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Review Article

Metallurgical and Stress State Factors Which Affect the Creep and Fracture Behavior of 9% Cr Steels

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EPRI-supported research has identified critical material information regarding the factors affecting the performance of creep strength-enhanced ferritic steels, in general, and Grade 91 steel, in particular. EPRI recommendations emphasize that a five-point, integrated strategy should be used for the effective life management of components fabricated from tempered martensitic steels. This integration promotes a balanced use of resources which, when properly focused, reduces uncertainty regarding creep and fracture behavior. Tighter control of processes from steel making, steel processing, and heat treatment ensures that alloys with deficient properties never enter service. One cornerstone of this proactive approach is the definition of 'Metallurgical Risk' which links the presence of inclusions and trace elements to the susceptibility for creep damage. The improved confidence in the high temperature performance of CSEF steel components promotes reliability, increases efficiency, and minimizes the risk of component fracture.

1. Background and Introduction

EPRI-coordinated, industry-sponsored research projects in creep strength-enhanced ferritic (CSEF) steels began in 2007 with a major effort to improve the life management of Grade 91 steel components. Since this initial project included more than 40 project participants and over $4 million in industry funding, several key follow-on projects have been completed or continue today. This series of related projects, illustrated in the schematic diagram shown in Figure 1, provides a critical base of information linking steel making and fabrication to microstructure and performance under simple and complex loading conditions.

These projects exemplify the EPRI mission statement "to provide thought leadership, industry expertise, and collaborative value to help the electricity sector . . . and addressed through effective research and development programs for the benefit of society."

It is important to emphasize that the learnings and findings were realized with direct input and perspective from stakeholders representing the entire electricity supply chain. A general outcome from this research is that for metallurgically complex steels, testing programmes should establish high temperature performance on steel sections which are carefully chosen and well characterized, and the full pedigree of the steel cast selected is known. Full details of the research performed and the reports, position papers, and documents are available from the EPRI website. Selected achievements directly linked to materials factors are summarized below.

(1) Detailed research examining the microstructure of tempered martensitic ferritic steels has provided key information concerning the rate controlling damage mechanisms for different conditions. Degradation during creep has been linked to many different factors; these include the formation of new phases, coarsening/dissolution of carbides, recovery of the dislocation substructure, and the nucleation, growth, and linking of microvoids [1–4]. Much of the prior research has been focused on the creep strength. However, from a standpoint of component performance assessment, it is important to evaluate both *when* a material fails (i.e., for laboratory testing, "time to rupture"), and also *how* it fails (i.e., for laboratory

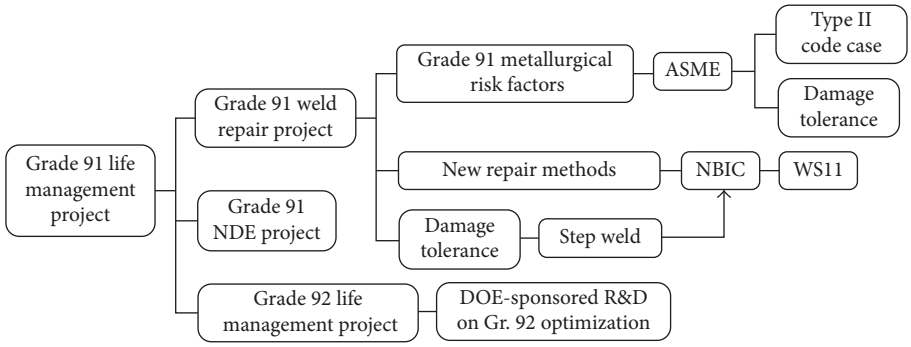

Figure 1: The evolution of major research and development projects specific to 9% Cr creep strength-enhanced ferritic (CSEF) steels within the Electric Power Research Institute since 2007.

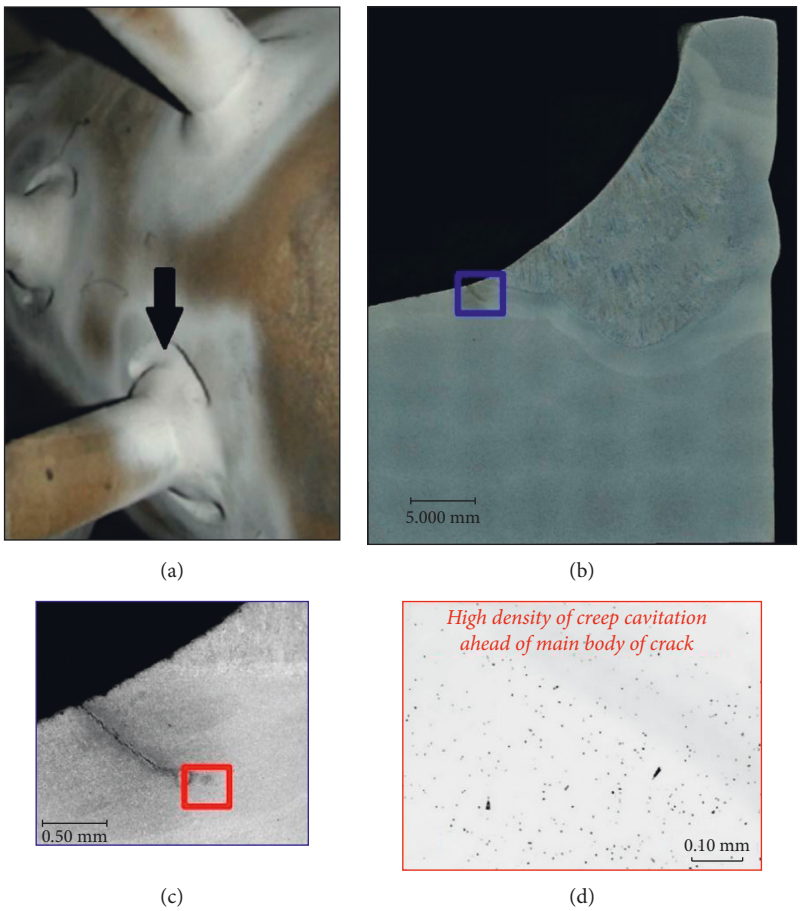

Figure 2: Example of in-service damage from Grade 91 heat "B2" [5]. The density of cavitation ahead of the crack varied from 700 to 1,000 voids/mm^2 and closely matches known maximum densities responsible for macrocracking in Grade 91 heat-affected zones (~1,000 voids/mm^2).

testing, "reduction of area (ROA)"). From assessment of in-service cracking, it is apparent that in the majority of cases, damage and failure are linked to the creep cavitation behavior. For example [5], the micrographs in Figure 2 show the very high number density of creep voids developed in the HAZ of a stub to header weld.

Root cause analysis established that the inherent cavity susceptibility of the Grade 91 base steel was a key factor linked directly to damage development in weld HAZs. Further details regarding the influence of specific trace elements on the nucleation and growth of cavities are presented later in this paper.

(2) The microstructure in the heat-affected zone (HAZ) of welds made from the 9% chromium martensitic steel is complex. Systematic microstructural investigations have defined the different regions of the

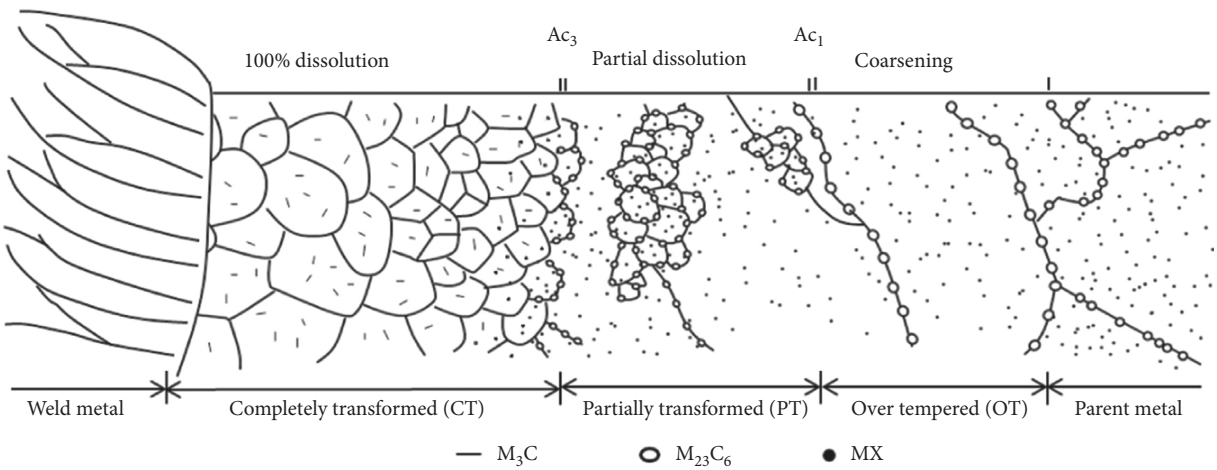

Figure 3: Schematic diagram illustrating the microstructural regions in the heat-affected zone of a fusion weld in a tempered martensitic steel [6, 7]. Meaningful characterization of microstructure in 9% Cr CSEF steels should take into account the phase dissolution and transformation behavior rather than simply describing the regions in the HAZ based on the size of the prior austenite grains.

