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Probing the deterioration of 316L stainless steel welds due to ageing and creep by indentation creep tests

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ARTICLE INFO

Article history: Received 19 April 2011 Received in revised form 26 August 2011 Accepted 12 September 2011

ABSTRACT

Authors have probed into the creep behaviour of AISI 316L stainless steel welds through the indentation creep test methodology and assessed the deterioration effects of these welds under different ageing conditions subjecting them to different test conditions. Comparison is made between the parent metal and the weld metal for integrity at different levels of ageing and test loads. It is concluded that although the aged weld's deteriorated status is not revealed at low temperature and low load test conditions, it is explicitly revealed when tested at higher temperature and higher loads. Microstructural evidences have been given by the authors and they have suggested mechanisms of creep at different test conditions.

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1. Introduction

Indentation creep testing has evolved as one of the useful laboratory techniques to characterize the given material that is normally available in small samples and for those where obtaining the standard test specimens of the material in the given condition is practically impossible. Aged weld material samples and cut samples of a failed component are the ideal samples for this type of test because no other test can substitute this test in characterizing the sample given. In this test, sample is made to creep by the indentation load of given magnitude and the creep strain is measured using the linear variable differential transformer (LVDT) instrument at different intervals of time so that creep rates can be evaluated. The results of the experiments conducted at different temperatures with different loads will enable the investigator to catch the accelerated deformation conditions under creep which further can be probed using scanning electron microscope (SEM) to assess the condition of the sample at the area of indentation/to look into the presence of voids, precipitates or cracks in the sample. Such a probe can depict the point of initiation of failure.

AISI 316L stainless steel is the structural material for nuclear power plants and its creep properties are of utmost importance to material scientists. In the present investigation, welds of this steel are tested along with the parent metal to probe into the creep behaviour of the material.

2. Experimental details

AISI 316L stainless steel weld samples and base metal samples differed slightly in their chemical compositions which are presented in Table 1.

Welding beads of around 10mm width and 10mm depth were drawn on the AISI 316L stainless steel plates of dimensions $200 \text{ mm} \times 100 \text{ mm} \times 12 \text{ mm}$ thick using TIG welding machine. Continuous TIG welding with welding current of 180A was chosen for operation keeping the electrode tip to base metal distance of 2 mm and welding speed of 5 mm/min. Argon with nitrogen gas was made to flow at flow rate of 121/min during welding. Implantation technique was used to introduce Cr into the welded region. These welded plates were chosen for samples preparation.

Rectangular pieces were cut at every 15 mm from one end from the above welded plates and further sample pieces were cut to the dimensions of $10 \text{ mm} \times 10 \text{ mm} \times 10 \text{ mm}$ at the welded portion as well as at the region of parent metal. These samples were used for all the creep tests.

Apparatus used for carrying out the impression creep test is the split cage-lever type with linear variable differential transformer (LVDT) probe mounted for measurement of creep strain, as shown in Fig. 1.

The split cage is made of superalloy Nimonics 90 to ensure error free strain readings during high temperature tests. Sample for the test is fixed at cage bottom while indenter which is of tungsten carbide, cylindrical in shape with 1 mm diameter is fixed at the top of the cage - sample moves upwards so as to affect indentation when

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^{0029-5493/\$ -} see front matter © 2011 Elsevier B.V. All rights reserved. doi:10.1016/j.nucengdes.2011.09.012



Fig. 1. Indentation creep test set-up (schematic). (1) Specimen, (2) indenter, (3) furnace/chamber of test, (4) fixed frame of cage, (5) moving frame of the cage, (6) thermocouple, (7) vertical rod, (8) cooling coil, (9) LVDT, (10) argon gas cylinder, (11) balancing weight, (12) weights, and (13) dashpot system.

the load is put into position during the test. It should be ensured that sample surface is perfectly flat and pull rod is exactly perpendicular to this surface before the start of the test.

Base metal and weld metal samples of 316L were aged for different durations of time at selected values of temperature (so that 26 samples were got) and each of the samples was tested at 5 different temperatures with 4 different loads. The heat treatment was carried out in a muffle type electrical furnace in which temperature is automatically controlled with an accuracy of ± 5 °C. Table 2 gives the scheme of ageing and of testing of the said samples.

