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Influence of Nitrogen Content on the Evolution of Creep Damage in 316 LN Stainless Steel

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Abstract

The influence of nitrogen content on the high temperature mechanical properties of 316LN stainless steel has been studied at nitrogen levels of 0.07, 0.11, 0.14 and 0.22 wt. %. These studies have shown that 316LN SS containing 0.14 wt. % nitrogen has optimum mechanical properties. Detailed creep studies were carried out on 316LN SS at 823 K, 873 K and 923 K at stress levels ranging from 140 to 300 MPa. The longest rupture life in this study was 32,500 hours. Creep strength was found to increase substantially with increase in nitrogen content. Metallographic studies were performed on creep tested specimens to understand the influence of nitrogen content on the evolution of creep damage. The extent of internal and surface creep damage decreased drastically with increase in nitrogen content. Area fraction of internal creep damage was measured and it was observed to decrease significantly with increase in nitrogen content. The steel containing the highest nitrogen content showed a negligible amount of creep damage irrespective of the stress level. Hence, increasing the nitrogen content increases the resistance to creep damage formation leading to improved rupture life. The extent of internal and surface creep damage decreased with increasing temperature and decreasing stress. Fracture mode was found to predominantly intergranular in the steel containing the higher nitrogen content.

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Keywords: 316 LN SS, nitrogen, creep rupture, creep damage

1. Introduction

Out-of-core structural components of sodium cooled fast reactors (SFRs) operating in the creep range are currently using 316L(N) stainless steel (SS) containing 0.02-0.03 wt% carbon and 0.06-0.08 wt% nitrogen [1]. Alloying with 0.06-0.08 wt% nitrogen helps to increase the high temperature strength of 316L(N) SS to levels comparable to that of 316 SS despite the low carbon content. Nitrogen is a strong austenite stabilizer, solid solution strengthener, and it improves pitting corrosion resistance. Nitrogen is known to improve creep and fatigue strength at high temperatures and fracture toughness at cryogenic temperatures in austenitic steels [2,3]. The influence of

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nitrogen content on the mechanical behavior of austenitic stainless steels has been reported extensively [2-6]. In order to increase the economic competitiveness of SFRs, there is a strong desire to increase the design life from the current level of 40 years to at least 60 years in the future designs. As part of the efforts to develop structural materials suitable for very long design life, the influence of nitrogen at concentrations higher than 0.08 wt. % on the high temperature mechanical properties of type 316L SS is being studied extensively[7-11]. Four heats of 316L SS, containing 0.07, 0.11, 0.14 and 0.22 wt% nitrogen (designated as 316LN SS) were produced to study the effect of nitrogen on the tensile and creep properties of 316 L SS. The carbon content in these heats was maintained at ~ 0.03 wt. % and the actual chemical composition of all other elements was similar (Table 1). Recent studies on the effect of nitrogen on the mechanical properties of 316LN SS have shown that the alloying element nitrogen increases the tensile, creep and low cycle fatigue properties of 316LN SS significantly [7-13]. These studies have shown that 316LN SS containing 0.14 wt. % nitrogen has optimum mechanical properties. Tensile properties such as yield strength, ultimate tensile strength, uniform elongation, total elongation and reduction in area are summarized in Table-2 for 316 LN SS containing 0.14 wt. % nitrogen.

Table 1. Chemical Composition of 316LN SS (wt. %).

Designation	N	C	Mn	Cr	Mo	Ni	Si	S	1. P	Fe	Grain size µm
7N	0.07	0.027	1.7	17.53	2.49	12.2	0.22	0.0055	0.013	Bal.	87± 9
11N	0.11	0.033	1.78	17.62	2.51	12.27	0.21	0.0055	0.015	Bal.	96± 8
14N	0.14	0.025	1.74	17.57	2.53	12.15	0.20	0.0041	0.017	Bal.	78± 8
22N	0.22	0.028	1.70	17.57	2.54	12.36	0.20	0.0055	0.018	Bal.	87± 11

Table-2. Tensile properties of 316LN SS containing 0.14 wt. % nitrogen.

