburisation of 9Cr1Mo steels and austenitic stainless steels. The effects on creep rupture were considered benign if not positive as observed in 9Cr1Mo steel by Soo and Rowlands (1980). However the results on fatigue were deleterious as shown by Priddle and Marshall (1986). No work on creep-fatigue loading has been identified. A review of possible effects of carburisation on creep was reported by Gutmann (1990) which summarises some of these observations.

Whilst the experimental work presented herein is specific to austenitic stainless steels in an AGR gaseous environment, the proposed changes to the R5 assessment methodology is generic and could be applied to other environmental-material interactions if suitable materials data is available. This may make such methodology a wider concern for those considering Gen IV plant, where higher temperatures are more likely to result in environmental interactions.

CHARACTERISATION OF CARBURISATION

Carburisation was first identified in ex-service material due to elevation in hardness measurements near gas side surfaces. Further examination identified that this elevation in hardness was due to carburisation. Metallurgical studies of specimens at different temperatures and exposure times have allowed an understanding to be established. As illustrated in Figure 1, carburisation occurs as the material oxidises. The exact mechanism by which carbon is injected into the metal is not fully understood, however it has

been observed that gas pressure does influence the extent of carburisation without impacting the extent of oxidation.

There is a clear temperature dependency on the extent of carburisation observed. Below approximately 480°C temperatures are too low for the chromia layer to be broken down and therefore material does not oxidise further and no carburisation is observed. Above this temperature a duplex oxide often forms. As the exposure temperature increases up to 550°C, so too does the rate of oxidation. Therefore, the amount of carburisation increases. Above 550°C the initial rate of oxidation continues to increase, however as the temperatures increase so too does the rate of material healing which impedes further oxidation and therefore carburisation. Above 600°C duplex oxide growth is initially rapid but a fully protective layer is formed within a couple of years of exposure which effectively stops further oxidation or carburisation. At long exposure times (>5 years) an exposure temperature of ~550°C results in the thickest oxide and most severe carburisation.

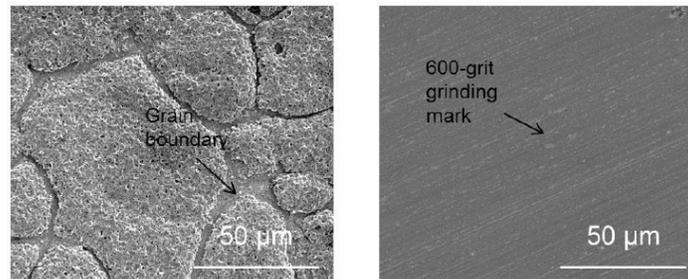


Figure 2: OPS polished (left) and 600-grit SiC ground (right) Type 316 specimens exposed for 500h at 550°C, taken from Chen *et al* (2015)

Other variables also have a significant impact on carburisation. The most significant is the surface finish of the material being exposed. If there is significant cold work at the surface a chromia oxide is likely to persist even at high temperatures, as seen in Figure 2 for the 600-grit surface finish. This is understood to occur because the high density of dislocations allows rapid diffusion of chromium to the surface to maintain the protective chromia layer. Conversely if all surface hardening due to cold work is removed, by polishing, using an oxide polishing suspension (OPS) for example, the material breaks into duplex oxide growth almost instantly when exposed. Figure 2 also shows that the diffusion of chromium is more rapid down grain boundaries as the grain boundaries are the last area to form duplex oxide as the chromia is maintained by the additional diffusion of chromium. Another significant factor is the material grain size which also relates to the diffusion of chromium to the surface to impede oxidation. As grain size is proportional to the number of grain boundaries to unit area, the larger the grain size the less diffusion paths there are for chromium to diffuse and impede oxide growth. Therefore large grained structures form faster and thicker oxide and therefore more extensive carburisation. This is particularly relevant to heat affected zones (HAZ) in weldments, where the heat input results in enlarged grains. This often results in local increases in oxidation as shown in Figure 3 (left).

As with diffusion of chromium to the surface, the diffusion of carbon into the material has different rates within the grain and down grain boundaries. As a result there are effectively two carburisation fronts, one in the grains and one related to grain boundary diffusion. By etching carburised samples this behaviour is evident, as seen in Figure 3 (right). The measurement of carbon content has also substantiated a different carbon profile if measured across grains as compared to the carbon content along grain boundaries, showing a greater depth of penetration along grain boundaries. The result is a grain boundary with lamellar formation of carbides and oxides along grain boundaries. It is expected it is this brittle layer in between grains which is the cause of the observed material embrittlement.