Stresses corresponding to the test loads were 440 MPa (8 kg), 495 MPa (9 kg), 550 MPa (10 kg), and 660 MPa (12 kg). With a constant lever ratio maintained between rod carrying weights and the one connected to cage, each 1 kg addition of load results in incremental stress of 55 MPa in the present test set-up. Each test was repeated twice to confirm consistency. For every new test, sample surface was polished afresh. Test results are recorded in PC and the creep curves were drawn for the analysis. Argon gas was passed during indentation creep testing at higher temperatures. Each test was conducted for at least 3 h.

3. Results

Table 3 shows the steady state creep strain rate values calculated from the creep curves obtained after conduct of experiments with 12 kg load. Similar tables are obtained for experiments conducted with other loads.

The following inferences could be drawn from the various tables on SSCR values:

(i) The ageing treatment on AISI 316L stainless steel welds seems to have a limited influence on SSCR values, particularly for

Table 1 Chemical composition of 316L stainless steel – parent metal and weld metal (wt%).

	С	Si	Mn	Cr	Ni	Mo	Nb	S
Parent metal	0.03	0.62	1.1	18.6	12.7	2.0	0.17	0.012
Weld metal	0.03	0.62	2.14	21.3	12.8	2.5	0.19	0.012

those aged at lower temperatures whereas parent metal does not respond to ageing treatment at all.

(ii) All the samples register higher SSCR values when tested at 12 kg load compared to similarly aged materials tested at 10 kg and 8 kg for all the conditions of testing temperatures. The increase in SSCR values with loads is quite significant when testing is carried out at higher temperatures, say at 700 °C. This fact is depicted in the graphs presented in Fig. 2 for the sample aged at 300 °C. Samples aged at 400 °C and 500 °C too follow the trend of those aged at 300 °C without any qualitative difference. The small variation in the values, though fall into certain trend, needs to be neglected as difference is less than the expected experimental error.

The effect of ageing on the creep behaviour of the weldment is perceptible only when testing is carried out with higher loads at higher temperatures, particularly for those samples aged at higher temperature.

(iii) In most of the cases, the increase in SSCR values with temperature of testing is only a marginal one. But there is visible change in the behaviour at higher load of testing, particularly at load of 12 kg. It is seen that they remain almost constant at lower range of testing temperature only to show a marked increase



Fig. 2. SSCR values plotted vs. testing loads for 316L weld samples aged at 300 °C.

Table 2	
cheme of ageing and indentation creep testing of 316L stainless steel samples	;.

Sl. no.	Sample condition (base	metal/weld)	Testing temperature (°C) [for each temperature, test load = 8, 9, 10, 12 kg]						
	Ageing temp (°C)	Time of ageing (h)	Room temp (32)	200	300	500			
1	-	(Unaged)	\checkmark	\checkmark	\checkmark	\checkmark			
2	300	500	\checkmark	\checkmark	\checkmark	\checkmark			
3		1000		~		~			
4		1500							
5		2000				\checkmark			
6	400	500	\checkmark	\checkmark	\checkmark	\checkmark			
7		1000							
8		1500	\sim						
9		2000							
10	500	500	\checkmark	\checkmark	\checkmark	\checkmark			
11		1000		~	, V	~			
12		1500							
13		2000							

at 500 °C and above. A typical of such a relation is depicted in the profile presented in Fig. 3. It also reveals that change in SSCR values with respect to loads is considerable only for those samples tested at 700 °C.

(iv) In general, creep resistance of weld metal is as good as or marginally better than parent metal. However, there are two exceptional situations. They are: one, weld samples aged at 500 °C for 2000 h exhibit considerably higher SSCR values compared to similarly aged parent metal when tested with 12 kg load at 500 °C. Two weld samples aged at 500 °C exhibit much higher SSCR values compared to similarly aged parent metals when tested with 12 kg load at 700 °C. The value of activation energy of indentation creep of the aged weld metal and parent metal at a load of 12 kg are presented in Table 4. Activation energy is calculated using the relation

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$$Q = \frac{R \ln(\hat{\varepsilon}_1 / \hat{\varepsilon}_2)}{1 / T_2 - 1 / T_1}$$

where Q is the activation energy for creep, $\hat{\varepsilon}_1$ and $\hat{\varepsilon}_2$ are the strain rates at temperatures T_1 and T_2 (absolute scale), and R is the Universal Gas Constant.