Sample No.	Temperature K	Yield Strength MPa	Ultimate Tensile Strength MPa	Uniform Elongation %	Total Elongation %	Reduction in Area %
14N-1	300	306	612	36	41	81
14N-2	523	207	502	35	40	81
14N-3	623	197	498	35	39	80
14N-4	723	162	478	39	42	75
14N-5	823	149	455	34	37	71
14N-6	923	139	397	36	40	76
14N-7	1023	131	297	29	39	53
14N-8	1123	124	206	20	39	48

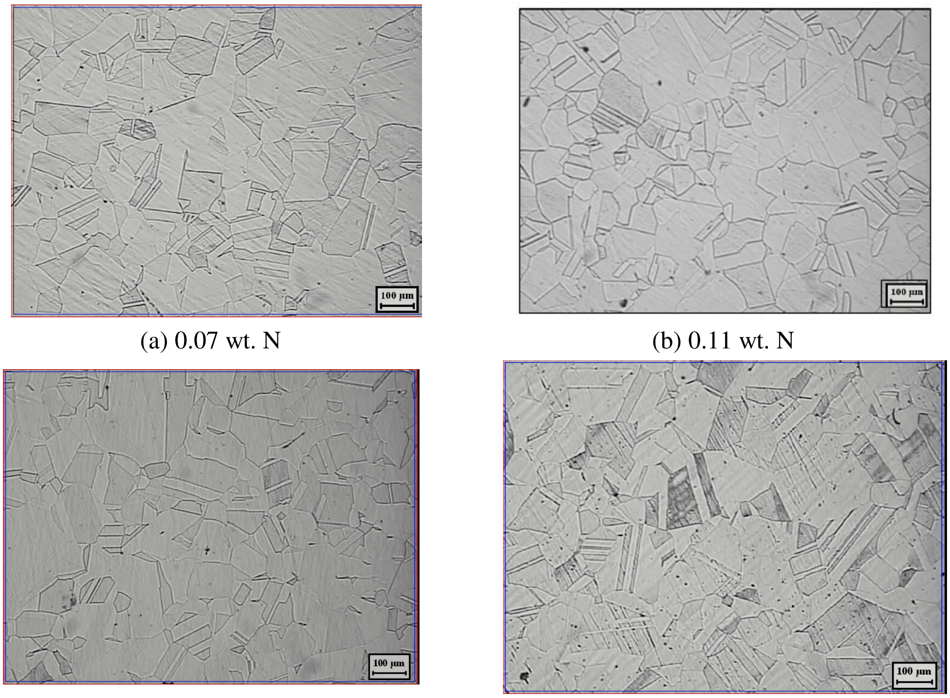
Detailed creep studies were carried out on 316LN SS at 823 K, 873 K and 923 K at stress levels ranging from 140 – 300 MPa. The longest rupture life in this study was 32,500 hours. The creep properties of four heats of this material have been reported previously [12]. Metallographic studies were performed on creep tested specimens to understand the influence of nitrogen content on the evolution of creep damage. This paper presents the metallographic studies carried out on the creep tested specimens.

2. Experimental

Constant load creep tests were conducted at 823 K, 873 K and 923 K at stress levels ranging from 140 to 300 MPa. A linear variable differential transformer (LVDT) was used for monitoring the specimen elongation. Elongation and temperature were continuously recorded using an automatic data logging system. All the creep tests were conducted in accordance with ASTM standard recommended practice E-139 and the test temperature was controlled within ± 2 K. Round specimens with 50 mm gauge length and 10 mm gauge diameter were used for the creep tests. Samples for optical metallograpy were prepared from longitudinal sections of fractured specimens and were observed under Leica optical microscope fitted with image analysis software. The samples were electrolytically etched using a solution of 10 gm oxalic acid and 100 ml water to reveal the microstructure. Image analysis software was used to estimate the area fraction of creep damage. Scanning Electron Microscope (SEM), Philips model PSEM 501 was used to study the fracture surfaces. For this purpose, the tip of fracture sample was cut and cleaned in acetone in ultrasonic cleaner before observing in SEM.

