Grain Boundary Damage Evolution and Rupture Life of Service-exposed 1.25Cr–0.5Mo Steel Welds

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For components operated under creep regime, Type IV cracking would be the most likely failure mode if they were fabricated from ferritic steels. In the present work, creep behavior of 1.25Cr–0.5Mo steel weldment operated at 500°C for 28 years has been examined. The morphology of grain boundary damage was observed to clear the cause of Type IV cracking. Intergranular failure at the Intergranular HAZ took place in spite of short testing duration, which was less than 1 000 h. Change in feature of damage was observed with time to rupture. The number of cavities and the area of cavitated at failure increased as time to rupture became longer. Specimens broken with short duration showed few cavities resolvable by optical microscopy adjacent to the final intergranular crack path. On the other hand, more profuse cavitation in terms of number and area was observed in the specimens tested for 1 000 h and longer. However, commonly observable feature in both short and long term specimens was preferential damage occurrence at the triple points, proving that Type IV damage was caused by grain boundary sliding. The change in failure location in laboratory tests, where parent material failure often occurs due to too much acceleration, can be explained by relatively low stress exponent and activation energy associated with grain boundary sliding. Therefore, life assessment by conventionally employed Time–Temperature Parameter such as Larson–Miller Parameter could result in non-conservative estimation since equivalent *LMP* at failure would be smaller with increase in time to rupture.

KEY WORDS: Type IV cracking; 1.25Cr–0.5Mo steel; grain boundary sliding; Larson–Miller Parameter.

1. Introduction

In recent years, enormous efforts have been made to develop the remnant life assessment technique since operating periods of existing high temperature components have well exceeded the design life of 100 000 h in a lot of plants. Among them, intensive attention has been paid to Type IV cracking, which is preferential creep damage accumulation at the Intergranular HAZ (ICZ) and most often results in the ultimate failure for welded components. Despite seriousness of the problem, mechanism and enhancing factors of Type IV cracking have not been fully explained. Therefore, further research works will be necessary for the quantitative life assessment of weldment. The largest obstacle to examine the behavior of Type IV damage, especially for low alloy ferritic steels, would be long testing duration to produce Type IV cracking experimentally. In the previous work on cross-weld creep behavior of 1.25Cr–0.5Mo steel by the author and co-workers,^{1,2)} genuine Type IV cracking was not generated when tested without enhanced multiaxiality by application of the vee notch. It is noteworthy that the previously examined weldment, which had been in service for 23 years, had got service-induced creep damage at the ICZ. However, the welds showed higher resistance to Type IV cracking compared with that of the present welds removed from the service after 28 years of operation and containing no service induced creep cavity. Despite the absence

of cavitation before tests, ultimate intergranular failures at the ICZ were observed with shorter testing duration than previous work. Therefore, a simple question on the physical influence of grain boundary cavitation upon the creep strength of weldment was arisen. Furthermore, the feature of creep damage at grain boundaries was not necessarily the same as that expected in the actual components. The difference in damage morphology was more pronounced with decrease in time to rupture.

In this paper, interpretation on the damage mechanism for Type IV cracking from the observation of creep cavitation, which is often utilized for the remnant life assessment, shall be discussed. In addition, anticipated drawback of Larson–Miller Parameter, which is extensively used to estimate the remaining life, shall be also addressed.

2. Experimental Procedure

2.1. Materials

For the present work, girth weldment for straight pipe and elbow was removed from the service after 28 years of operation in a refining plant. External diameter and thickness was 508 mm and 22 mm, respectively. Operating temperature was approximately 500°C. Pipe and elbow were fabricated from plates, ASTM A 387 Gr.C. Chemical compositions of parent materials are shown in **Table 1**. Relatively large difference in chromium content between

Table 1. Chemical compositions of parent materials.

	Pipe	Elbow
C	0.10	0.15
Si	0.55	0.58
Mn	0.54	0.53
P	0.014	0.008
S	0.016	0.010
Cr	1.02	1.23
Mo	0.44	0.46
Cu	0.091	0.049
As	0.006	0.009
Sn	0.006	0.005
Sb	0.001	0.001

Fig. 1. Hardness distribution through weldment.

pipe and elbow is observed. Cr contained in pipe parent is close to the minimum level in ASTM standard. No other significant compositional difference, including impurities, can be found. Weld preparation was single V, and Manual Metal Arc (MMA) weld was applied except the root run where Tungsten Inert Gas (TIG) weld was employed. Post Weld Heat Treatment (PWHT) was conducted at 700°C for two hours.