It is seen that values of activation energy remain almost constant in the range of 9-12 kJ/mol for all categories of samples except for the ones which are aged at 500 °C. Moreover, the latter samples show variation in activation energy in different range of

Table 3

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Stords	r ctato crooi	n rnto (VALUAC FOR		in Indentation	croon too	ne thet load	· • • • • • • • • • • • • • • • • • • •	ICTOO	n rates are	in Illa	/ w/ith ii		/ רח רח / ר	ուոլ
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Ageing temperature (°C)	Ageing time (h)	Weld metal				
		$\hat{arepsilon}_1$ at 32 °C	$\acute{arepsilon}_2$ at 200 $^\circ$ C	έ₃ at 300 °C	$\acute{arepsilon}_4$ at 500 $^\circ$ C	$\acute{arepsilon}_5$ at 700 $^\circ$ C
Unaged	-	1.29	2.71	5.24	8.76	9.62
300	500	1.29	2.86	5.88	8.75	9.60
	1000	1.25	2.68	4.57	8.76	9.62
	1500	1.09	2.17	4.02	7.93	9.58
	2000	1.47	2.36	3.99	8.04	9.38
400	500	1.44	2.76	5.06	8.10	9.51
	1000	1.24	2.33	4.11	8.04	9.30
	1500	1.09	2.17	4.02	7.93	9.57
	2000	0.99	2.11	3.82	7.84	9.31
500	500	1.42	2.71	4.96	8.00	9.43
	1000	1.22	2.29	4.56	7.94	9.10
	1500	1.09	2.17	6.02	9.93	10.2
	2000	1.09	2.61	6.72	17.6	21.7
Ageing temperature (°C)	Ageing time (h)	Parent metal				
		$\hat{arepsilon}_1$ at 32 °C	$\hat{arepsilon}_2$ at 200 $^\circ$ C	É₃ at 300°C	<i>É</i> ₄ at 500 °C	$𝔅_5$ at 700 °C
Unaged	_	1.59	3.57	7.13	9.39	9.73
300	500	1.53	3.57	7.13	9.39	9.73
	1000	1.53	3.57	7.13	9.39	9.73
	1500	1.53	3.57	7.13	9.32	9.64
	2000	1.52	3.45	7.07	9.19	9.43
400	500	1.53	3.57	7.13	9.39	9.73
	1000	1.53	3.57	7.13	9.39	9.73
	1500	1.53	3.57	7.13	9.32	9.64
	2000	1.52	3.45	7.07	9.19	9.43
500	500	1.53	3.57	7.13	9.39	9.73
	1000	1.53	3.57	7.13	9.39	9.73
	1500	1.51	3.55	7.10	9.29	9.61
	2000	1.51	3.41	7.02	9.16	9.38

Table 4

Activation energy (Q) values for 316L stainless steel weld metal and parent metal tested at 12 kg load [creep rates are in 10e-4 with unit mm/mm].

Ageing temperature (°C)	Ageing time (h)	Weld metal		Parent metal		Q values (kJ/mol)		
		$\hat{arepsilon}_1$ at 32 °C	<i>ɛ̂</i> 2 at 300 °C	$\hat{\varepsilon}_1$ at 32 °C	$\hat{arepsilon}_2$ at 300 °C	Weld metal	Parent metal	
(A) Values in the lower temp	erature range							
Unaged	-	1.29	5.24	1.59	7.13	7.57	8.10	
300	2000	1.47	3.99	1.52	7.07	5.38	8.30	
400	2000	0.99	3.82	1.52	7.07	7.29	8.30	
500	2000	1.09	6.72	1.51	7.02	9.82	8.30	
Ageing temperature (°C)	Ageing time (h)	Weld metal		Parent metal		Q values (kJ/mol)		
		$\hat{arepsilon}_1$ at 500 °C	É₂ at 700°C	$\acute{arepsilon}_1$ at 500 °C	$\hat{arepsilon}_2$ at 700 °C	Weld metal	Parent metal	
(B) Values in the higher temp	oerature range							
Unaged	-	8.76	9.62	9.39	9.73	10.52	9.41	
300	2000	8.04	9.38	9.19	9.43	12.86	9.45	
400	2000	7.84	9.31	9.19	9.43	12.65	9.45	
500	2000	17.6	21.7	9.16	9.38	16.36	9.05	