microstructure across the HAZ as a function of the welding process [6, 7]. The microstructure in the HAZ of a single-pass, bead-on-plate weld on a Grade 92 parent was systematically investigated by using an extensive range of advanced electron and ion-microscopy-based techniques [6]. In addition, controlled thermal cycles were applied to simulate the microstructures in the different regions of the HAZ. It was found that the microstructure in the HAZ should be categorized in terms of the dissolution and transformation behavior as a function of a decreasing peak temperature during welding. The three primary microstructural regions, shown schematically in Figure 3, were defined as follows [6, 7]:

(a) The completely transformed (CT) region, in which the original matrix is completely reaustenitized with *complete* dissolution of the pre-existing secondary precipitate particles. The time at peak temperature is typically *sufficient* to dissolve inclusions present in the base steel.
(b) The partially transformed (PT) region, where the original matrix is partially reaustenitized along with a *partial* dissolution of the secondary precipitate particles from the original matrix. The time at peak temperature is typically *not sufficient* to dissolve inclusions present in the base steel.
(c) The over tempered (OT) region, where the pre-existing precipitate particles coarsen. No significant changes have been recorded to the size, number, and distribution of the inclusions present.

The PT region is the most susceptible area for creep damage base. This is consistent with evidence from the commonly reported HAZ failures in weldments constructed from these types of steels. This damage susceptibility arises because this location will always experience thermal cycles which degrade the local creep strength. However, the tendency for creep voids to form is directly linked to inclusions and trace elements. This may be present in following steel making and processing in steel where the level of control is below the Guidelines from EPRI. The peak temperatures and hold times achieved in the PT region are not sufficient to dissolve the inclusions which may be present in the base steel. Thus, damage susceptible HAZs will have low creep strength (from the weld thermal cycle) and poor resistance to creep void nucleation when large number densities of inclusions are present.

The link between creep ductility and each of these areas is critical in establishing the metallurgical risk factor(s) which control the susceptibility for low ductility failure in Grade 91 steels. It is clear that further evaluation of specific test results is of value to review potential trends in behavior. It is apparent that under conditions of low ROA, low ductility creep fracture is promoted as a result of the nucleation, growth, and link-up of creep voids.

The concept that creep cracking of in-service components is typically linked to the nucleation and growth of microvoids or creep "cavitation" is not new. This form of damage is well documented following root cause analysis of creep cracking during service in a large number of Grade 91 steel components. In addition, low creep ductility has been linked to cracking susceptibility of other established engineering steel systems. For example, in bainitic CrMoV steels [8–11], martensitic Grade 122 steel [12–14], and 316H stainless steel [15, 16], a link between low creep ductility and in-service cracking has been established. However, the specific materials factors which enhance cavitation vary and the key knowledge necessary to justify improved control of manufacturing process for CSEF steels has only recently been established.

Designers, manufacturers and operators of high energy equipment seek to use the most cost effective alloys which provide the required level of performance. Increasingly, this performance should be achieved without the use of extreme conservatism. Thus, it is important that the preferred alloys do not exhibit excessively large variations in properties. The

present paper summarizes key information from recent research which has shown that for metallurgically complex alloys such as Grade 91 and Grade 92 steel, changes in composition and/or heat treatment have a significant influence on high temperature behavior. Key materials evidence for improved control of steel making and processing to minimize the risk of components entering service with deficient properties is reviewed and summarized.

2. Effect of Chemical Composition on Microstructure and Properties

Historically, the American Society of Mechanical Engineers Boiler and Pressure Vessel Code (ASME B&PV) for Grade 91 type steel specifies control of 14 elements. A specific example for SA-335 P91 is given in Table 1 [17].

In 2011, the Electric Power Research Institute published a "Best Practices" guideline for the specification and procurement of Grade 91 components [18], this was updated in 2015 [20]. In addition to the stated ASME requirements, this document recommended the control of four additional elements (namely As, Cu, Sb, and Sn). It is important to mention that one of the original ORNL reports on Grade 91 steel which laid the foundation for the acceptance of Grade 91 steel, recommended the control of three other elements (O, W, and B) [19]. It is therefore important to note that the traditional specification of the chemical composition mandated by ASME B&PV Section II and other codes (such as EN or ASTM) lack sufficient control of elements known to influence the properties in CrMo steels [9]. Direct evidence regarding the need for improved control of Grade 91 composition [21–26] suggests that steel makers around the world are monitoring a far wider range of elements than that is mandated by minimum requirements in code; see the example for SA 335 P91 in Table 1.

The need for control of additional elements in Grade 91 steel is directly linked to the susceptibility for nucleation of creep voids and the associated reduction in the creep ductility [26]. The problems associated with trace elements and creep brittle behavior are of increasing concern because of the growing desire for recycling of steel, that is, the use of scrap steel in the melting process. Poor control and inadequate sorting of scrap can introduce elements such as tin and copper into the steel. Once present these elements are very hard or even impossibly to remove. Although steel scrap is typically classified into various grades and steel makers may monitor scrap to obtain the required quality, it is clear that the influence of even trace levels of undesirable elements can have a detrimental effect either on strength or ductility or both. Furthermore, the nucleation of creep cavities can be linked to the local microstructure and composition. Thus, even relatively small (as measured in weight percent) additions of sulfur (S), copper (Cu), tin (Sn), antimony (Sb), lead (Pb), and arsenic (As) have been shown to be particularly damaging under creep conditions. There is circumstantial evidence from other martensitic CSEF steels to suggest that the explicit control of boron (B) is of benefit to mitigate the risk of formation of boron-nitride (BN).

In addition to the effects of chemical composition on creep ductility, there are known effects of specific elements in reducing the creep strength namely nickel (Ni), the nitrogen to aluminum (N : Al) ratio, and excessive additions of boron (B). The influence of these elements on performance has been reviewed and is summarized in the following sections.

2.1. Influence of Tin (Sn). A series of systematic studies evaluating the influence of Sn on the creep strength and fracture behavior has been published [27–29]. The steel compositions used by Masuyama et al. [27] are detailed in Table 2. Results showed that Sn appeared to be particularly damaging as it can have a detrimental effect on strength and creep ductility (Figures 4 and 5). As shown in Figure 4, the observed creep strength is clearly reduced from mean Grade 91 behavior (typical results are shown using a Larson Miller parameter comparison ($C = 31$)). The lives of the Sn-doped heat shown in Figure 4 have been used to make specific performance estimates for selected temperatures (Table 3).

As noted in Table 3, a reduction in creep life was not calculated until the anticipated creep temperature was above 550°C (1022°F). In Figure 5, Sn-doped Grade 91 steel exhibited a pronounced drop in the measured reduction in area in creep tests performed at 650°C (1202°F). This trend occurred even for creep test lives of less than 1000 hours [27].

The research outcomes published by Masuyama et al. [28] are consistent with the results of other research [30]. The recent work has established detailed information on the metallurgical behavior of Sn using field emission gun and scanning transmission electron microscopy (FEG-STEM). Song et al. [30] reported concentrations of Sn at subgrain boundaries that were over 10 times the values measured in the matrix. Based on these observations, it was suggested that the effect of Sn would be to reduce the boundary cohesion (as in creep) creating preferential sites for the nucleation of creep cavities. These effects would nucleate a higher number density of cavities leading to reduction in creep ductility in Grade 91 material where Sn was present. As reported in previous work [27–29], the authors also found a marked reduction in creep strength [30].

These effects were summarized by consideration of the results of creep tests at 600°C (1112°F) and 100 MPa (14.5 ksi) [30]:

(1) controlled Grade 91 composition life = 100,641 hours

(2) Sn-doped Grade 91 composition = 35,290 hours

It has been suggested (Table 1) that the maximum permissible value of Sn in Grade 91 steel should not exceed 0.010 weight percent [19]. In the early 1990s, a major supplier of Grade 91 material suggested a maximum value of 0.020 weight percent [21, 22]. For manufacture of thick-section Grade 22 CrMo steel, a value of 0.010 weight percent maximum has been recommended. In practice, where the Grade 91 composition has been controlled, Sn values are routinely <0.005 weight percent [19, 22, 26, 31].

Thus, EPRI recommend that the maximum limit for *Sn in Grade 91 steel should be ≤ 0.010 weight percent.*

Table 1: Comparison of recommended chemical composition ranges for Grade 91 steel products.