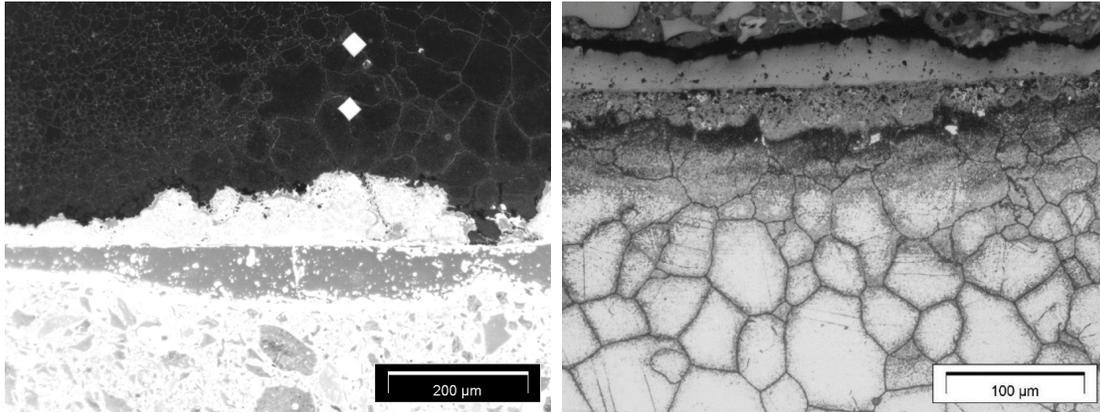


Figure 3: (Left) An etched image of a Type 321 HAZ showing a clear difference in grain size in the HAZ (i.e. larger grains on the right hand side). (Right) An etched Type 316 specimen showing duplex oxide, carburisation of the topmost grains and grain boundary carburisation below.

Whilst quantification of carbon and carbide density has been carried out to characterise the extent of carburisation, the crudest but most reliable method to measure the extent of carburisation is micro-hardness measurements. Whilst individual hardness indents are prone to scatter depending on how they impinge on grain boundaries, taking the trend from a series of indents can provide a reliable measure of carburisation behaviour. Figure 4 shows the extent of carburisation in a 4mm thick tube. Note the severity of carburisation at the bore and outer diameter (OD) differs slightly. This is most likely due to a different grain structure and surface finish at the two surfaces of the extruded tube which is commonly observed.

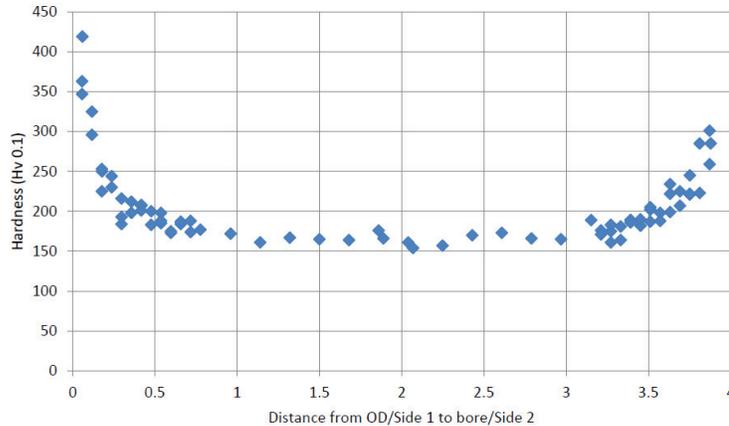


Figure 4: Hardness trace on 4mm Type 316 tube exposed to AGR gas (OD and bore) for 185kh at ~500°C

Hardness traces typically show an exponential decay from the peak hardness at the surface. The following form fits the hardness traces well.

$$C(x) = (C_{peak} - C_{bulk}) \exp\left(-\frac{x}{X}\right) + C_{bulk} \quad (1)$$

Where $C(x)$ is a measure of carburisation (whether hardness or other material property affected by carburisation) as a function of depth, C_{peak} is the value at the surface, C_{bulk} is the bulk value, x is the distance from the surface and X is a characteristic depth (depth at which the peak has reduced by 63%).

CHANGES IN CARBURISED MATERIAL PROPERTIES

A range of material properties is understood to be affected by carburisation. A challenge with characterising these changes by mechanical testing is any specimen has a gradation of material properties. For example if a load is put upon a specimen the stress distribution is unknown if elastic modulus or yield stress varies through the section.

