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Original Citation

Yang, Xin, Lu, Zhongyu and Xu, Qiang (2015) The Interpretation of Experimental Observation Data for the Development of Mechanisms based Creep Damage Constitutive Equations for High Chromium Steel. In: IEEE International Conference on Automation and Computing (ICAC 2015), 11th - 12th September 2015, Glasgow, Scotland.

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The Interpretation of Experimental Observation Data for the Development of Mechanisms based Creep Damage Constitutive Equations for High Chromium Steel

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Abstract—It is very important to design a safe factor or estimating the remain lifetime for electric power plant components of steam pipes which mostly manufacture by high chromium steels and work at high temperature and low stress level. The author will develop the mechanisms based on creep damage constitutive equations for high chromium steel under lows stress in initial stage: (1) Creep cavities mostly formed attaching with the precipitation of Laves phase or on grain boundary for high chromium steel under low stress. The Laves phase should play an active role in the nucleation of creep cavities and suggest to explore the function between cavity nucleation and the evolution of Laves phase; (2) The dominant cavity nucleation mechanism is adapted to high chromium steels under low stress level; (3) Brittle intergranular model is appropriate for high chromium steels at high temperature under low stress level; (4) High density number of cavity of crept test high chromium steel at high temperature under low stress could be as fracture criterion.

Keywords—high chromium steel; cavitation; Constitutive equations; low stress

I. INTRODUCTION

With the increasing demand for power supplies and reducing CO₂ emissions, it is essential to improve the efficiency in energy conversion for chemical and petrochemical plants or power generation systems etc. by higher operating temperature and design stresses [1]. The modified 9Cr-1Mo steels strengthened with addition of Niobium (Nb) and Vanadium (V) or Mo with W have been extensively used in new advanced fossil-fired steam power plants operating at around 873/923K with higher efficiency [2].

The application of computational approach, particularly the development of creep damage constitutive equations is important for industry and academy. In order to design the safe components for power generation industry, particularly in fossil fuel plants and nuclear reactors, it is necessary to estimate their long-term creep behaviour such as creep deformation, rupture time by experimental or calculations. On the other hand, obtaining long term creep data is time consuming and cost expensive, thus the long term creep data is limited, and the extrapolation using the conventional empirical methods

may not be reliable [3]. With more accurately predicting the life, components could work for longer time and in a more operationally flexible manner before being retired or fracture [4]. Therefore, it is necessary to develop valid creep damage constitutive equations to accurately predict the long-term creep life time.

During the past several decades, there were many various developed creep damage constitutive equations models for describing creep damage process and predicting the lifetimes of the components operating at high temperature [5]-[10]. The classic Kachanov-Robotnov (KR) equations (1) (2) are one of the earliest to use the concept of continuum creep damage mechanics (CMD) in the development of constitutive equations by defining a single empirical mathematical parameter ‘ ω ’ [5]. The damage parameter as denoting creep damage of materials changes between 0 and 1. It’s an obvious advantage for computations of creep damage with a single empirical damage parameter formulation. However, it cannot explore a satisfactory definition with single one damage parameter, even if there is more than one physical creep damage mechanism during creep damage process.

$$\dot{\epsilon} = \dot{\epsilon}_0 \left[\frac{\sigma}{\sigma_0(1-\omega)} \right]^n \quad (1)$$

$$\dot{\omega} = \dot{\omega}_0 \left[\frac{\sigma}{\sigma_0(1-\omega)} \right]^m \quad (2)$$

Dyson physically based on continuum damage mechanisms have provided a framework to contain dominant mechanisms reflecting the evolution of materials’ creep state. Dyson’s equations (3) have coupled with the four various creep damage mechanisms of strain hardening, coarsening of the strengthening particles, solid solute depletion, and grain boundary creep cavitation [5]. Comparing with KR equations, the advantage of Dyson’s equations is different microstructural damage parameters based on phenomenological could be defined a dimension damage variable for each different creep damage mechanism. However, there are two difficult points for practically using Dyson’s constitutive equations with multitude creep damage mechanisms. One is any given alloy steels may be operated including all, some or none of creep damage mechanisms. Depending on the given alloy steels’ microstructure and operating conditions such

as the stress and temperature level, the development of creep damage constitutive equations based Dyson's equations should be coupled the effective and relevant creep damage. The other point is to identify the effective operating creep damage mechanisms or parameters based on the quantification analysis of creep damage, especially for cavitation.