Close metallurgical examination was made before creep tests, and no grain boundary damage was found through the weldment.

In **Fig. 1**, hardness distribution across the weldment measured in the middle of wall thickness is shown. The maximum value, Hv 195, is found at the center of weld metal. Hardness of pipe parent with lower Cr content is slightly lower than that of elbow. As for the ICZ hardness, identical value is observed despite higher susceptibility to Type IV failure on the pipe as described below. In addition, hardness at the pipe ICZ is Hv14 higher compared to base metal.

2.2. Experiments

Specimens for parent material and weldment were taken longitudinally and those for weld metal were taken circumferentially. All the specimens were a standardized cylindrical type with 10 mm of diameter, except the weld metal specimen with 6 mm of diameter since it was the maximum size available. The gauge length of each specimen was five times of diameter.

Grain boundary damage evolution at welds associated with life consumption was observed by sectioning the interrupted creep specimen since it had been pointed out Type IV damage was often maximized inside the wall^{1,3)} where higher hydrostatic stress was generated. Interruptions were

Table 2. Creep properties of parent materials.

Temp. (°C)	Stress (MPa)	Pipe			Elbow			Weld metal		
		t_r (h)	El (%)	RA (%)	t_r (h)	El (%)	RA (%)	t_r (h)	El (%)	RA (%)
610	100	31	55	91	71	61	91	196	36	87
630	60	512	53	91	804	58	90	712	29	87
630	80	65	66	93	93	63	91	263	40	88
650	40	1220	45	85	1696	45	90	1426	47	78
670	40	393	56	92	450	49	93	-	-	-

Fig. 2. Larson–Miller Parameter–stress correlation for homogeneous and cross-weld specimens.

made when strain reached approximately 5%. Life consumption of these specimens was around 70%. To quantify damage at the ICZ, the cavity density was employed and measurement was made based upon the methodology described in the paper by Walker *et al.*⁴⁾ An optical microscope with magnification of 450 times was applied for cavity counting. In addition, Scanning Electron Microscopy (SEM) was applied for closer observation of grain boundary damage since grain boundary damage of interrupted specimens was not fully resolvable with optical microscopy. For identification of precipitates, Energy Dispersive (EDX) X-ray Analysis was employed. Creep tests were conducted with a constant load technique in air.

3. Results

3.1. Creep Strength of Homogeneous Specimen

Testing results by homogeneous specimens (pipe, elbow and weld metal) are tabulated in **Table 2**, and rupture data including those for cross-weld specimens are plotted using Larson Miller Parameter ($C=20$) in **Fig. 2**. For the comparison, mean creep strength and lower boundary (mean strength- 3σ) of the NIMS data sheet for normalized and tempered 1.25Cr–0.5Mo–Si steel plate⁵⁾ is also shown. Despite significantly longer exposure to high temperature condition than design life of 100 000 h, all the experimental data still lie above the lower boundary of virgin materials. Creep strength of pipe with lower Cr is weaker than elbow at all the testing temperatures, from 610 to 670°C. This tendency is also consistent with the difference in hardness between pipe and elbow as shown in **Fig. 1**. Townsend *et al.*⁶⁾ correlated Vickers hardness with creep strength of parent material for new and service-exposed low alloy steels (1Cr–0.5Mo, 1.25Cr–0.5Mo and 2.25Cr–1Mo steel). In

Fig. 3. Comparison of creep strength between 1.25Cr-0.5Mo-Si steel and 1Cr-0.5Mo steel.

their work, it was found that time to rupture of parent material was predicted with accuracy of factor of three by simply measuring hardness. In the case of homogeneous microstructure like parent material, hardness, which will be a function of spacing of carbides preventing the dislocation movement, can be an effective indicator to predict creep strength. However, the most likely area to be failed in actual components fabricated from low alloy ferritic steels is the ICZ exhibiting higher hardness than parent material as shown in Fig. 1. Therefore, Vickers hardness is not representative of creep strength against Type IV failure.