A testing programme has been established, considering tensile, creep, fatigue and creep-fatigue testing of Type 316 stainless steel test specimens which have been carburised in an autoclave simulating AGR conditions. Exposure for 3kh at 600°C resulted in a carburised layer approximately 0.2-0.3mm thick. To accelerate the preconditioning the specimens were polished to an OPS finish to ensure rapid carburisation. The same material was also thermally aged to the same extent so any effects of thermal aging could be separated from effects of carburisation.

A summary of the material properties affected are presented below along with the current level of understanding.

Elastic Modulus: Preliminary results suggest that the elastic modulus in carburised material may be slightly elevated (order of 20% increase). These results have been obtained using nano-indentation.

Yield Stress: Assuming yield stress is proportional to hardness then a significant increase is expected to be observed in the carburised layer (up to 300% increase). There is concern that the presence of residual stresses in the carburised layer may be influencing the hardness measurements, therefore overestimating the extent of yield stress increase.

Tensile Ductility: Due to grain boundary embrittlement the tensile ductility is reduced. Tensile testing at 550°C with the specimen loaded up to 320MPa, corresponding to 5% strain, and then stopped, demonstrated cracking of the carburised layer. Being a standard specimen (7mm) with a thin carburised layer (0.2mm) it is unclear what the stresses may have been. Tensile ductility of uncarburised material is expected to be in the order of 40% at 550°C.

Cyclic Stress-Strain Properties: It is assumed cyclic properties will be changed in line with the yield properties. The presence of a 0.2mm carburised layer on a 7mm specimen resulted in a significant strengthening in cyclic stress-strain properties. Work on hollow fatigue specimens (to maximise the impact of the surface layer) is looking to quantify the changes in more detail.

Compressive Residual Stresses: It is known that carburisation results in a compressive residual stress in the affected material, however the significance of this is currently unknown. If the scale length of these residual stresses is small, then it may have no bearing on the structural integrity performance. If significant plasticity is seen in the material these residual stresses are likely to be removed.

Creep Deformation: It is known that carburisation results in a reduction in creep deformation rates. The exact impact is complex as historical work on 9Cr1Mo rarely separates out the impact of increased stress in the carburised layer and reduction in creep rate.

Creep Ductility: The major concern of carburisation is the negative impact it has on creep ductility due to grain boundary embrittlement. A reduction in creep ductility of carburised Type 316 material has been observed but has not yet been fully characterised.

Creep Rupture Strength: The effect of carburisation on creep rupture strength is effectively the balance of change in creep ductility and creep deformation. Work on 9Cr1Mo suggests the creep rupture strength of

carburised specimens (carburised outer layer and unaffected bulk) is improved. However this must be dependent on the geometry. In one extreme, if a specimen is completely carburised there would be no significant elevation in stress and historic evidence suggests the creep rupture would be greatly improved. In the other extreme, if a specimen is large compared to the thickness of the carburised layer the specimen will deform at a rate defined by the bulk material however the ductility at the surface would have been reduced. This can only lead to premature cracking at the surface and a detrimental affect to specimen creep rupture strength.

Fatigue Endurance: Historic evidence suggests that fatigue endurance of carburisation is greatly reduced, especially the nucleation phase of fatigue. Preliminary results in this programme have shown two types of behaviour can result. At low strain ranges carburisation results in the hardened carburised layer (possible with a compressive residual stress) suppressing fatigue nucleation, resulting in extended fatigue endurance. However at higher strains the opposite seems to occur, where the stress or strain can not be accommodated by the carburised layer and nucleation occurs prematurely, reducing the fatigue endurance. The effect of austenitic cyclic hardening is believed to be significant in this situation. Work in this area looks to substantiate the two behaviours and identify the boundary between them. It is also observed this nucleation phase within the carburised layer results in intergranular cracking. This is significantly different from what is observed in normal fatigue tests and will require a reassessment of how fatigue nucleation and short crack growth are considered.

CANDIDATE ASSESSMENT METHODOLOGY

Overview of R5 Volume 2/3 Assessment Methodology

The R5 Volume 2/3 assessment procedure, Dean (2014) considers creep-fatigue crack initiation for a defect free structure which is likely to be most affected by the presence of carburisation. R5 Volume 4/5 considers the growth of an existing defect which may be affected by carburisation but this is expected to be a lesser effect and not the focus of this paper. The methodology discussed herein relates to R5 Volume 2/3 and crack initiation from a defect free structure.