Fig. 3. Creep rate vs. test temperature profiles at three different loads for weld sample of 316L aged at 500 $^\circ\text{C}$ for 2000 h.

testing temperature, unlike other samples. These exceptional samples have registered the activation energy of 16.4 kJ/mol at higher temperature level

Assuming the relation

 $\hat{\varepsilon} = k \times \sigma^n$

between creep rate $\hat{\varepsilon}$ and stress σ , where *k* is a constant and *n*, the stress exponent, *n* can be calculated for which values of creep rates at two different stress levels are to be taken, so that

$$n = \frac{\log(\hat{\varepsilon}_2/\hat{\varepsilon}_1)}{\log(\sigma_2/\sigma_1)}$$

Stress exponent values for a set of tests are depicted in Table 5.

Stress exponent value remains almost constant in the range of 6-6.8 for all the categories of samples except for one sample which has been aged for 500 °C. This exceptional sample registers a stress exponent value of 8.4.

Ferrite number values for the weld samples are recorded in Table 6.

It is noted that ferrite content of the weld metal is affected significantly by ageing process. Ferrite content decreases from 3.4 for unaged (welded) samples to 2.0 for the ones aged at 500 °C for 2000 h. The decrease in ferrite number values is significant when ageing is carried out at 500 °C. Parent metal has not registered any ferrite number values in any of the differently aged samples.

4. Discussion

4.1. Creep behaviour of parent metal

It is obvious that ageing has absolutely no influence on creep behaviour of parent metal. For these materials SSCR values change only with testing load and temperature. It is expected as parent metal consists of single phase, equi-axed, stable austenite phase. Delta ferrite is not present in the parent metal. So, any possibility of delta-ferrite transforming to other intermediate phase on ageing is ruled out.

4.2. Creep behaviour of weld metal

Microstructure of weld metal is a solidified complex cast structure as revealed by SEM photomicrograph presented in Fig. 4(a) and (b). It illustrates how delta ferrite precipitates over the cell boundary region. Delta ferrite undergoes transformation resulting in precipitation of some intermediate phase and carbide on ageing at higher temperature. It is expected that change in the microstructure on ageing would influence the creep behaviour of the weld metal. However, contrary to expectation, the influence of ageing on SSCR values is not revealed when tested at a lower load. Further, influence of ageing can be seen for the samples tested at higher loads of 10 and 12 kg.

Table 5

Stress exponent values for 316L stainless steel weld metal and parent metal tested at 700 °C [creep rates are in mm/mm/min].

Ageing temperature (°C)	Ageing time (h)	Weld metal		Parent metal		Stress exponent (n) values		
		$\hat{arepsilon}_1$ at 440 MPa	$\hat{\varepsilon}_2$ at 660 MPa	$\hat{arepsilon}_1$ at 440 MPa	$\hat{\varepsilon}_2$ at 660 MPa	Weld metal	Parent metal	
Unaged	-	0.74	11.80	0.82	9.73	6.8	6.1	
300	2000	0.71	10.91	0.80	9.43	6.7	6.1	
400	2000	0.71	9.31	0.78	9.43	6.4	6.1	
500	2000	0.72	21.70	0.77	9.38	8.4	6.2	



Fig. 4. (a) SEM photomicrograph showing cracks near the edge of indentation – weld metal sample aged for 2000 h at 500 °C and tested with 12 kg load at 700 °C. Polishing the edge of the indentation is not successful because of unevenness, sample un-etched. (b) SEM photomicrograph of crack developed over grain boundary in the vicinity of indentation for weld samples aged at 500 °C for 2000 h and tested at 700 °C with 12 kg load; void formation over the grain boundary on the crack path may be noted, sample not etched. (c) SEM photomicrograph of weld metal aged at 500 °C for 2000 h and tested at 500 °C for 2000 h and tested at 700 °C with 12 kg load; void formation over the grain boundary on the crack path may be noted.