Creep strength of weld metal is the strongest at high stresses, and it becomes similar to that of elbow when stressed at 60 MPa and lower. It can be presumed that higher strength of elbow is attributable to higher Cr content. In practice, a Japanese designing standard for pressure vessels (JIS B 8270) gives higher allowable stresses in the creep regime to 1.25Cr-0.5Mo-Si steel compared to 1Cr-0.5Mo steel. The same conclusion can be derived by comparing the data sheet for 1.25Cr-0.5Mo steel with that for 1Cr-0.5Mo steel⁷⁾ as shown in Fig. 3. It is interesting to note, however, allowable stresses of 1Cr-0.5Mo steel in these temperatures given by the equivalent American design code (ASME Sec. II) are higher than those of 1.25Cr-0.5Mo-Si steel at temperatures ranging from 482 to 510°C. Prager *et al.*⁸⁾ examined the difference in creep strength between 1Cr-0.5Mo steel (ASTM A387 Gr. 12) and 1.25Cr-0.5Mo-Si steel (ASTM A 387 Gr. 11), and found that 1Cr-0.5Mo steel was stronger in the above temperature range where these materials were mainly selected. At higher temperatures, clear difference was not appreciated. In addition to the effect of Cr, higher Si content could contribute to extension of experimental rupture life since it significantly improved oxidation resistance. Si content in Gr.11 ranges from 0.5 to 1%, and that in Gr. 12 is specified to be lower than 0.5%. Therefore, it might be necessary to test in vacuum in order to examine actual creep strength of these materials.

Table 3. Creep properties of weldment.

Temperature (°C)	Stress (MPa)	t_r (h)	El (%)	RA (%)	Failure location
610	60	1442	15	61	ICZ on the pipe side
630	60	544	22	71	ICZ on the pipe side
630	80	60	36	93	Parent material on the pipe side
650	40	1080	16	66	ICZ on the pipe side
650	50	351	19	81	ICZ on the pipe side
670	40	326	27	88	ICZ on the pipe side

3.2. Creep Strength of Cross-weld Specimen

The final failures have taken place only on the pipe side as shown in Table 3. Although high susceptibility to premature cracking at the Coarse Grained HAZ (CGZ) in 1Cr-0.5Mo and 1.25Cr-0.5Mo steel has been reported,^{9,10)} the ultimate failure with a Type III manner has not taken place. As shown in Fig. 2, time to Type IV failures lies above lower boundary of virgin materials in NIMS data, suggesting that service-exposed weld examined would have creep strength enabling another 100 000 h operation unless operating condition were largely changed. However, the preciseness of predicted life derived by extrapolation using Larson-Miller Parameter needs to be reviewed as discussed later. Ultimate failures at the ICZ were obtained except a test at 80 MPa despite relatively short testing duration. Brear *et al.*¹¹⁾ pointed out the long term nature of Type IV cracking and proposed the map for 2.25Cr-1Mo steel weldment to predict the failure location that is uniquely determined by rupture life. In their work, it was addressed that too much acceleration in terms of stress and temperature would result in failures at parent material, CGZ or weld metal. According to the map described above, time to onset of the Type IV failure at 600°C becomes longer than 8 000 h.

In the present work, however, ultimate failure at the outer edge of HAZ took place with short term tests, the shortest one was only 326 h. In the work by Fujibayashi *et al.*¹⁾ using old 1.25Cr-0.5Mo steel pipe/flange girth welds operated for 23 years at 500°C, genuine Type IV failure was not

Fig. 4. Feature of the final crack path of specimen failed at 326 h (tested at 670°C and 40 MPa).

observed in the experiments using 12×12 mm² square cross-weld specimens. Although testing conditions in terms of stresses and temperatures were similar to the present work, welds did not fail at the ICZ up to 2 700 h. Most often, the failure took place at the parent material on the pipe side. However, it was found by the testing using notched specimens that ICZ on the flange side whose parent material showed higher creep strength than pipe parent was more susceptible to Type IV cracking. From the findings described above, it can be considered that the susceptibility to Type IV cracking is not determined simply by creep strength of base metal. It was pointed out that higher susceptibility could be arisen from higher non-metallic inclusion,¹²⁾ higher impurities^{2,13)} and morphology of fine grains, which became preferential cavity nucleus sites.²⁾ Further research on factors determining the sensitivity to this phenomenon should be conducted.