R5 Volume 2/3 has a series of steps to demonstrate the integrity of a component. A key benefit of the R5 assessment methodology is it allows the use of the elastically calculated stresses to carry out the assessment. R5 Volume 2/3 provide checks against plastic collapse, ratchetting and creep rupture but the main purpose is quantify the creep-fatigue damage at a specific location to determine when crack initiation is predicted to occur. When both creep and fatigue are significant, creep-fatigue damage is calculated by constructing a representative hysteresis loop for the assessment location, which provides the strain range and the stress-strain behaviour during the creep dwell to calculate the creep strain. Neuber construction is used to extrapolate from elastic stresses to elastic-plastic stresses. Fatigue damage is calculated using the calculated strain range using Miner's rule and creep damage is calculated using the accumulated creep strain in a creep ductility exhaustion damage model. The total damage is then the linear combination of these two damage calculations and crack initiation is conceded when the total damage reaches unity.

The Impact of Carburisation on a Load Controlled Creep Dwell

This example considers a single 1,000 hour load controlled creep dwell for a carburised 7mm and 2mm uniaxial specimen. The carburised layer is assumed to be the same as seen in the experimental preconditioned material, an exponential distribution of visual depth 0.2-0.3mm, corresponding to a characteristic depth, X , of 76.4 μ m. It is assumed that the carburised material has been cyclically hardened and therefore the stabilised cyclic stress-strain behaviour describes the stress-strain behaviour. The nominal tensile dwell of 300MPa is obtained from a stress range of 600MPa. This results in the boundary

condition of a load of 11.5kN on the 7mm specimen and 0.94kN on the 2mm specimen. The temperature considered is 550°C. The cyclic stress-strain behaviour is described by a Ramberg-Osgood curve:

$$\Delta \epsilon = \frac{\Delta \sigma}{E} + \left(\frac{\Delta \sigma}{A} \right)^{\frac{1}{\beta}} \quad (2)$$

Where $\Delta \epsilon$ is the strain range, $\Delta \sigma$ is the stress range, E is the elastic modulus, A and β and cyclic properties. E and β are considered to be unchanged by carburisation and accepted Type 316 data has been used. The A parameter is considered to be affected by carburisation. Using the form of equation 1, based on experimental data, A_{bulk} equals 1714 and A_{peak} equals 5485MPa, an increase of a factor of 3.2 at the surface.

Creep rate behaviour has been modelled using secondary power creep law:

$$\dot{\epsilon} = B \sigma^n \quad (3)$$

Where $\dot{\epsilon}$ is the creep rate, B is a creep constant and n is the creep exponent. For bulk properties B and n have been taken as accepted values for Type 316 stainless steel. The creep exponent, n , has been considered unaffected by carburisation. The creep constant, B , is considered to be affected. Thus far no reliable data has been obtained to estimate how creep is affected so it is assumed that $B_{peak} = 0.1 B_{bulk}$, with a distribution again defined by the form of equation 1.

As the cyclic stress-strain properties and creep properties across the carburised layer differ, the stresses, and creep strains differ. However by definition the total strain across the specimen must be equal. This has been modelled to show the stresses on initial loading, how they evolve when the specimen creeps, the total accumulated creep strain across the specimen and the total strain range and how they differ from what is expected in a non-carburised specimen.