Element	SA-335 P91 [17]	ORNL 6303 spec. [18]	ORNL 6303 target [18]	1023199 [19]	P91 book [20]	V&M [21]	Arcelor Gr. 91 plate [22]	Arcelor Gr. 22 plate [23]	EPRI proposal for Grade 91 Cl. 2
C	0.08–0.12	0.08–0.12	0.1	0.08–0.12	0.08–0.12	0.08–0.12	—	—	0.08–0.12
Mn	0.30–0.60	0.30–0.50	0.4	0.30–0.60	0.30–0.50	0.30–0.50	—	—	0.30–0.50
P	0.020 max	0.020 max	<0.010	0.020 max	0.020 max	0.020 max	0.010 max	0.008	0.020 max
S	0.010 max	0.010 max	<0.010	0.010 max	0.005 max	0.005 max	0.005 max	0.005	0.005 max
Si	0.20–0.50	0.20–0.50	0.2	0.20–0.50	0.20–0.40	0.20–0.40	—	—	0.20–0.40
Cr	8.00–9.50	8.0–9.0	8.5	8.00–9.50	8.00–9.50	8.00–9.50	—	—	8.00–9.50
Mo	0.85–1.05	0.85–1.05	0.95	0.85–1.05	0.85–1.05	0.85–1.05	—	—	0.85–1.05
V	0.18–0.25	0.18–0.25	0.21	0.18–0.25	0.18–0.25	0.18–0.25	—	—	0.18–0.25
Nb	0.06–0.10	0.06–0.10	0.08	0.06–0.10	0.06–0.10	0.06–0.10	—	—	0.06–0.10
N	0.030–0.070	0.030–0.070	0.05	0.035–0.070	0.040–0.070	0.030–0.070	—	—	0.035–0.070
Ni	0.40 max	0.20 max	<0.10	0.20 max	0.40 max	0.20 max	—	—	0.20 max
Al	0.02 max	0.04 max	<0.02	0.020 max	0.025 max	0.025 max	—	—	0.015 max
Ti	0.01 max	0.01 max	<0.01	0.01 max	0.010 max	0.010 max	—	—	0.01 max
Zr	0.01 max	<0.01	<0.01	0.01 max	—	—	—	—	0.01 max
Cu	—	0.20 max	<0.10	0.25 max	0.10 max	0.10 max	—	—	0.10 max
As	—	—	—	0.012 max[1]			—	0.01	0.010 max[1]
Sn	—	—	—	0.010 max[1]	0.020 max	0.020 max	—	0.01	0.010 max[1]
Sb	—	<0.001	<0.001	0.003 max[1]	0.010 max	0.010 max	—	0.0025	0.003 max[1]
B	—	<0.001	<0.001	—	—	—	—	—	<0.001
W	—	<0.010	<0.010	—	—	—	—	—	<0.010
Pb	—	—	—	—	—	—	—	—	<0.001 max[1]
N : Al ratio	—	—	—	4.0 min	—	—	—	—	4.0 min

[1]The combined value of Sn + As + Sb + Pb < 0.010 weight percent.

Table 2: Chemical composition of examined Grade 91 heats with controlled additions of impurity/tramp elements P, S, Cu, Sn, and Sb [27].

	C	Si	Mn	P	S	Cu	Ni	Cr	Mo	V	Nb	Sn	Sb	N
A	0.10	0.42	0.40	0.012	0.001	—	0.06	8.53	1.00	0.21	0.080	—	—	0.054
P1	0.09	0.30	0.38	0.006	0.007	0.01	0.09	8.63	0.94	0.24	0.060	0.003	0.004	0.031
P2	0.10	0.30	0.38	0.021	0.007	0.01	0.08	8.66	0.94	0.23	0.061	0.003	0.004	0.032
P3	0.09	0.31	0.38	0.030	0.008	0.01	0.09	8.64	0.95	0.24	0.061	0.003	0.004	0.033
S1	0.10	0.31	0.38	0.010	0.002	0.01	0.09	8.61	0.95	0.24	0.058	0.003	0.004	0.032
S2	0.10	0.30	0.37	0.010	0.014	0.01	0.09	8.64	0.95	0.24	0.061	0.003	0.004	0.033
S3	0.10	0.30	0.38	0.009	0.024	0.01	0.09	8.62	0.95	0.24	0.059	0.003	0.004	0.034
Cu1	0.10	0.34	0.39	0.008	0.008	0.05	0.09	8.62	0.89	0.24	0.062	0.004	0.005	0.031
Cu2	0.10	0.34	0.39	0.009	0.008	0.15	0.09	8.58	0.90	0.24	0.064	0.005	0.005	0.031
Cu3	0.10	0.34	0.39	0.008	0.008	0.27	0.09	8.51	0.90	0.24	0.063	0.004	0.005	0.034
Sn1	0.10	0.33	0.39	0.008	0.008	0.01	0.09	8.63	0.93	0.24	0.063	0.011	0.005	0.033
Sn2	0.10	0.33	0.39	0.009	0.008	0.01	0.09	8.63	0.93	0.24	0.063	0.056	0.005	0.033
Sn3	0.10	0.33	0.39	0.008	0.008	0.01	0.10	8.64	0.94	0.24	0.063	0.110	0.005	0.037
Sb1	0.10	0.34	0.38	0.009	0.007	0.01	0.09	8.65	0.92	0.24	0.056	0.004	0.011	0.032
Sb2	0.10	0.34	0.38	0.009	0.007	0.01	0.09	8.73	0.92	0.24	0.057	0.004	0.053	0.032
Sb3	0.10	0.34	0.38	0.009	0.008	0.01	0.09	8.66	0.91	0.23	0.056	0.004	0.110	0.034

Note: the "A" heat is the control heat and may be identified in associated figures as "P0" or "Cu0", and so on.

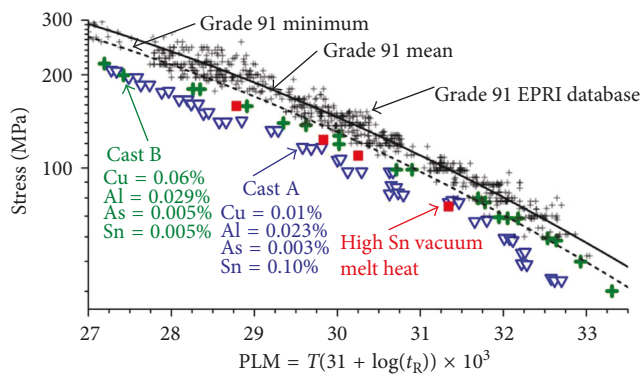

Figure 4: Analysis regarding the behavior of Tin (Sn), as plotted against the EPRI database of Grade 91 base metal using a Larson Miller parameter ($C = 31$), adapted from [27]. Note the reduction in strength for all casts with Sn. Even at the lowest value of 0.005 weight percent, the behavior is at the minimum (mean-20%) line. This reduction in strength may also be partially attributed to the presence of relatively high levels of Al in all examined heats.

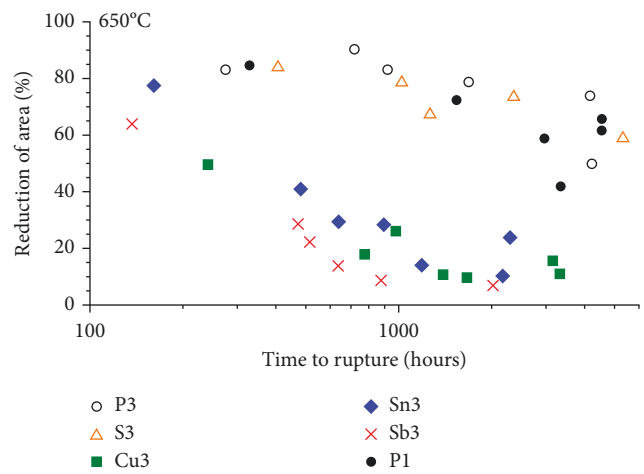

Figure 5: Analysis of Grade 91 behavior for experimental heats doped with P, S, Cu, Sn, and Sb and as compared to a control heat (P1) with low levels of tramp elements. Adapted from [27]. Note the marked decrease in reduction of area for heats doped in Cu, Sn, and Sb and the more gradual drop in behavior with time for heats doped with S and P.

2.2. Influence of Antimony (Sb). The effect of Sb, as analyzed by Masuyama et al. [27], has also been shown to influence the creep ductility. The steel compositions used in this research study [27] are detailed in Table 2. The ductility reduction was reported to initiate even at relatively low levels (Sb1 = 0.011 weight percent). The trend to lower ductility was accelerated by higher temperature and with increasing level of Sb (Figure 6).

As with the Sn-doped specimens, the reduction in the rupture life resulting from the higher amount of tramp elements at 650°C (1202°F) does not appear to be caused by the increase in the creep rate but rather by the decline in creep ductility. This is consistent with both the Sb- and Sn-doped samples showing enhanced creep microvoid nucleation leading to minimal reduction of area. As a consequence of the enhanced damage susceptibility, the rupture ductility reported with the Sb- and Sn-doped samples was lower than that of the P- and S-doped samples (Figure 5).