3.3. Morphology of Grain Boundary Damage

Feature of the final crack path of the specimen broken with the shortest duration (tested at 670°C and 40 MPa) is shown in Fig. 4. Significant deformation of grains to tensile direction can be seen and few grain boundary cavities are found adjacent to the final crack path by optical microscopy. In Fig. 5, SEM observation of the interrupted specimen ($t/t_r \approx 0.7$) is shown. Wedge cracking at the triple point is associated with sharp cracks located at grain boundaries which are inclined to tensile direction. From this feature, it can be considered that a wedge crack was generated by grain boundary sliding resulting in sharp cracks at inclined grain boundaries. And grain boundary sliding, which should precede the generation of wedge cracking, takes place at fine grain whose grain size is smaller than 10 μm. In contrast, more profuse cavitation is generated near the final crack path in the specimen tested at the same stress and lower temperature (650°C, 40 MPa) as shown in Fig. 6. Cavity density near the final crack path became 1 171 (n/mm²). In Fig. 7, grain boundary damage observed by SEM at the interruption of $t/t_r \approx 0.7$ is shown. Cavities are preferentially located at triple junctions. In ad-

Fig. 5. Feature of grain boundary damage of an interrupted specimen observed by SEM (tested at 670°C and 40 MPa). The test was interrupted at 224 h and strain at the interruption was 5%.

dition to triple points for nucleus sites of grain boundary damage, cavitation associated with non-metallic inclusion was also found as shown in Fig. 8. Cavities are nucleated at the interface of a elongated precipitate despite that damaged grain boundary is parallel to tensile direction. This precipitate was identified as manganese sulfide by EDX as shown in Fig. 9.

Walker *et al.*⁴⁾ found that cavity density at Type IV failure in 0.5Cr–0.5Mo–0.25V steel decreased with the increase in testing stresses. They ascribed negative dependency of cavity density upon stress to accommodation of grain boundary sliding by larger deformation inside the grains. However, cavity density was independent of testing temperatures ranging from 600 to 640°C unlike experimental results in the present work, where cavity density at the failure is the function of both stress and temperature. Type IV cracking was not associated with cavities resolvable by an optical microscope near the final crack path when testing duration was shorter than 1 000 h. In addition, grain bound-

Fig. 6. Feature of the final crack path of specimen failed at 1080 h (tested at 650°C and 40 MPa).

Fig. 7. Typical feature of grain boundary damage of an interrupted specimen observed by SEM (tested at 670°C and 40 MPa). The test was interrupted at 743 h and strain at the interruption was 5%.

Fig. 8. Cavitation at the interface of manganese sulfide observed by SEM (tested at 670°C and 40 MPa). The test was interrupted at 743 h and strain at the interruption was 5%.

Fig. 9. Energy dispersive X-ray spectrum of the elongated precipitate in Fig. 8.

Fig. 10. Temperature compensated rupture time-stress correlation for Type IV failure and parent material failure.

ary damage tended to spread from the CGZ to ICZ as time to rupture became long. The tendency of spreading cavity generation in the actual power generating components was reported by Parker *et al.*¹⁴⁾

3.4. Time to Type IV Failure–Stress and Temperature Correlation

Although difference in the morphology of grain boundary damage at failure was observed, time to Type IV failure was consistently described by power law relationship given by Eq. (1).

$$t_r = B\sigma^n \exp(Q/RT) \dots \dots \dots (1)$$

where B is the constant, Q is the activation energy, R is the universal gas constant and T is the absolute temperature in Kelvin.

In Fig. 10, relationship between stress and temperature compensated rupture time ($\ln t_r - Q/RT$) for pipe parent and cross-weld specimen failed at the outedge of HAZ is shown. Activation energy for pipe parent and Type IV failure is 439 kJ/mol and 383 kJ/mol, respectively. As for the stress exponent n , 6.7 for base metal of pipe and 4.5 for Type IV failures were derived. Lower values of activation energy and stress exponent for Type IV failure suggests that low temperatures and low stresses, which are expected in actual components, are preferred for Type IV failure. And lower stress exponent and activation energy are commonly found characteristics in grain boundary sliding.¹⁵⁾