3. Results and Discussion

Figure 1 shows the typical microstructure of the materials in the solution treated condition. Equiaxed grains free from carbide precipitates were observed in all the heats. The grain size of the four heats were in the range of 78 to 96 µm. Vickers hardness of the material increased from 155 to 192 on increasing the nitrogen content from 0.07 to 0.22 wt.%. Creep strain-time curves at a stress level of 200 MPa and 140 MPa are shown in Fig. 2 for the four different heats. The creep curves of 316LN SS showed a small instantaneous strain on loading followed by typical primary, secondary and tertiary creep stages for all the nitrogen contents.



(a) 0.07 wt. N (b) 0.11 wt. N

(c) 0.14 wt. N (d) 0.22 wt. N

Fig. 1. Microstructure of 316 L(N) SS.

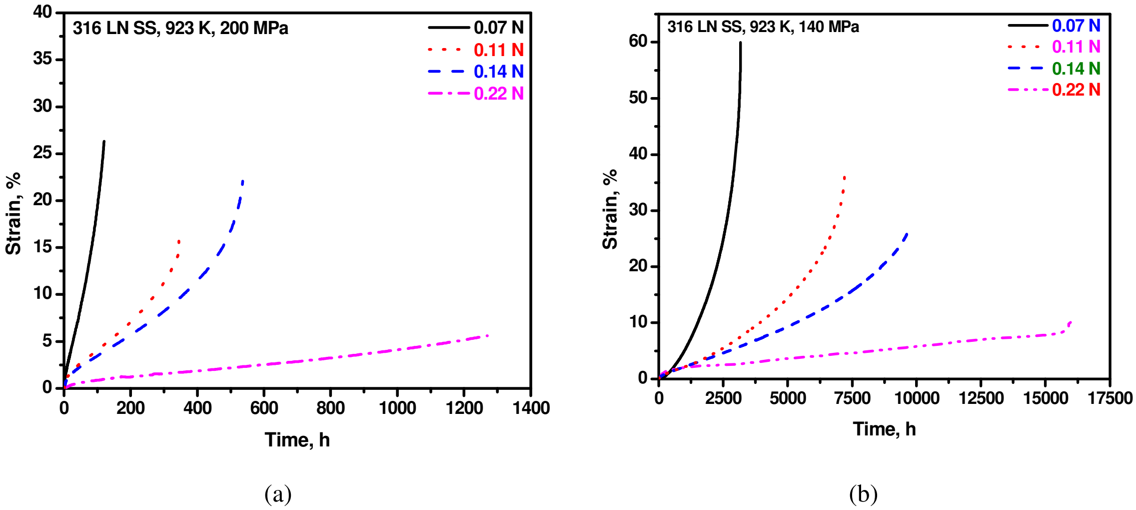


Fig. 2. Influence of nitrogen content on creep curve of 316 LN steel at (a) 200 MPa and (b) at 140 MPa

The creep properties of this material with various nitrogen content at 923 K have been reported earlier [12]. Nitrogen was found to be beneficial to creep properties at all the stress levels. Creep rupture strength increased substantially with increase in nitrogen content; rupture life increased almost 10 times by increasing nitrogen content from 0.07 wt. % to 0.22 wt. %. Steady state creep rate showed a decrease with increase in nitrogen content.

Metallographic studies were performed on creep tested specimens to understand the influence of nitrogen content on the extent of creep damage. Surface creep cracks and internal creep damage were observed in all the specimens. The cracks were generally oriented perpendicular to the loading direction. Similar observations were made in all the creep tested specimens. Figure 3 shows typical micrograph of surface of creep tested specimen at 923 K at 140 Mpa. Leica QWin image analysis software was used for calculating number of surface cracks, number of internal cracks and % area fraction of internal creep damage. These measurements were made for all the specimens up to a distance of 9 mm from the fracture surface.