In the summary documentation prepared to support recommendations for Grade 91 steel, ORNL proposed limiting Sb to be less than 0.001 weight percent [19]. The more potent effect of Sb (e.g., as compared to Sn) is reflected in other guidance [25] where the Sb is recommended to be <0.010 weight percent. Furthermore, for Grade 22 CrMo steel, Sb should be <0.0025 weight percent. More recently, EPRI recommended controlling Sb to <0.003 weight percent in CSEF steels. In practice, the Sb level in commercial heats [22, 26] of Grade 91 steel has been highly controlled to values ≤0.0014 weight percent. This clearly reflects the more

Table 3: Analysis of the effect of Sn on performance as a function of service temperature for a given stress of 80 MPa (11.6 ksi).

Grade 91 material	Expected life (hours) at 80 MPa (11.6 ksi) and indicated temperature			
	550°C 1022°F	575°C 1067°F	600°C 1112°F	625°C 1157°F
Mean Grade 91	>1,000,000	>1,000,000	588,518	55,732
Min. Grade 91	>1,000,000	>1,000,000	204,913	19,983
Cast A (high Sn)	>1,000,000	360,250	32,340	3,320
Cast B (low Sn)	>1,000,000	>1,000,000	157,406	15,464

Data were taken from Figure 4 [27]. Note the dramatic drop off in anticipated life >550°C (1022°F).

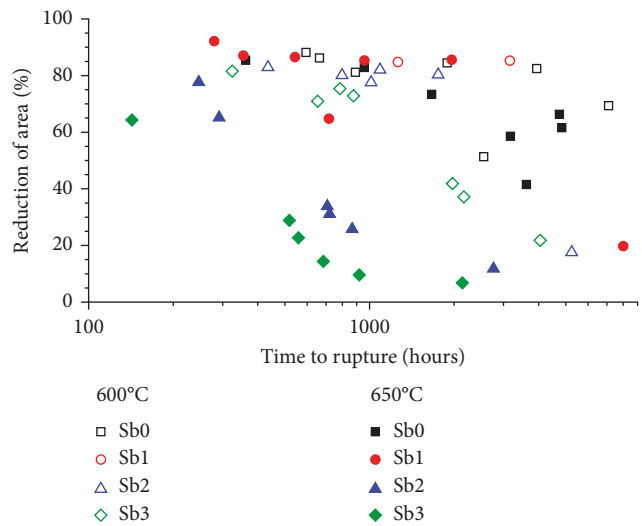

Figure 6: Analysis of Grade 91 behavior for experimental heats doped with Sb and tested at 600°C (1112°F) and 650°C (1202°F). Adapted from [27]. For even the heats with the low levels of Sb (Sb1 = 0.011 weight percent), there was a marked drop in the measured creep ductility, this drop was accelerated with increasing levels of Sb.

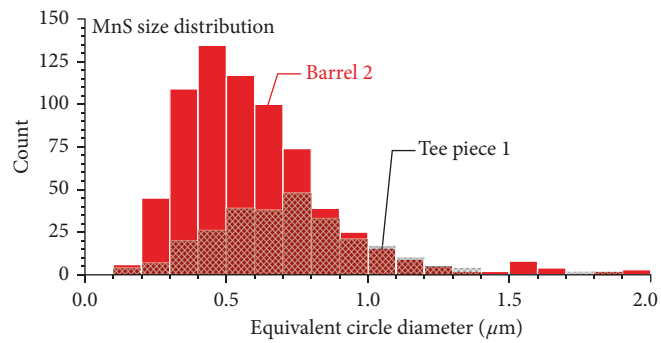

Figure 7: Number density of inclusions for two different but code acceptable Grade 91 steels. A higher density of MnS was measured in the creep damage susceptible steel which contained 0.009 wt.% S (labelled barrel 2) as compared to the cavity resistant steel which contained 0.002 wt.% S (labelled tee piece 1).

stringent pr+ocedures used by steel makers regarding the control of Sb. *Limiting Sb to < 0.003 weight percent* in Grade 91 steel, as originally suggested by ORNL [19], has been reinforced by the analysis presented here.

2.3. Influence of Copper (Cu). The influence of Cu on creep behavior has been examined [27]. It was reported that the influence of Cu on creep behavior was similar to that of Sn- and Sb-doped samples (Figure 5). Thus, Cu reduced the observed reduction of area even in plain bar uniaxial creep; the compositions used by Masuyama et al. [27] are detailed in Table 2. Interestingly, in a companion paper [29], the authors stated that, "the rupture life associated with the Cu-doped sample was seen to be independent of the content of Cu. This is because the Cu-doped samples exhibited poor ductility irrespective of Cu content."

With respect to the recommended composition limits for Cu in Grade 91 steel listed in Table 1, there were concerns reported by researchers at ORNL [19] regarding the effect of Cu on stabilizing the austenite phase at high temperature. Therefore, even at alloy introduction, it was recommended to keep the target chemistry at Cu < 0.10 weight percent [19]. This is the same value recommended in other early publications [21, 22]. Controlled composition Grade 91 type steels invariably contained less than 0.10 weight percent Cu [19, 23, 26, 31].

Thus, it is recommended that the *copper content of Grade 91 steel should be < 0.10 weight percent.*

2.4. Influence of Sulfur (S) and Phosphorous (P). In common with many engineering steels, these elements have been controlled from the first introduction of Grade 91 steel, [19]. The initial maximum levels of P and S appear to have been based on experience rather than detailed research study. The influence of S and P on the creep performance of Grade 91 steel has been examined in several subsequent research studies. It is noteworthy that in the work by Masuyama et al. [27], the levels of P and S used were below the specified maximum from typical design codes (Tables 1 and 3). A general trend of lower reduction in area was noted with increasing S for increasing time to rupture for creep tests conducted at 650°C (1202°F). Furthermore, and in the analysis of Grade 91 steel casts which exhibit excellent creep ductility and the stated maximum values quoted by some material suppliers, the values for S are specifically limited to values <0.005 weight percent.

EPRI-recommended methods for a component life management approaches identify that wherever possible root cause investigations should be performed to understand the factors influencing instances of service cracking. Indeed, several papers and reports document findings from investigations into cracking found during service in Grade 91 steel components. In particular, detailed laboratory

Table 4: Chemical composition (given in weight percent) for two exservice steels compared to the requirements in a typical standard specification [23].

Material	C	Cr	Mn	Mo	Ni	Si	Al	P	S	N	Nb	V	Ti	Zr	Cu	Pb	As	Sn	Sb	B	O
SA-387 P91	0.08 0.12	8.0 9.5	0.30 0.60	0.85 1.05	0.40 max	0.20 0.50	0.02 max	0.020 max	0.010 max	0.030 0.070	0.06 0.10	0.18 0.25	0.01 max	0.01 max	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Damage susceptible heat	0.10	8.77	0.41	0.94	0.12	0.41	0.040	0.009	0.010	0.0454	0.071	0.21	<0.002	<0.002	0.19	0.00075	0.0128	0.008	0.0023	<0.0003	0.003
Damage resistant heat	0.10	8.30	0.40	0.94	0.19	0.33	0.020	0.012	0.002	0.0424	0.070	0.21	<0.002	<0.002	0.05	0.00003	0.0042	0.003	0.00063	<0.0003	0.0024

characterization and metallurgical evaluation have been carried out on sections from a secondary superheater header that was removed from a coal fired power plant after about 79,000 hours operation [5]. This replacement was required due to large numbers of cracks in which formed in the heat-affected zones of stub tube to header welds. The damage formed a pattern with different header sections showing different susceptibilities to creep crack formation. Study of the exservice steel thus permitted evidence to be obtained regarding the specific metallurgy of steel heats which had developed cracks (identified as damage susceptible) and those which were uncracked (identified as damage resistant). Comprehensive compositional analysis of these heats showed that the damage susceptible heat (identified as Barrel 2 in Figure 7) contained S at the top of the then code allowed specification (Table 4).

Further study was performed to understand details of how the sulfur level in the steel could influence susceptibility to formation of creep cavities. It was found that there was a consistent trend to increased number density of inclusions for Grade 91 steels with increasing S content. This increase was particularly notable for S values ≥0.003 wt.%. Additional analysis, undertaken for two heats of material using a refined and higher resolution approach developed by EPRI, confirms the presence of a higher density of MnS in creep damage susceptible steel (0.009 wt.% S) as compared to cavity resistant steel (0.002 wt.% S) (Figure 7). This figure shows the distribution of the size of MnS particles, represented as an equivalent circular diameter, as being centered at ~500 to 800 nm. For conventional assessment methods using basic SEM-EDS approaches, the detection of very small MnS inclusions would be unreliable. Thus, it is apparent that the minimum particle diameter identified reliably using conventional SEM-EDS is typically ~1,000 nm (1 μm). Only when a specialist procedure, optimized for detection of smaller particles, is used can accurate number densities below this size be recorded.