4.3. Creep mechanisms

Two important characteristics are cited to be indicative of mechanism prevailing during creep process. They are: (i) activation energy (Q) and (ii) stress exponent values (n). The creep mechanism is expected to change very much with temperature of testing and load (stress) at which test is carried out.

It is found that Q values fall in the range of 9-14 kJ/mol for all categories of samples except for the ones aged at $500 \,^{\circ}$ C. These exceptional samples record Q values up to $16 \,\text{kJ/mol}$ when tested in the range of $500-700 \,^{\circ}$ C with the load of $12 \,\text{kg}$. Having gone through the literature, it is found that activation energy values for tensile creep is more than those for the indentation creep by a factor of

Table 6

Ferrite number values for 316L SS weld sample.

Ageing temperature (°C)	Ferrite number of weld sample
Unaged	3.4
300 °C	3.2
400 ° C	2.5
500°C	2.0

5–7. The Q values for tensile creep of stainless steel are quoted to be around 120 kJ/mol (Shi and Northwood, 1993). The values of activation energy determined in the present work are at a low level.

The discrepancy in the values of activation energy between tensile and indentation creep test is important. It should be noted that stress affected zone below the indenter can be visualized as a zone of non-hemispherical shape during primary stage of creep. The shape of the zone develops into a hemispherical one during secondary stage of creep. These aspects are illustrated in the modeling study carried out by the present investigation and reported elsewhere. The difference between tensile and indentation creep can be explained as follows: in the case of tensile creep test, the whole specimen (except shoulders) is effectively available for the creep process whereas in the case of indentation creep test only that material in the region of stress affected zone (which defines the activation volume) is available for the creep process. Even though activation volume is dependent on both the stress levels and temperature of testing, the mechanism of creep seems to be more sensitive to the temperature of testing. From the literature it is found that in the case of indentation of creep of low carbon steels, the mechanism corresponding to the activation energy of 5 kJ/mol is 'dislocation creep' and that corresponding to the activation energy of 10 kJ/mol is grain boundary sliding (Yun et al., 2000).

Stress exponent values are indicative of mechanism of creep as recorded in literature (Shibutani et al., 2006). It should be noted that there is no discrepancy in stress exponent values between tensile creep and indentation creep tests (Dieringa et al., 2005). Based on the information given in the literature (Nam et al., 1995), following mechanism can be proposed for indentation creep of AISI 316L stainless steel weld metal and parent metals:

(i) dislocation creep corresponding to n value of 6–7 and (ii) grain boundary sliding corresponding to n value of 8.4.

4.4. Metallographic evidence

SEM photomicrographs taken in the vicinity of indentation for weld metal sample aged for 2000 h at 500 °C and tested at 700 °C with the load of 12 kg are presented in Fig. 4(a) and (b).

The process of cracks getting initiated at the edge of the indentation and propagating to other regions over the grain boundary is clearly visible in the images (Fig. 4(a)). Apart from this, many voids/pores are seen to be present over the grain boundary in the vicinity of the indentation (Fig. 4(b)). The presence of voids over the grain boundary is not observed in far away regions from the edge of the indenter. This fact indicates that void formation over the grain boundary is the part of mechanism of grain boundary sliding and not because of long ageing process at 500 ° C. SEM photomicrograph presented in Fig. 4(c) shows the difference in the levels of the grains as if one has slid down with respect to the other. Further, these events in the vicinity of indentation are not observed in other categories of samples. The area adjoining the area of indentation for which the SEM photomicrograph is presented in Fig. 4(c), is almost near the centre of the specimen.