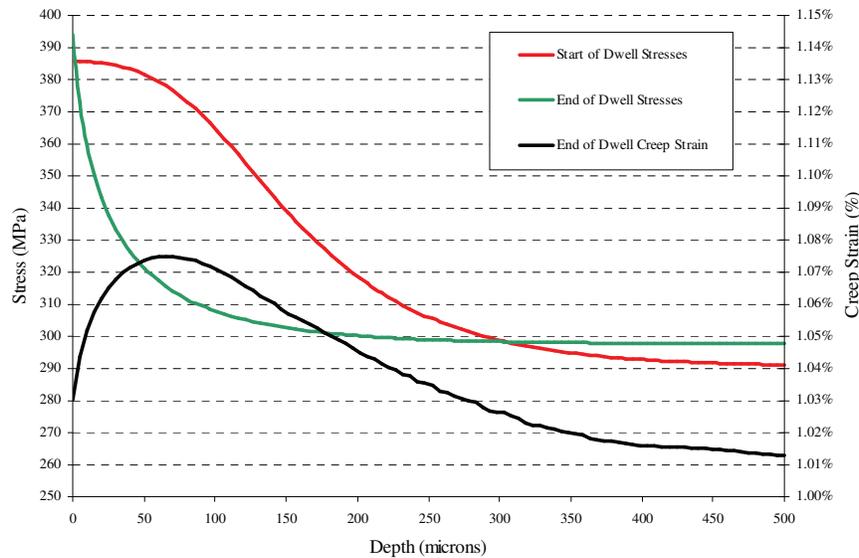


Figure 5: Stress distribution and accumulated creep strain in a 7mm carburised uniaxial specimen under load control with a nominal stress of 300MPa after a 1,000h dwell

Figure 5 shows the stresses on loading and after 1000h of creep for the 7mm specimen, by which time the stresses have reached a steady state, and creep strain accumulated in the uniaxial specimen. On loading the presence of the carburised layer reduces the elastic-plastic strain range from the estimated uniaxial value of 0.52% to 0.49%. Although a modest reduction, for a relatively thick component and thin

carburised layer this would still impact upon predicted fatigue damage. The stress distribution on loading deviates from an exponential decay distribution because in the very near surface region the increase in A has little impact as the stress-strain behaviour becomes elastic, limiting the stress peak. After creep, at the surface the stresses have increased slightly, as the mismatch in creep rate was greater than the mismatch in tensile properties. However in the majority of the carburised layer a significant stress drop is observed which is balanced by an increase in stress in the core. The maximum stress drop is observed at a depth of 65 μm where the stress drop was 62MPa.

Due to the stress redistribution in the carburised layer there is a greater amount of creep strain accumulated compared to the bulk material, by a maximum of 0.06%. Whilst alone it may seem insignificant, if this extra increment of creep strain occurred in multiple dwells then its contribution to creep damage could be significant. In this case as the total creep strain in the bulk was 1.01% the additional strain due to stress redistribution was insignificant. When compared to a homogenous structure with bulk properties, which would have been at a constant uniform stress of 300MPa, the total amount of creep strain expected in 1,000h would be 1.08%. So overall the presence of the carburised layer resulted in a reduction in the creep strain in the specimen.

The results for the 2mm specimen are presented in Figure 6. The model demonstrated the elastic-plastic strain range on loading would reduce to 0.44%, from a non-carburised range of 0.52%. Also the maximum creep strain in the specimen is reduced to 0.92% from a non-carburised value of 1.08%. So, unsurprisingly, as the carburised layer becomes a larger proportion of the specimen the impact upon the specimen behaviour becomes greater. However, even in a small specimen the extent of the change is likely to fall within the scatter of Type 316 material properties.

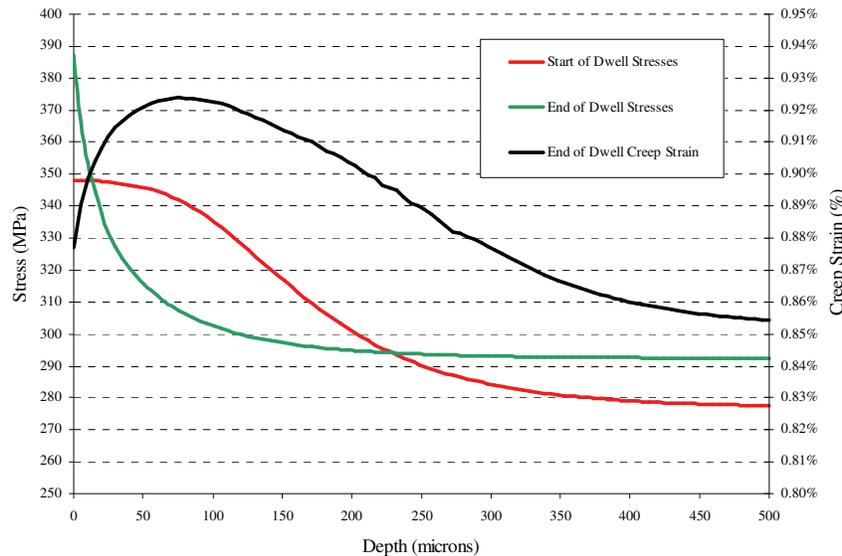


Figure 6: Stress distribution and accumulated creep strain in a 2mm carburised uniaxial specimen under load control with a nominal stress of 300MPa after a 1,000h dwell

This simple example shows how the initial stress distribution and strain is defined by the mismatch in tensile properties. Once the structure starts to creep the stresses redistribute to ensure the structure is creeping at the same rate. The additional creep strain accumulated in the carburised layer compared to the bulk is dependent on the mismatch between the tensile properties and creep properties with the possibility of additional creep strain due to stress redistribution. In these cases it was bound by the creep strain

expected in non-carburised specimen. Currently the impact of carburisation on creep deformation is uncertain so this may not be the case once further data is available.