$$\begin{aligned}\dot{\varepsilon} &= \frac{\dot{\varepsilon}_0}{(1-D_d)} \sinh \left[\frac{\sigma(1-H)}{\sigma_0(1-D_p)(1-D_N)} \right] \\ \dot{H} &= \frac{h'}{\sigma} \left(1 - \frac{H}{H^*} \right) \dot{\varepsilon} \\ \dot{D}_d &= C(1-D_d)^2 \dot{\varepsilon} \\ \dot{D}_p &= \frac{K_p}{3} (1-D_p)^4 \\ \dot{D}_n &= \frac{k_N}{\varepsilon_{fu}} \dot{\varepsilon}\end{aligned}\quad (3)$$

Yin et al [7] proposed the development of cavitation equation have been applied for high chromium steel at high temperature under middle and high stress, the modelling results of creep life time for P92 agreed with experiment data. However, there are no experiment results for high Cr steels agreements with the extension application of Yin's new constitutive equations for low stress [11]. Chen et al [9] developed new creep damage constitutive equations for high chromium steels, Yang have validated the developed constitutive equations working well under middle and high stress, however, the modelling results of creep curve under low stress level is still show high strain at failure [12]. The research of causes of breakdown of creep strength in 9Cr-1.8W-0.5Mo-VNb steel by Lee et al [13] could be one reasonable exploration for these developed constitutive equations could not apply from high stress range to low; firstly, confirming the typical breakdown of creep in ASTM grade92 steel, transition from ductile transgranular fracture to brittle intergranular fracture is the major cause of the breakdown; secondly, most of cavities are nucleated at coarse Laves phase particles on grain boundaries, Laves phase precipitates and grows during creep exposure, and coarsening of Laves phase particles over critical size triggers the cavity formation and the consequent brittle intergranular fracture; thirdly, the coarsening of Laves phase can be detected non-destructively by means of hardness testing of the steel exposed to elevated temperature without stress. Otherwise, creep damage mechanisms and materials parameters of constitutive equations should be changed under different working conditons.

Generally accepted terms of the main cause of creep failure or fracture is the damage process dominated by starting with cavity nucleation, then growth, finally coalescence of cavity leading to rupture[1] [14] [15] [16]. The cavitation is significant internal damage mechanism contributing to the ultimate failure of the material, it indicated in the study of the relative significance of various internal creep damage mechanisms on the overall creep damage and lifetime of P91 steel [17]. However,

there is still lacking of the relationship of the architecture evolutions of cavitation damage and no clear correlation of final damage with cavity. Recently, the advanced technique of synchrotron X-ray micro-tomography, at large synchrotron source for characterizing ex-situ creep damage in materials, has been used in investigating on the evolution of cavitation and provided the 3D spatial distribution and cavitation characteristics with increasing creep expose time [18]-[22]. And quantitative analysis also provided a fundamental understanding of the underlying cavity damage mechanism of creep failure, in order to develop more advanced and accurate damage constitutive equations for estimating the lifetime of high chromium steels. In this paper, author will interpret the experimental observation data for the development of mechanisms based creep damage constitutive equations for high chromium steel.

II. CURRENT CRITICAL EXPERIMENTAL DATA ON CAVITATION DAMAGE

Recent decade years, the evolution process or fracture surface of cavitation of high chromium steel during or after creep expose have been investigated by traditional optical microscope (OM), transmission electron microscopy (TEM), scanning electron microcopy (SEM), backscattering electron(BSE), and synchrotron X-ray micro-tomography. The section will be summarized and analyses critical experiment data on cavitation in order to understanding the underlying cavitation damage mechanisms for developing creep damage constitutive equations for high chromium steels.

A. Cavitation Nucleation Site for High Chromium steel at High Temperature

After studying of the materials of ASTM grade 92 steel crept at 550-650°C for up to 63151h, Lee et al (2006) suggested the precipitation and coarsening of Laves phase are responsible for the intergranular fracture by SEM images of cavity formation in specimens crept with showing cavities are attached with the coarsening Laves phase, more precisely, cavities were nucleated at the coarsening precipitation of Laves phase along grain boundaries [13].

A backscatter SEM micrograph of the ruptured specimen of a 12% Cr tempered martensite ferritic steel under the condition of long term creep (823K, 120MPa, 139971h) shows creep cavities appearing a prior austenite grain boundary perpendicular to the direction of applied stress (2009) [23].