Review of the compositions in Grade 91 steel: it appears that the *S content should be limited to ≤ 0.005 weight percent*, and if possible it would appear to be beneficial to restrict S *to ≤ 0.003 weight percent*.

2.5. Influence of Boron (B). Boron has been used as an alloying addition in a number of CSEF steels, including Grades 92, 122, and SAVE 12 AD [32–36]. In controlled compositions, and with the correct heat treatment, boron can be beneficial to the creep behavior of tempered martensitic CSEF steels. Evidence suggests that one benefit of adding B is a reduced coarsening rate of $M_{23}C_6$ carbides. However, when nitrogen is present in sufficient quantities, excessive levels of B introduce a risk of forming BN [32]. The formation of BN can be problematic for two reasons. First, it reduces the amount of available nitrogen in the steel matrix and so can limit the formation of precipitation strengthening MX carbides. Secondly when of sufficient size, the BN inclusions can act as initiation sites for creep cavities, lowering the creep life and reducing the creep ductility (Figure 8).

Detailed metallographic characterization has documented the development of BN in Grade 92 and Grade 122 steels. Although BN has not been reported in Grade 91 steel, its formation could take place because B is not specifically controlled in most standards. Abe [33] has researched the formation of BN in Grade 92 steel in detail. The diagram shown in Figure 9 has been developed based on this previous work [33]. For the range of nitrogen in the Grade 91 specification, controlling the amount of $B \leq 0.001$ weight percent helps to prevent the formation of BN.

In practice, it appears that most suppliers of Grade 91 steel have controlled the levels of B to ≤0.001 *weight percent maximum*; this would be a sensible upper limit on the B content in alloy specifications for this steel.

2.6. Influence of Nickel (Ni). The original ORNL reports detailing the development for Grade 91 steel [24] recognized the risk that excessive Ni levels in Grade 91 steel could reduce the long-term creep strength. More recently, the results of long-term creep tests have confirmed that Ni level within the currently allowable range in alloy specifications can reduce the long-term creep strength [37]. Based on these results, estimated long-term creep lives for Ni at the maximum allowed (0.40 weight percent) would be below the minimum expected by the ASME code. The long-term detrimental effect of Ni is attributed to an increase in the rate of recovery of the microstructure and an increased tendency to form Z-phase. It has been reported that one of the main motivations behind having a specified limit for Ni of 0.40 weight percent maximum was the perceived benefit of being able to melt a wide range of steel scrap. Despite the recommendation by ORNL, when Grade 91 type steel was accepted by ASME [19], the nickel level was set at 0.40 weight percent maximum [17] as *it was believed to be a harmless element*.

The observations reported by NIMS [37] have recently been confirmed in work reported at the European Creep

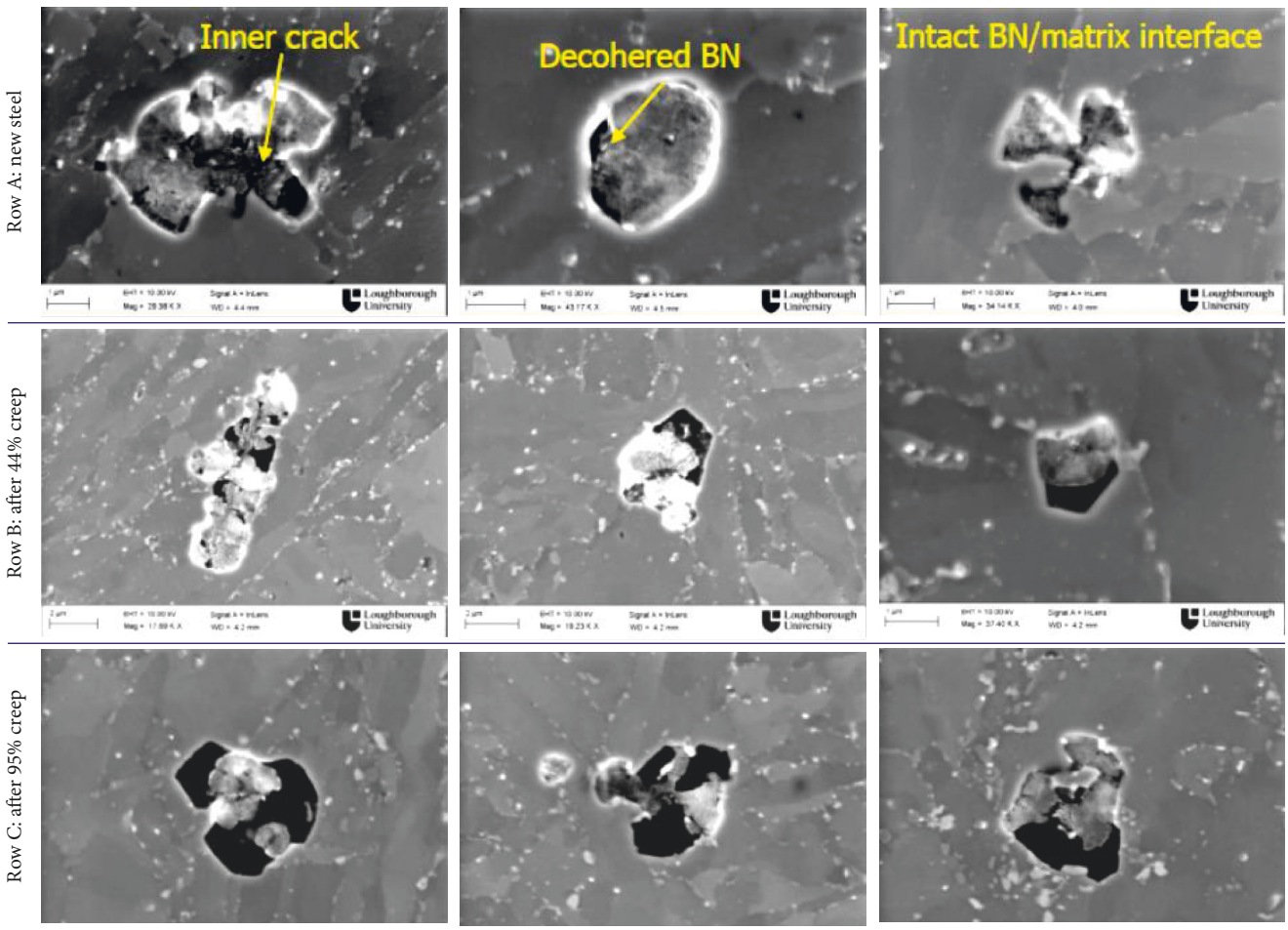

Figure 8: Micrographs from Grade 92 CSEF steel showing the development of creep cavities associated with BN particles, row A BN particles in new steel, row B after exposure to 44% creep life, and row C after exposure of 95% creep life.

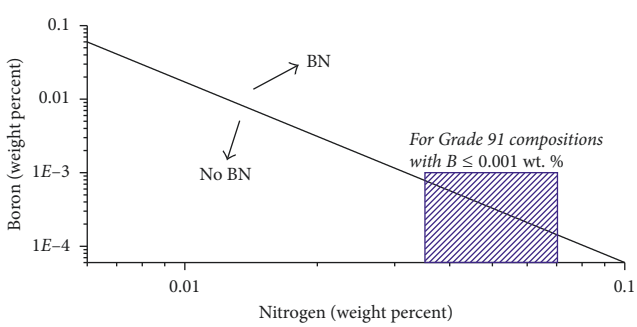

Figure 9: Relationship developed for the formation of BN in Grade 92 steel with different levels of B and N based on previous research [33]. The blue hashed box shows the range of N typically specified in Grade 91 steel for a B level of ≤0.001 weight percent.

Collaborative Committee (ECCC) [38]. It is noteworthy in that an early analysis of the ECCC database indicated no effect of Ni on performance of Grade 91 steel. More recently, however, the ECCC database was reevaluated using only long-term data for rupture lives >10,000 hours. Upon this reassessment of the ECCC database, the author remarked [38], "The long-term harmful influence of nickel on Grade 91 creep strength, proposed by Japanese researchers, is a real effect. . .. A restriction to 0.20 weight percent nickel maximum, as is now being introduced into many codes and standards is therefore to be strongly encouraged. . . Nevertheless, its effect [ref. nickel content] is comparable with that of aluminum, and should certainly be taken into account in component life management." It is thus concluded that excessive Ni can be a significant influence in long-term behavior with regard to strength.