4. Discussion

For users operating old high temperature components, the technique to assess the remnant life should be a great concern. Although a couple of techniques have been developed, each technique has its own advantage and disadvantage. A replication technique for direct observation of grain boundary damage, which is often employed in situ, is associated with following problems. Firstly, grain boundary damage is not always the highest at the external or internal surface where this technique is available. Secondly, apparent grain boundary damage such as cavity density is not necessarily representative of the life fraction consumed as observed in the present work. To predict the remnant life quantitatively from grain boundary damage, the extent of

damage at failure must be known. Bissel *et al.*¹⁶⁾ proposed the following equation to correlate cavity density with the remaining life.

$$t/t_r = 1 - (1 - N/N_r)^\Lambda \dots \dots \dots (2)$$

where t_r is the time to rupture, N is cavity density at time t , N_r is the cavity density at failure and Λ is the tertiary ductility ratio.

If N_r were the function of both stress and temperature, enormous data would be required for quantitative life assessment. In addition, N_r might not be a material constant and could significantly vary from cast to cast. For example, Westwood¹⁷⁾ reported very low values of cavity density in the work for 1.25Cr–0.5Mo steel welds. The cavity density at the failure, tested at 600°C and 40 MPa using a square cross-weld specimen, was only 800 in spite of 37 000 h of testing duration. It can be considered that highly populated and spreading cavity formation is resultant from higher contribution of grain boundary sliding to damage evolution. As discussed above, the stress exponent n and activation energy Q in Eq. (1) become lower than those of ductile transgranular failure in base metal when associated with grain boundary sliding. Therefore, contribution of grain boundary sliding to total strain accumulation will be larger by reducing stress and temperature. With decrease in time to rupture, grain boundary damage should be concentrated on particular grain boundary where sliding readily takes place. As described below, it is considered that the microstructure at the ICZ has got the characteristic to promote the localized grain boundary sliding. In the work on Type IV cracking for 0.5Cr–0.5Mo–0.25V steel by Walker *et al.*,⁴⁾ it was found that cavitation preferentially took place at fine grains whose grain sizes were smaller than 5 μm . The similar finding was made in the work on 1.25Cr–0.5Mo steel by Fujibayashi *et al.*²⁾ They found that 90% of cavities were associated with fine grains which were smaller than 10 μm . It can be considered that fine grains at the ICZ are products of partial transformation during welding. Therefore, difference in creep properties between newly formed grains and untransformed grains can be expected. From higher micro-Vickers hardness of fine grains compared with that of large grains, they concluded that fine grains owned higher creep strength and concentrated damage around fine grains was attributable to preferential grain boundary sliding to accommodate to higher strain of larger grains. This bimodal feature would promote the localized damage at the ICZ and this tendency would be more pronounced under high strain rate, which was an inverse function of time to rupture.

In considering the physical meaning of grain boundary cavitation, the following finding on damage evolution will be important. Gooch *et al.*¹⁸⁾ reported in the work on Type IV cracking for 0.5Cr–0.5Mo–0.25V steel that the significant difference in grain boundary damage between at failure and half life was the size of cavity rather than the number of cavities. Because of this tendency, Kimura and co-workers¹⁹⁾ applied void area fraction as a parameter to predict life consumption of welds for modified 9Cr–1Mo steel. It should be noted that significant difference in this parameter between their experimental work and that for the same material by Tanoue *et al.*²⁰⁾ can be found. Despite similar testing conditions and resultant similar rupture lives (tested

at the same temperature of 650°C and stress was 49 MPa for the former and 58.8 MPa for the latter), void area ratio at failure was 8% and 1%, respectively. Though difference between the two is significant, void area ratios are still rather low. Therefore, it can be also interpreted that formation of cavities is not the cause of further damage leading to higher loading upon uncavitated area but simply the result of grain boundary sliding. And resultant number of cavities or void area ratio might depend upon original microstructure such as the distribution of fine grains. Further research on the physical meaning of cavities at grain boundaries will be necessary.