Later on (2010), more than 100000h of creep expose under 80 MPa at 600°C for the microstructure of the Grade 91 steel had been studied. According to SEM images from the distance of 7 nm fracture surface, it shows cavities nucleated at boundaries next to the precipitation of Laves phase [24].

Parker (2013) pointed out the formation of Laves phase could influence creep resistant behaviour attributing to relatively hard Laves phase can provide preferred sites for nucleation of creep voids [25].

Recently, creep rupture tests of the 9% chromium steel T92 had been done at different high temperature (600°C, 650°C and 700°C) under between 46 and 200 MPa (2014). The BSE micrographs of creep specimen which creep at 700°C for 8232h in the necked section shows cavities nucleation at the interface of the large Laves phases, and $M_{23}C_6$ carbide and Laves phases provide potential sites for creep cavities nucleation [26].

Zhu et al (2014) performed a creep test of 9.5% Cr chromium steel without addition of C and N at 650°C under different stresses, in order to avoid the influence of other phase precipitations such as $M_{23}C_6$ and MX, and summarized and reported three points [27]: (1) The precipitation of Laves phase mainly on the grain boundary during creep in the alloy studies; (2) There is no influence for the rate of Laves phase precipitation or the rate of growth and coarsening of Laves phase precipitation; (3) Suggest that increasing cavity nucleation triggered by the coarsening of Laves phase is the reason for the more rapid drop in creep rupture strength at lower stresses testing.

Focuses on the characterization of cavities evolution in a P91 steel pipe, creep samples were applied an initial tensile stress of 60MPa for 7000 and 9000h at 650°C. The SEM micrograph of the gauge section of 9000h crept specimen shows that creep cavities appeared along the lath boundaries and in the vicinity of second phase particles such as Laves phase [22].

Based on above critical experimental observations, in general speaking, creep cavities mostly formed attaching with the precipitation of Laves phase or grain boundary for high chromium steel under low stress. Taking all above viewpoints into account, the Laves phase should play an active role in the nucleation of creep cavities. So it is important to explore quantitative functional relations between the evolution of Laves phase and cavitation damage mechanisms for the development of mechanisms based creep damage constitutive equations.

B. Dominant Cavity Damage Mechanism for High Chromium Steels

According to different applied creep stress level, a change cavitation damage mechanism will govern creep damage fracture for high chromium steels at a given temperature. This section will discuss the dominant cavity damage mechanism for high chromium steels under high and low stress levels.

1) Dominant cavity growth mechanism for high chromium steels under high stress level

In more earlier studying, the principle mechanism at high stress levels is the viscoplasticity-assisted ductile rupture mechanism. The damage mechanism begins with void nucleation at the preferential stress concentration areas inside the grains; then void growth it assisted by grain deformation and followed by coalescence of the cavities, the evolution of cavities damage is equivalent to the creep strained cavity constrained growth dominant [5] [16] [28].

Recently, with three dimensional techniques of X-ray micro-tomography, the spatial distribution and 3D characteristics of the creep void with increasing creep

expose time for high chromium steel have been provided at high temperature and under a stress range between 120MPa and 180MPa [18]. Based on the experiment data of cavitation volume fraction, the average diameter of voids and the number density of cavitation curves, void volume fraction and the number density both increase, void volume fraction increases more rapidly. It reveals which of void nucleation or growth and coalescence process dominate. Furthermore, this results mean that the void growth by coalescence is progressively strengthened over nucleation as the stress is reduced in the range of 120-180MPa [18].

2) Dominant cavity nucleation mechanism for high chromium steels under low stress level

General speaking, the creep deformation under low stress is of diffusional and the void nucleation is controlled by the maximum shear stress; which is in line with the general understanding reported by Miannay, according to Xu's conference paper [29]. With finer resolution of cavity size, Gupta et al [18] has observed and reported the number density and mean size of crept specimen under different stress. It reveals that though at failure, the number density under low stress is much higher than that under high stress, and it is in the order of 2.5; this strongly indicated the significant effect of time and the nucleation is visco-type rather than stress controlled; it is useful and important to obtain the nucleation rate with time under different stress level and collaborative research on this is sought.

Creep test of P91 steel cross weld specimens were performed at 650°C under stress of 66MPa and interrupted at 20%, 40%, 60%, and 80% of the creep damage which is defined as the ratio of interrupted time to the rupture time [30]. The void number density in cross weld creep damage specimens were observed from 20%, 40%, 60% and 80% creep damage process and slowly grow during 60% and quickly increase from 60% to 80%. The microstructures of the 32% creep damage specimen and of the rupture specimen at 6740 h were observed by the optical microscope, in order to understand void initiation and growth state. It shows more creep cavity nucleated than cavitation growth from 32% to rupture time. Although P91 steel weldment related stress redistribution is different from base material, it gives research reference under the lack of experiment data for high chromium steel under low stress level at high temperature.