A value of 0.20 weight percent maximum was first proposed in the ORNL summary report to ASME and ASTM in the early 1980s [19]. This recommendation was reinforced by the published maximum value in [21] and has more recently been recommended in [20] (Table 1). In practice, consideration of the maximum nickel value reported for components made to controlled compositions shows a value consistently below 0.20 weight percent [22–24, 26, 32].

There has never been any evidence reported to support the position that levels of Ni above 0.20 weight percent offer any benefit to the performance of Grade 91 steel components. It is thus concluded that this value should be regarded as an absolute maximum moving forward. Thus, in Grade 91

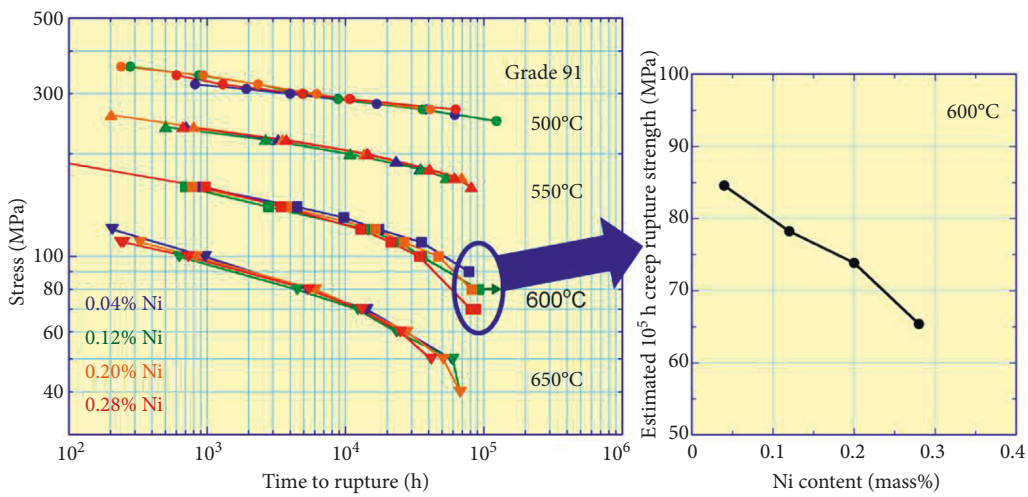

Figure 10: The influence of Ni on the creep strength in Grade 91 steel as reported by [37]. The detail at right shows that the 100,000 creep rupture strength decreases with increasing Ni content.

steel, a *maximum Ni content of 0.20 weight percent is recommended.*

2.7. Influence of the Nitrogen to Aluminum (N : Al) Ratio. As a result of postservice examination of Grade 91 steel components which cracked relatively early in life, a broad review of the composition of different Grade 91 components in UK power stations was undertaken. Because high levels of Al were commonly found in cracked components, this review also considered the challenges associated with accurate measurement of Al [39]. The results of this review supported the concept of a link between the ratio of N : Al present in the steel and the susceptibility for developing creep damage. This effect was explained on the basis that the formation of AlN was such that there was then insufficient nitrogen available to for the MX carbonitrides needed to ensure high temperature strength [39].

As a consequence of creep damage, a secondary superheater header was replaced after 79,000 hours. Background regarding the in-service experience with this header has been published previously [5]. Detailed root cause analysis was performed on sections of header which had developed hundreds of weld HAZ cracks in service. In summary, the HAZ cracking was found to be overwhelmingly associated with the three cylindrical sections (described as "barrels"). The two component sections with the highest percentage of cracks were identified as Barrel 2, >70% of stub welds cracked (N : Al = 1.42) and Barrel 5, >60% of stub welds cracked (N : Al = 1.57) (Figure 10). These two sections were reported to have been fabricated from the same ingot material. It is interesting to note that the third heavily damaged barrel, almost 50% cracking in the stub welds, Barrel 4 had an N : Al ratio of 3.67. An N : Al ratio of 3.67 is significantly higher than reported in earlier problem casts [8, 39]. Indeed, this ratio would be considered acceptable by today's ASME code requirements (Figure 11).

The reasons why an excessive N : Al ratio can be deleterious to performance of Grade 91 steel have been investigated [20]. One key finding was that when significant levels of Al are present, the AlN forms preferentially and so the amount of available N to form MX carbonitrides is reduced. In extreme cases, the number density of MX is not sufficient to stabilize the substructure.

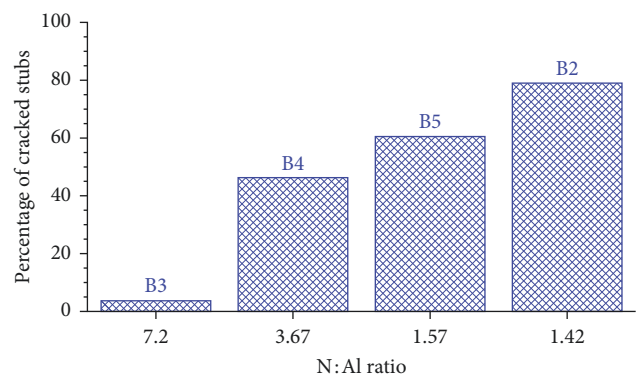

Figure 11: Analysis of the N : Al ratio for specific casts of Grade 91 steel as a function of the percentage of stubs cracked in a give barrel section. Adapted from [17].

The poorly stabilized substructure means that the steel has reduced resistance to tempering and shows a rapid degradation under creep conditions. The lower resistance to tempering has been shown in experiments tracking changes in hardness with increasing thermal exposure. These results which were obtained for Grade 91 steel samples with N : Al ratios of 1.3 G11, ~5 G8, and 13.5 J4 are summarized in Figure 12. For Grade 91 steel with very high N : Al ratios (J4), the resistance to tempering during exposure to elevated temperatures is remarkable. In this example, the examined material with a very high N : Al ratio (sample J4) exhibited a lower initial hardness value. However, for the most extreme condition, namely, tempering at 790°C (1450°F), that is, just below the A_1 temperature, for 50 hours, it can be seen that the J4 material has exceptional resistance to softening. Thus, even after a high degree of

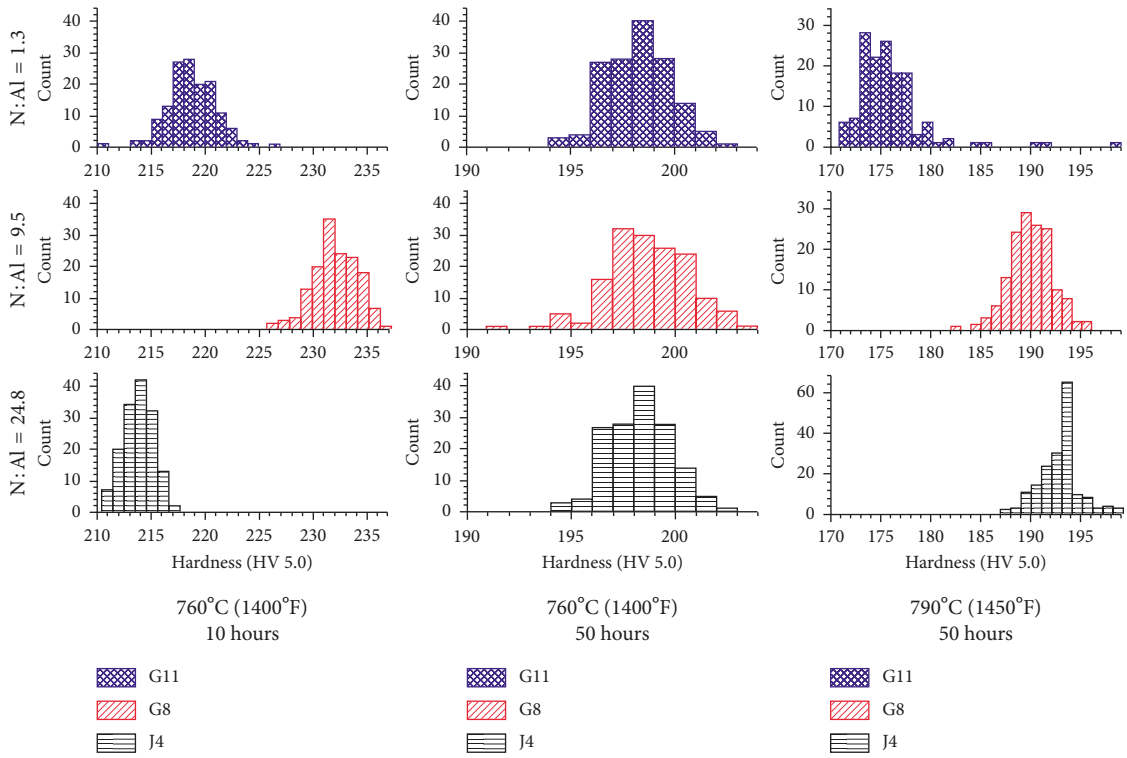

Figure 12: Analysis of the N : Al ratio on tempering behavior for specific casts of Grade 91 steel. Note that although steel J4 starts with the lowest initial hardness value, as demonstrated in the 760°C (1400°F) for 10 hours experiments, it exhibits the highest resistance to tempering and a more consistent (i.e., lower standard deviation) hardness for each examined tempering treatment.

tempering the measured hardness was above 190 Hv (Figure 12). In contrast, the steel with the lowest N : Al ratio, sample G11, has the least resistance to tempering. For this heat, tempering at 790°C (1450°F) for 50 hours reduced the vast majority of measured hardness values to less than 180 Hv (Figure 12).