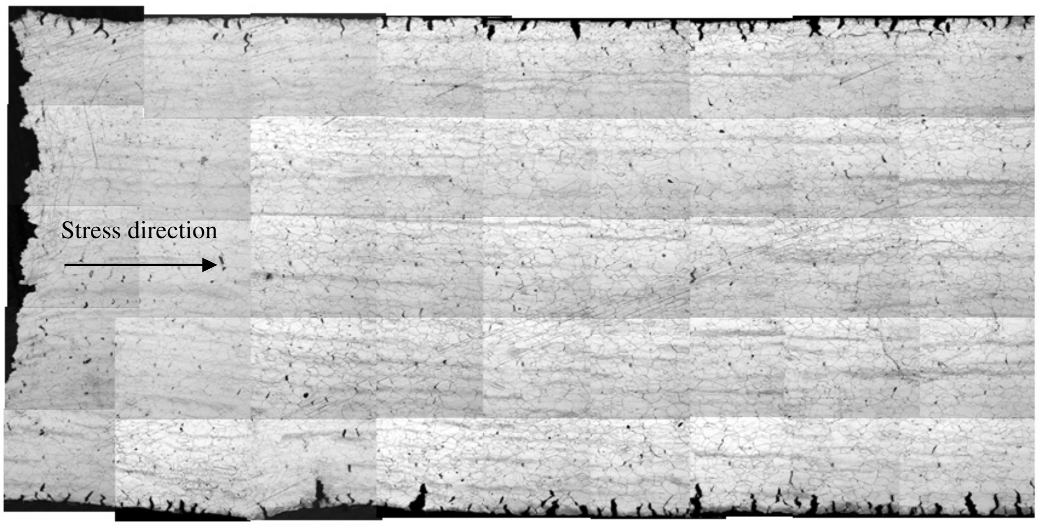
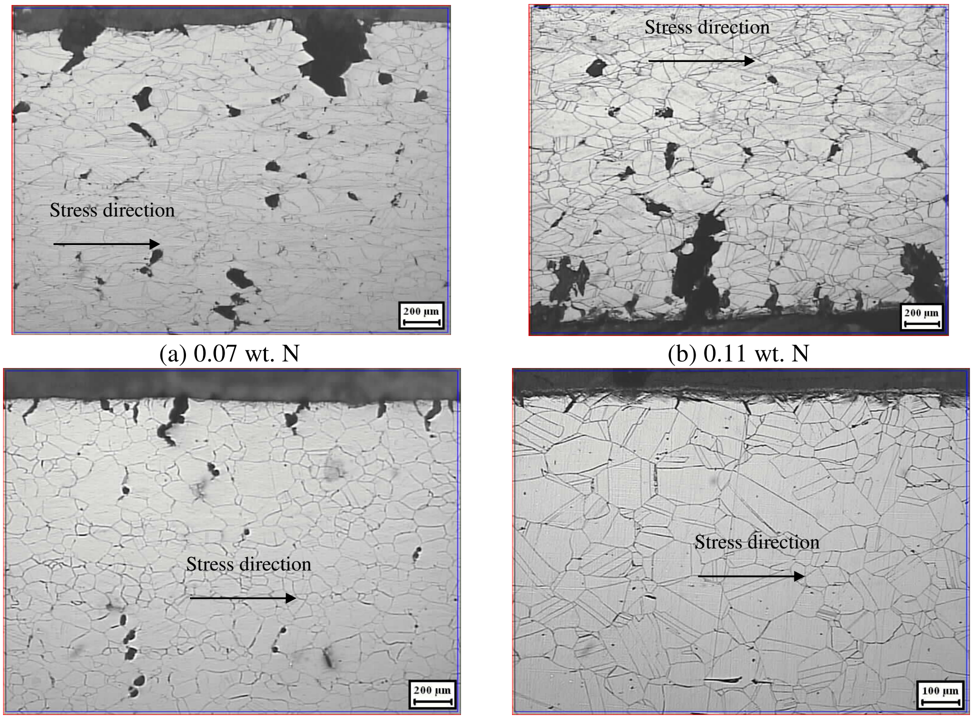


Fig.3. Typical micrograph of surface of creep tested specimen at 923 K at 140 Mpa