It should be noted that another category of sample which is similarly aged and tested with 12 kg load at 500 °C (not 700 °C) shows



Fig. 5. SEM photomicrograph of sample aged at 500 °C for 2000 h and tested with 12 kg load at 500 °C. Formation of voids in the vicinity of the indentation is seen, but no crack over the grain boundaries.

some indication of void formation, though not the crack formation in the vicinity of indentation. This is revealed in Fig. 5.

4.5. Grain boundary cavity and creep

Oval type cavities are seen on the grain boundaries in the vicinity of indentation for the weld sample aged at 500 °C for 2000 h and tested with 12 kg load at 700 °C (Fig. 4). Further, similarly aged samples tested with 12 kg load at 500 °C has shown a line of cavity flanked by 'fold-like' things in some places (Fig. 5). This type of grain boundary cavitation has attracted the attention of many investigators. Reed-Hill and Abbasehian have presented a comprehensive view about this subject. The study of nucleation and growth of cavity are two important aspects of the investigation. Zener (1948) is of the opinion that the cavity nucleation cannot occur unless the stress concentration exceeds the cohesive strength of the grain boundary. Any factor that strengthens the grain region relative to grain boundary tends to promote the grain boundary shear. Movement of dislocation is made more difficult in grain region by solid solution hardening and precipitation hardening. In the present investigation, both seem to have played their roles. Ageing for a long duration of time at high temperature levels could cause precipitation of second phase particles over the grain region. This strengthens the grain relative to grain boundary, albeit, the extent of the effect may be small.

Further, because of the homogenization process (both during ageing and testing), segregated alloying atoms like Mo migrate from grain boundary to grain region, further strengthening grain region relative to grain boundary. This makes the grain boundary relatively favourable for grain boundary shear, leading to nucleation of cavity. It is proposed that the pre-existing jog that interfere with the shear deformation along the boundaries are responsible for the nucleation of the cavities. Tang and Plumtree (1985) working on AISI 304 stainless steel determined the vacancy concentration in the grain region and at grain boundary at different temperature levels. They concluded that excess concentration of vacancies found near the grain boundary is due to localization of strain. They proposed that this super-saturation of vacancy produces a chemical stress, like the applied stress promoted nucleation of cavities on the grain boundary. However, non-uniform(random) nature of nucleation site on the grain boundary as is noted in this work (Fig. 4(b)) is not well understood. It was argued that grain boundary migration away from the stressed region alleviates necessary condition for the formation of cavities on the grain boundary. In the present investigation, it is noted that the parent metal tested at 700 °C with 12 kg load shows a tendency for grain coarsening. By this mechanism, grain boundary could escape the nucleation of pore and hence crack over the grain boundary. On the basis of diffusion theory, Ballufi and Seigle (1957) postulate that cavity grows by movement of vacancies from the boundary to cavity. Further, the growth of the pores can be prevented by plastic flow in the grains by upheavals and 'folds' in the grain regions. This is revealed in the SEM photomicrographs in Fig. 5 for the weld metal sample aged at 500 °C for 2000 h and tested with 12 kg at 500 °C. Here string of cavities with 'folds' is seen but not the crack over the grain boundary.

Yu et al. have given the mechanical analysis of indentation creep tests conducted on structural ASTM A-36 steel. Evidence of grain boundary sliding with micro-structural evidence after conducting indentation creep tests on 316L stainless steel is hardly reported in any of the available reports that cover creep aspects of this material. More investigations in this direction are needed to reveal finer aspects of creep deformation in this material.

5. Conclusions

- (i) Ageing process does not influence the creep behaviour of parent metal. In the case of weld metal, influence of ageing is perceptible when samples are tested with higher load of 12 kg.
- (ii) Mechanism of creep is 'dislocation creep' in almost all the cases except in one case in which 'grain boundary sliding' is the operative mechanism. The exceptional case is the one in which sample has been aged at 500 °C for 2000 h and tested with 12 kg load at 700 °C. Metallographic evidences are provided for grain boundary sliding.
- (iii) In all the cases of ageing and testing, weld metal is as good creep resistant or marginally better than the parent metal except for the cases in which grain boundary sliding is the mechanism of creep.

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