No additional recommendations are suggested for control of the Al content in Grade 91 steels. Thus, to *reduce the risk of formation of AlN, it is recommended by EPRI that a minimum N : Al ratio of 4 should be adopted.*

3. Implications of Controlled Composition on Performance

The high temperature performance of CSEF steels is dependent on steel making, steel processing, composition, and heat treatment. It has been very well established that poor control of heat treatment leads to incorrect microstructures and poor high temperature performance. Thus, when irregularities of heat treatment result in a ferrite microstructure, the creep strength is significantly reduced compared to the creep strength of tempered martensite. In contrast, variability in the measured reduction in area during creep has been reported for steels exhibiting a tempered martensitic microstructure. This variability is such that at similar creep lifetimes, failure has been reported with either high ductility or low ductility (e.g., see Figure 13).

Posttest characterization of selected laboratory samples has shown that the low ductility failures were predominantly

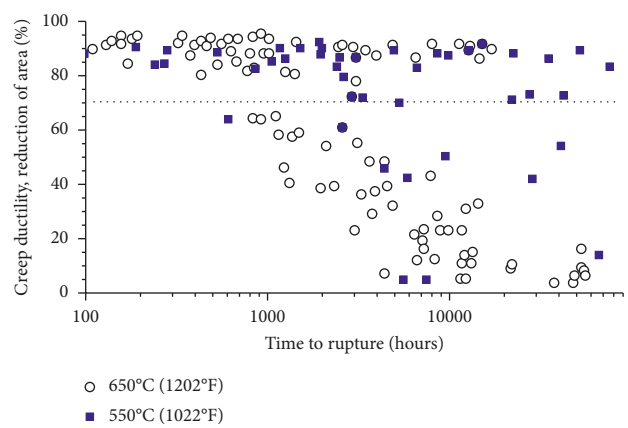

Figure 13: Creep ductility (reduction of area) for Grade 91 steel supplied by one manufacturer [40]. A dashed line is shown at 70% for the reduction of area since this was an acceptance criterion proposed by ORNL in the development of Grade 91 steel [20].

a consequence of the nucleation, growth, and link-up of cavities. The tendency for creep brittle behavior in CSEF steels is thus due to the formation of microvoids on prior austenite grain boundaries and at other microstructural features such as lath boundaries. The range of different sites for void nucleation means that in contrast to low allow steels, there are high number densities of creep voids present prior to microcrack formation. The diversity of void nucleation sites is also a challenge to tracking in-service component

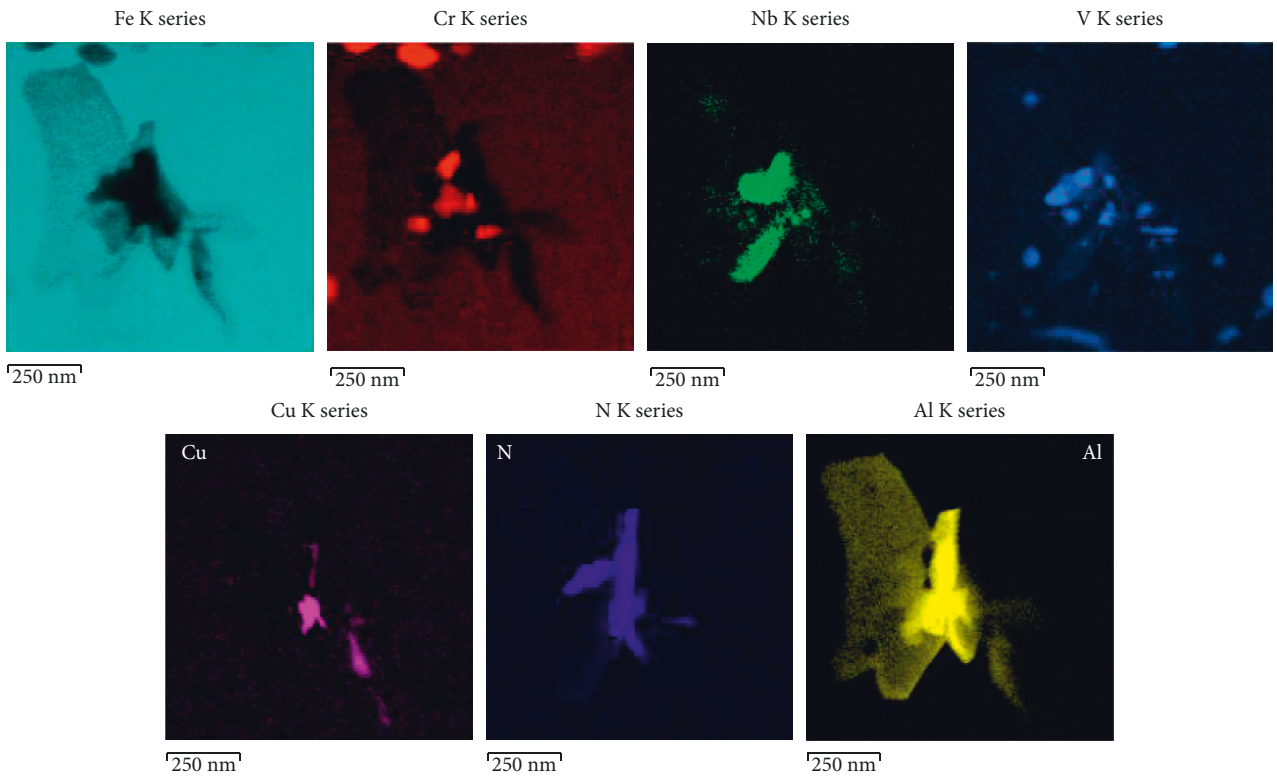

Figure 14: Detailed micrograph showing inclusions associated with an individual creep cavity as well as compositional maps showing the distribution of the elements Cr, Nb, V, Cu, N, and Al associated with this location.

damage using traditional inspection methods. While the details of the number of voids formed, and the tendency for reductions in strain to fracture, is different for the different CSEF steels, research to date shows that void nucleation is related to the presence of trace elements and hard non-metallic inclusions. An example of the link between the nucleation of a creep void, the presence of inclusions and segregation of copper is illustrated by the analysis results shown in Figure 14. A key factor in determining whether the inclusions 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 be thermodynamically stable and thus able to act directly as nucleation sites.

It is also clear from the recent root cause analyses of Grade 91 steel components [5, 40] that steel composition and processing variables are linked to low creep ductility in base metal and weld HAZs. The observed in-service component cracking cannot be simply explained as a 'one off' anomaly. Examination of failures in several Grade 91 welded components [40] indicates a trend where an increasing number of failures of Grade 91 welds are occurring in times below that expected based on design analysis. The trend in the reduced creep ductility in Grade 91 steel and the link to very low HAZ creep life may be a characteristic of a significant number of components which entered service with a high susceptibility for cavity formation. Poor performance of weld HAZs is a key reason for the introduction of weld-efficiency factors. Further, reductions in weld creep performance could lead to increases in recommended weld strength reduction factors (WSRF) or weld-efficiency factors. In contrast, using Grade 91 steel with low densities of inclusions and controlled levels of deleterious trace elements should significantly reduce the risk of creep cracking associated with weldments.

It is thus clear from detailed examination that the creep ROA is vastly improved when the levels of inclusions and impurities in Grade 91 steels are low. Metallographic characterization using advanced techniques has established detailed trends in behavior. To consider a broader assessment of *Metallurgical Risk*, data were collated from the results of specific test programs. Test information was obtained from global sources including from EPRI projects, ORNL, Japan, and Europe. Although efforts were made to consider a broad range of research results, accurate and comprehensive compositions linked to long-term creep data were only available for a smaller number of heats than was expected. Thus, rather than being able to make a quantitative analysis of recorded values of creep ROA, the creep performance factor was simply organized as

(i) high susceptibility to damage (poor creep ductility),

(ii) low susceptibility to damage (high creep ductility).