(a) 0.07 wt. N (b) 0.11 wt. N

(c) 0.14 wt. N (d) 0.22 wt. N

Fig. 4. Micrographs of creep tested specimens at 923 K and at 225 MPa

Figure 4 shows micrographs of creep tested specimens at 923 K at 225 MPa. The extent of internal and surface creep damage can be found to decrease drastically with increase in nitrogen content. The steel containing the highest nitrogen content showed a negligible amount of creep damage. Nitrogen is known to be a strong solid solution strengthening element in austenitic stainless steels. The major creep strengthening mechanism of nitrogen is by way of decreased stacking fault energy and thereby increasing slip planarity [12]. Nitrogen interacts with solutes to form complexes [14] and short range ordered precipitates [15], which contribute to strengthening. At high temperatures, nitrogen helps to refine the carbide precipitates on grain boundaries thereby retarding grain boundary embrittlement and formation of creep damage [16]. Hence, increasing the nitrogen content increases the resistance to creep damage formation leading to improved rupture life and rupture ductility.

The % area fraction of internal creep damage was measured and plotted against the rupture life. Figure 5 shows the variation of internal creep damage with rupture life at various nitrogen contents. At lower rupture level, because of high rupture ductility the % area fraction of internal creep damage is low. As the applied stress decreases, rupture life is longer, grain boundary sliding process becomes significant, and this leads to formation of sharp triple point cracks and consequent high % area fraction of internal creep damage. At still lower applied stresses, creep time is very long and during this period, grain boundary carbide precipitation takes place which strengthens the grain boundary, and retards grain boundary sliding. The amount of grain boundary damage formed under these conditions is low. Figure 6 shows the variation of internal creep damage with rupture life at various temperatures for the steel containing 0.14 wt. % nitrogen. Internal creep damage decreased with increasing rupture life and increasing temperature.

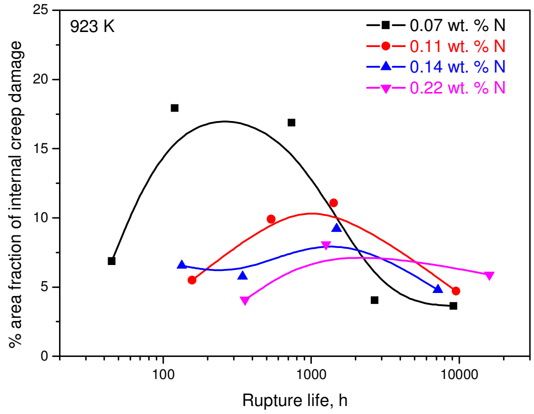


Fig. 5. Variation of internal creep damage with rupture life at various nitrogen contents.

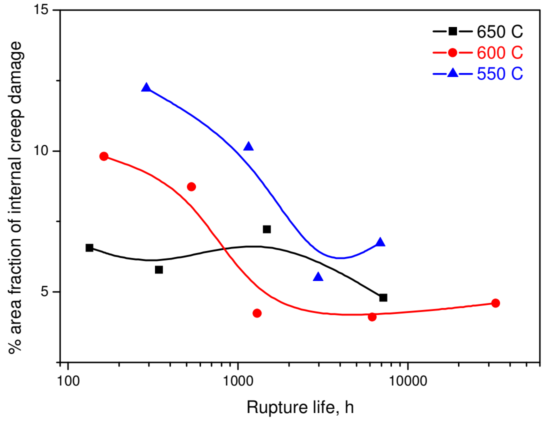


Fig. 6. Variation of internal creep damage with rupture life at various temperature for the steel containing 0.14 wt.% nitrogen.

Figure 7 shows the variation of internal creep damage with rupture life at various nitrogen contents. The number of cracks per unit area was not changed up to 1000 hours and beyond that it increased with rupture time.