3) Exploring the relationship function between cavity nucleation and Laves phase

There are many literatures about researching the evolution of Laves phase and its influence on the stability of microstructure and creep rupture strength in creep expose for high chromium steels.

Several years ago, experiments were taken for investigating on the growth kinetics of Laves phase in high chromium steels. The results of simulation of growth of the Laves phase model agreed with scanning TEM measurements [31] [32]. In the creep test of modified P911 heat resistant steel at 823K, it shows that Laves phase appeared after a creep strain of 1%, and then the man size of Laves increased from 190 to 265nm with

increasing strain to 18% [33]. Recently, the Laves phase process was characterized by SEM images, it found that creep rupture strength started decreasing more rapidly before the Laves phase reached equilibrium; there was no significant effect on the early stages of Laves phase formation by creep stress and strain[34], and the growth kinetics of Laves phase by creep deformation[27].

Secondary phase (M23C6, Laves phase) don't have influence on cavity growth during creep expose [21]. As discussed in section II part B, the cavity nucleation is dominant damage mechanism for high chromium steel under low stress. Otherwise, section II part A presents the cavity nucleation site is attached to Laves phase. So the void nucleation rate is very important factor to taken into account with the evolution of Laves phase. The author try to the relationship function between cavitation nucleated and the evolution of Laves phase for high chromium under low stress level at high temperature. However, the experiment data of cavity nucleation at the beginning of creep damage is lack in current, so more experiments should be study the cavity nucleation rate for high chromium steels under low stress in future.

III. FRACTURE CRITERIA

A. Transgranular or Intergranular model

The transgranular and intergranular phenomenon of fracture experiment rupture samples were obverted after creep tests by SEM. This section will summary and analyses the fracture model for high chromium steels under different stress and temperature based on experimental observation in past 15 years as shown in below Table1.

TABLE I. TRANS-GRANULAR OR INTER-GRANULAR PHENOMENON OF HIGH CHROMIUM STEEL AT HIGH TEMPERATURE UNDER DIFFERENT STRESS

Materials	Temperature	Applied Stress	Fracture Model	Reason of Fracture
9Cr-1Mo Steel (Goyal et al.,2014) [35]	873K (600°C)	150MPa	Ductile transgranular fracture	Resulting from coalescence of microvoids
Grade 91 Steel (Shrestha et al., 2013) [36]	700°C	200MPa	Ductile transgranular Fracture	Resulting from diffusive cavity growth and coalescence
9Cr-1Mo Ferritic Steel in quenched and tempered (Choudhary, 2013) [37]	873K (600°C)	100MPa	Ductile transgranular Fracture	Resulting from micro-void coalescence
9Cr-1Mo Ferritic Steel in quenched and tempered (Choudhary, 2013) [37]	873K (600°C)	60MPa	Ductile transgranular Fracture	Resulting from micro-void coalescence
9Cr-1Mo Steel (Vanaja et al.,2012) [38]	773K (500°C), 823K (550°C), 873K (600°C)	120MPa,140MPa,160MPa,180MPa	Ductile transgranular fracture	Resulting from coalescence of microvoids

Modified 9Cr-1Mo steel (Masse et al.,2012) [28]	625°C	120MPa	Ductile intergranular fracture	Cavities nucleate, Cavity growth,coalescence of small intergranular cavities
Modified 9Cr-1Mo Ferritic Steel (Choudhary et al.,2011) [39]	823K (550°C)	200MPa	Ductile transgranular fracture	Resulting from coalescence of microvoids
9Cr-1.8W-0.5Mo-VNb Steel (P92) (Lee et al., 2006)[13]	550°C	270MPa	Ductile transgranular fracture	Resulting from coalescence of microvoids
9Cr-1.8W-0.5Mo-VNb Steel (P92) (Lee et al., 2006) [13]	650°C	80MPa	Brittle intergranular fracture	Suggesting the precipitation and coarsening of Laves phase are responsible.
9Cr-1Mo Ferritic Steel in quenched and tempered (Choudhary et al., 1999) [40]	793K (520°C)	150MPa	Ductile transgranular fracture	Resulting from void coalescence
9Cr-1Mo Ferritic Steel in quenched and tempered (Choudhary et al.,1999) [40]	873K (600°C)	90MPa	Ductile transgranular fracture	Resulting from void coalescence