A total of 36 detailed data sets were available for analysis, and the results are presented in Figure 15. As shown, this assessment covered the following elements–Cr, Mo, W, V, Nb, Ti, C, Mn, Ni, Si, Al, N, Cu, S, P, As, Sn, Sb, and Pb. In Figure 15, the heats which exhibited low susceptibility to creep damage are shown as blue box with heats which

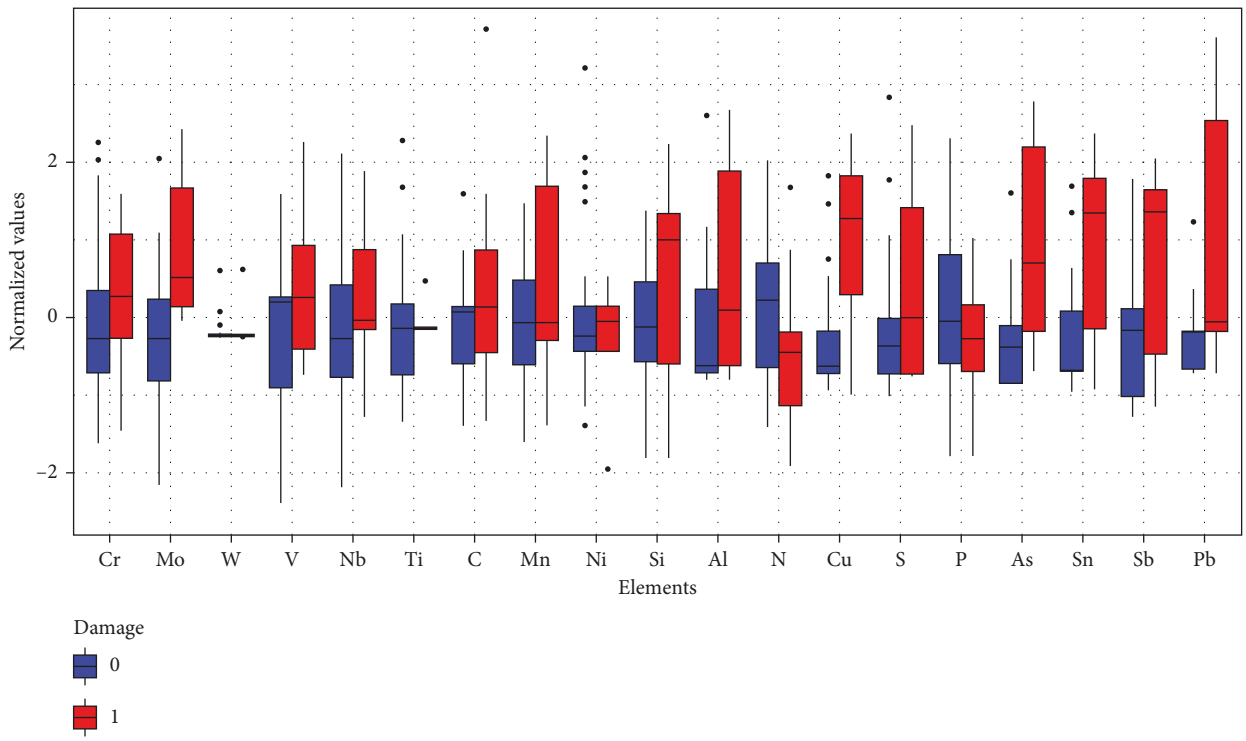

Figure 15: Trend showing how the level of the elements Mo, V, Nb, C, Mn, Si, Al, Cu, S, As, Sn, Sb, and Pb in Grade 91 steels varies for damage susceptible steels (red boxes) and damage resistant steels (blue boxes).

exhibited high susceptibility to creep damage shown as red boxes. The size of each of the boxes designates the range of each element in the data set with the horizontal line indicating the average value. It is apparent that the statistical trend shows that elements Mo, V, Nb, C, Mn, Si, Al, Cu, S, As, Sn, Sb, and Pb show a significantly higher level in the damage susceptible heats. An interpretation of these observations is that Mo, V, Nb, C, and Mn will be linked to creep strengthening and as such should be expected to enhance the deformation resistance of the steel. The elements Si, Al, Cu, S, As, Sn, Sb, and Pb are typically associated with the formation of inclusions and exhibit a tendency to promote the nucleation and growth of cavities. The combination of these trends is consistent with the previous metallographic examinations linked to observed decreases in creep ductility.

It is apparent that the results from the different assessment approaches show consistent outcomes; for example, for Grade 91 tempered martensitic steels, a controlled composition with minimal amounts of impurities and tramp elements can have a profound effect on the creep ductility. A controlled Grade 91 composition, specified as code case 2864 "Class 2" material, has recently been accepted by ASME. This Class 2 steel has a composition in line with the information presented in Table 1. It is believed these recommendations will have a dramatic effect on improving creep ductility and therefore provide significantly better in-service performance. These benefits will be particularly important for components operating under creep condition, that is, above about 550°C.

The trend showing that controlled composition and fabrication leads to improved behavior has been identified in other tempered martensitic steels. A comprehensive research program studying Grade 92 steel, from three distinct sources made in separate parts of the world, exhibited a range of behavior with regard to creep ductility. Clearly, the performance of "BM C" as opposed to "BM A" and "BM B" increased with respect to both creep strength and ductility. This improved creep performance was directly related to the controlled composition and variations in the size and distribution of inclusions in each of the steel heats.

For materials which may exhibit high levels of tramp elements, it would be advantageous to define a metallurgical risk factor. However, it must be emphasized that changes in ductility should be established for appropriate stress states, component geometry, and operational conditions. It is apparent that the component performance factors with respect to strength and ductility are different for changing operating regimes. For example, in the mid-2000s, the service experience with Grade 91 in the USA was particularly positive. It appears that this was mostly because the operating conditions were around 540°C (1004°F) and 240 bar (3,480 psi). Similarly, satisfactory performance has been reported from Europe. Clearly then the steam conditions of the power plant with respect to temperature and pressure are important in being able to anticipate future issues with performance of the Grade 91 material.

It is important to note that the chemical composition is only part of the overall proposal for more highly controlled Grade 91 steel. Research linked to understanding and

modelling of the factors affecting creep behaviour should consider careful control and selection of the following metallurgical factors—steel composition, steel making, hot working method conditions and degree of hot reduction, normalizing temperature and time, cooling rate from normalizing, and tempering temperature, time, and controls. The influence of inclusion content and impurity elements on performance is recognized from the present results as being synergistic, that is, when both high levels of trace elements and inclusions are present, the creep performance is very poor. The evidence from the present work is supported by data from publications and the results of root cause analysis of cracking in-service. It is also important that research programs consider the role of stress state, particularly as constraint effects have an influence of creep damage development.

4. Conclusions

Design codes for components operating at high temperatures have traditionally assessed expected performance based on an evaluation of creep strength, underpinned by the expectation that alloys would be creep ductile. Thus, for CrMo type low alloy steels, relationships which combine defined trends in tensile and creep strength with Factors of Safety have typically ensured that the agreed allowable stress values provide the basis for achieving at least minimum performance. The usefulness of the traditional approaches is severely challenged when assessing the behavior of metallurgically complex steels. In particular, problems are encountered when steels exhibit poor creep ductility. In circumstances of low creep ductility, local stress relaxation can promote creep damage. It is now well established that the creep properties of CSEF steels are critically dependent on factors which include control of composition, steel making, processing, and all heat treatments. Lack of correct control of these factors can severely compromise strength, ductility, or both. Indeed, in-service failures of Grade 91 steel components have occurred relatively early in life. The very large range of creep behavior observed in Grade 91 steel components introduces many risks for life management and assessment. The greatest concern is that, as opposed to the expected leak-before-break failure, a crack in low ductility material will propagate rapidly and cause catastrophic failure. Moreover, the existence of significant densities of voids prior to crack initiation complicates in-service assessment of condition and weld repair of these steels.

Changes in composition and heat treatment clearly have a marked effect on creep strength. Tempered martensitic steels such as X20, Grades 91, 92, E911, and 122 all exhibit a trend to relatively low ductility as creep rupture times increase. The tendency for brittle behavior is, in all cases, due to the formation of creep voids on prior austenite grain boundaries and at other microstructural features such as lath boundaries. The detail of the number of voids formed and the tendency for reductions in strain to fracture is different for each of the steel grades. However, it appears that void nucleation is related to the presence of trace elements and other metallurgical risk factors such as nonmetallic inclusions or evolution of Z-phase. It is clear that proper control of the composition in Grade 91 steel can result in an increase in the performance of the material with respect to creep ductility.

The link between creep ductility and in-service damage has been shown for a wide range of materials. To ensure that more consistent Grade 91 steel can be procured through the relevant suppliers, it is suggested that the composition be further controlled by ASME and other Design codes.

5. Declaration

The Electric Power Research Institute (EPRI) conducts research, development, and demonstration projects for the benefit of the public in the United States and internationally. As an independent, nonprofit organization for public interest energy and environmental research, we focus on electricity generation, delivery, and use in collaboration with the electricity sector, its stakeholders, and others to enhance the quality of life by making electric power safe, reliable, affordable, and environmentally responsible.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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