If loading was displacement controlled the response of the carburised material has no effect on the underlying bulk and vice-versa, so specimen size has no impact. The amount of creep strain accumulated in the carburised layer is only dependent on the start of dwell stress and creep deformation properties of the carburised layer. Like the load controlled scenario the elevation in initial start of dwell stress is controlled by the tensile/cyclic mismatch, and the relaxation is defined by the creep deformation mismatch. Again, this could result in an additional creep strain in the carburised layer compared to a non-carburised specimen dependent on the balance of mismatch in tensile and creep properties.

Options for Treatment of Carburisation in R5 Volume 2/3 Assessments

Consideration has been made on how these effects can be incorporated in to the existing R5 Volume 2/3 assessment methodology. Two options have been proposed.

Option 1 is a simplified route where changes in stress and strains due to carburisation are ignored and a component would be assessed as would a non-carburised component. This is in effect treating the carburised layer as being thin compared to the bulk, so strain is controlled by the bulk. The impact of carburisation would solely be considered within the damage calculation, where a modified value for creep ductility and fatigue endurance would be used, derived from experimental testing. Such an approach is likely to be overly conservative for fatigue damage as carburisation would be likely to reduce the effective strain range, as shown in the example. For calculating creep strain during a dwell the proposed methodology may be conservative in estimating the total creep strain, as it was in the example model. However, due to a difference in cyclic stress-strain mismatch and creep mismatch it is possible additional (or reduced) creep strain may be accumulated in the carburised layer upon every cycle. This may effectively result in an extra increment (positive or negative) of creep strain occurring during every dwell, which could be significant over a components life. Further modelling is required to demonstrate whether this is significant compared to any reduction in creep strain due to the presence of carburisation. This is likely to be dependent on loading type and further modelling is required to establish this as a conservative option.

Option 2 is a more complex, but a more realistic approach to assessing carburised components. This would account for all the real changes in material properties, including tensile, cyclic and creep changes. This would mean the stress analysis would have to be modified and effectively a cyclic elastic-plastic-creep finite element analysis would be required for an assessment. Creep deformation properties as a function of stress would have to be suitably quantified (hard to determine when testing specimens including carburised and bulk material). Again the appropriate creep ductility and fatigue endurance would be required to calculate damage as in Option 1. Whilst this option is likely to be more realistic than option 1, uncertainties in material properties may be compounded resulting in a result which is overall less reliable than could be achieved from a simpler approach.

At this time it is expected that Option 1 is the most likely outcome for the treatment of carburisation in the R5 Volume 2/3 assessment procedure. Regardless of which method is selected the most significant impact on assessment result will be the change in creep ductility and fatigue endurance. In Option 1 the change in creep and fatigue damage will be proportional to the change in creep ductility and fatigue endurance respectively.

CONCLUSION

An understanding of the effects of carburisation on the creep-fatigue crack initiation of austenitic stainless steels has been discussed. Carburisation occurs due to the process of oxidation, resulting in carbon diffusion into the steel, preferentially along grain boundaries, resulting in an embrittled carburised layer. The material properties are modified by the carburisation which impact upon the materials susceptibility to creep-fatigue cracking. The most significant impact is expected to be a change in creep ductility and fatigue endurance of carburised material, however other changes will effect stresses, strain ranges and creep deformation behaviour.

By use of a simple model of a uniaxial specimen it has been shown how changes in material properties could affect the creep-fatigue behaviour, reducing fatigue strain ranges and changing the amount of creep strain accumulated. No advice is explicitly provided in the R5 assessment procedure for the treatment of carburised specimens. Two methodology options have been proposed, with the simpler option (Option 1) most likely to be adopted. This approach assumes it is conservative to calculate the stresses and strains as would be done for a non-carburised specimen and only consider the effects of carburisation in the damage calculation by modifying the creep ductility and fatigue endurance for values appropriate for carburised material. This specifies the creep ductility and fatigue endurance as the most important material properties required for the proposed modified assessment procedure.

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