From the comparison of Table1, in general accepted view, ductile tansgranular model is appropriate for high chromium steels at high temperature under high stress level and resulting from the coalescence of micro-void, meanwhile, brittle intergranluar is suitable for at intermediate and low stress. But Choudhary's experiment observation for high chromium steels under 60MPa at 873K (600°C) shows ductile transgranular fracture on surface, the results is inconformity with above summarized brittle intergranluar model at intermediate and low stress, because the experiment materials of 9Cr-1Mo steel have been quenched and tempered resulting with more stronger strength and ductility. Confirming the typical breakdown of creep in ASTM grade92 steel, transition from ductile transgranular fracture to brittle intergranular fracture is as the major cause of the breakdown [13].

B. Fracture phenomenon

In early year, creep tests of smooth and notched hollow cylinders were performed under internal pressure and additional axial load, there was the number of creep cavities on the outer surface, the middle of the cross section and the inner surface of the hollow specimens after the end of the experience [41]. Later on, the creep test of an ASME Grade 91 steel has been done for 113431 h under a load of 80MPa, the SEM images were observed a high number density of cavities through the fracture surface of the crept specimen [24]. And high densities of creep cavities of fracture Grade 92 steel specimen surface

at high temperature under low stress was also observed by typical optical micrographs [25] [26].

C. Fracture criterion

Base on section III part B described fracture phenomenon of a density of cavities on fracture surface, the fracture criteria would be built basing on quantifying cavitation and determining the end of creep model end of materials lifetime.

Classic Kachanov-Robotnov Hayhust (KRH) creep damage constitutive equations developed for low stress Cr alloy. The variable ω represents intergranular cavitation damage and varies from zero and is related to the area fraction of cavitation damage. The maximum value of ω is approximately 1/3 at failure [42].

Petry and Lindet modified new creep damage constitutive equations based on Hayhust's (KRH) and predicted the creep life time for high chromium steels (P91, P92) [8]. Because of numerical convergence reasons, the computations were stopped when the macroscopic strain reached 10% value. At this point, the strain rate was so high that remain life could be estimated as less than 0.1% of the elapsed time. The time at a 10% strain was taken as the time-rupture t_R . It was assumed that the damage varied between 0 at initial state and threshold value D_c which is taken between 0.1 and 0.3. The modified model of Petry and Lindet applied the weakest link approach, so the fracture criterion should be $t_R = \text{Min}\{t(\varepsilon = \varepsilon_c = 10\%), t(D = D_c)\}$.

Above two methods provides suggestions for fracture criterion of developing novel constitutive equations for high chromium steels under low stress. The critical size of average cavity diameter could not be the fracture criterion; because of the experiment data shows the size and volume fraction of cavitation increase with increasing stress. Based on section III part B of fracture phenomenon, a high number density of cavities for fracture surface of high creep steels at high temperature under low stress could be regarded as fracture criterion through quantified analysis of fracture specimens section.

IV. CONCLUSION AND FURTHER WORK

1) *The following points will be accepted and applied for the development of mechanisms based creep damage constitutive equations for high chromium steel:*

a) Creep cavities mostly formed attaching with the precipitation of Laves phase or grain boundary for high chromium steel under low stress. The Laves phase should play an active role in the nucleation of creep cavities.

b) *The dominant cavity nucleation mechanism is appropriate for high chromium steels under low stress level. The Laves phase does not influence the creep void growth rate and suggest exploring the function between cavity nucleation and the evolution of Laves phase.*

c) Brittle intergranular model is appropriate for high chromium steels at high temperature under low stress level.

d) High density number of cavity of crept text high chromium steel at high temperature under low stress could be as fracture criterion.

2) More research needs to do as summarized as below:

a) Creep nucleation of high chromium steels at high temperature under low stress should be measured using OM, TEM, SEM or X-ray;

b) Quantify analysis experiment data of creep nucleation of high chromium steels at high temperature under low stress, build the relationship function between cavity nucleation and the evolution of Laves phase;

c) Quantify the fracture surface of crept specimens; make sure the value of high density number of cavity as fracture criterion.

d) Coupling the new function of creep cavitation mechanisms and creep deformation for novel creep damage constitutive equations

ACKNOWLEDGMENT

Thanks for the School of Computing and Engineering, University of Huddersfield for partial scholarship and this work is carried out as part of first author's PhD research, et al.

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