Currently, the most reliable testing technique for the remnant life assessment should be iso-stress extrapolation if destructive tests were allowed. Viswanathan *et al.*²¹⁾ compared the predicted life by iso-stress extrapolation with that by Larson–Miller Parameter using repaired welds of service-exposed 1.25Cr–0.5Mo steel. For weld repair using MAW, the temper bead technique to refine microstructure at HAZ was employed in order to avoid premature cracking at the CGZ. Two types of specimen were prepared, namely, repair weld with PWHT at 704°C for 1/2 h and without. In their work, it was found that predicted lives based upon $\ln t_r - T$ or $\ln t_r - 1/T$ correlation derived by iso-stress extrapolation became significantly shorter than those derived by the extrapolation of Larson–Miller Parameter. In the case of Larson–Miller Parameter, extrapolation from higher stressed data, ranging from 55.2 to 103.4 MPa, was applied to estimate the life at 49 MPa. Repair welds showed good ductility at stresses higher than 49 MPa, Reduction of Area (RA) well exceeded 50% except one case. In Fig. 11, relationship between $1/T$ and LMP for the current work and that by Viswanathan *et al.* is shown. Iso-stress creep rupture tests for repair welds were conducted at 49 MPa at temperatures ranging from 621 to 676°C. LMP values at failures for repair welds with PWHT show abrupt decrease when $1/T$ is 0.001101 and higher (635°C and lower). And the drop of LMP value is associated with decrease in RA. RA of three specimens tested at higher temperatures and showing larger LMP values were higher than 50%. On the other hand, RA of those tested at low temperatures and showing lower LMP

became lower than 50%. Though they did not address the ultimate failure location, decrease in ductility at failure suggests the occurrence of change in failure mode from ductile parent material failure to Type IV cracking. The fact that time to rupture of the specimens failed with a brittle manner can be significantly shorter than that showing high ductility suggests that fracture mode must be identical to predict the creep life of welds by iso-stress extrapolation. As shown in Fig. 11, extrapolation using these data set will result in pessimistic life prediction. In the case of cold repaired welds, RA of all the specimens became lower than 50%. LMP values continuously decrease with time to rupture and LMP which is equivalent to predicted rupture life derived by iso-stress extrapolation become rather small in comparison with experimental data at higher temperatures.

In the application of Larson–Miller Parameter, relationship between logarithmic life $\log t_r$ and $1/T$ is assumed to be a straight line for each testing stress with the intercept of $-C (-20)$. And the slope of a straight line becomes Larson–Miller Parameter. Therefore, apparent activation energy derived from $\log t_r - 1/T$ correlation becomes higher with decrease in stresses. Although difference in rupture lives between pipe parent and ICZ generated in pipe was not remarkable in the present work, predicted time to Type IV failure using LMP should be less conservative with decrease in testing temperature. As previously noted, the activation energy for Type IV failure in the present work becomes 383 kJ/mol which is equivalent to LMP value of 20 000 when C of 20 is employed. With decrease in stress, equivalent LMP should be higher and it would result in a larger error with decrease in temperature.

As discussed above, it often takes long h to break weldment with a Type IV manner. Therefore, iso-stress extrapolation which requires at least four cross-weld specimens and gives the estimation at only one stress might be too time consuming. The extrapolation based upon power law relationship employed in the present work might be a compromising method though suitability must be examined in longer term tests.

5. Summary

The findings in the present work on creep behavior of the service-exposed 1.25Cr–0.5Mo steel welds should be summarized as described below.

(1) Large difference in the susceptibility to Type IV cracking was observed in the service-exposed 1.25Cr–0.5Mo welds. In the present work, ultimate failures with a Type IV manner have taken place at the ICZ generated in the parent material with lower chromium content and lower creep strength. However, it should be born in mind that other factors determining the susceptibility to Type IV cracking could exist.

(2) It was found by SEM observation that Type IV cracking was promoted by grain boundary sliding, resulting in grain boundary damage at triple points.

(3) Cavity density at failure increased with time to rupture. Increase in temperature and stress resulted in Type IV cracking with fewer cavities around final crack path since relative contribution of grain boundary sliding became smaller.

(4) To predict life of weldment by destructive tests, the same failure mode must be obtained in experiments. Time to rupture becomes shorter when Type IV failure occurs in comparison with failure at parent material. The difference in rupture life will be more pronounced with decrease in testing temperatures because of smaller activation energy associated with Type IV cracking. Therefore, extrapolation using Time–Temperature Parameter such as Larson–Miller Parameter could give non-conservative life prediction.

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