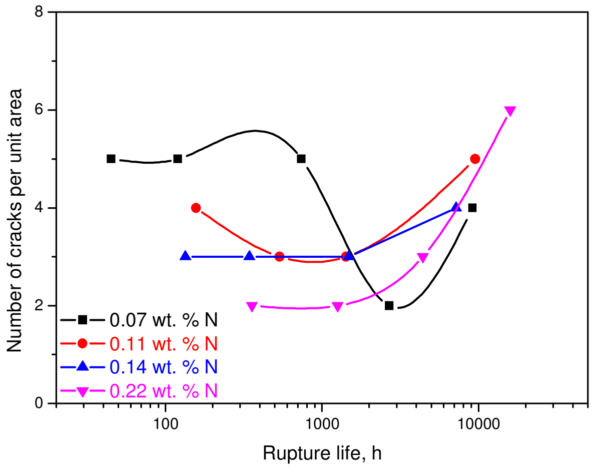
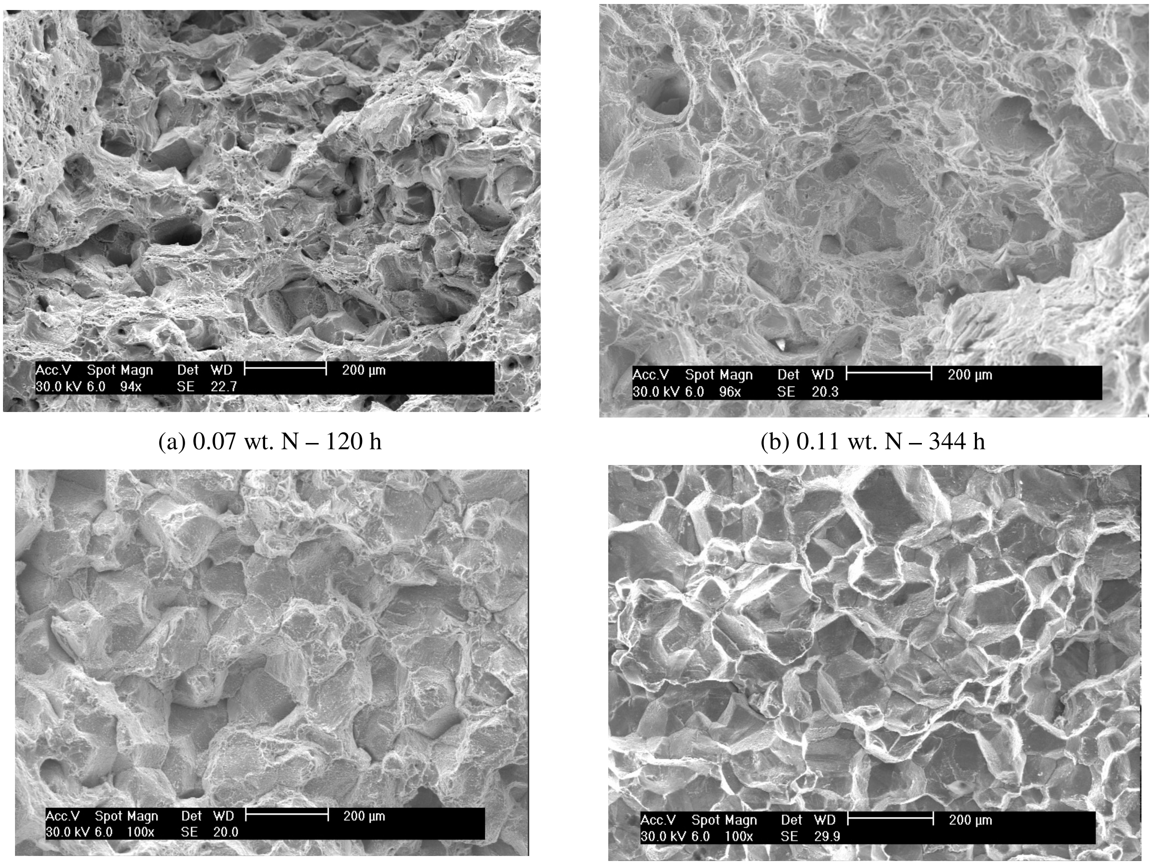


Fig. 7. Variation of number cracks/unit length with rupture life at various nitrogen contents.

Scanning electron microscopic examinations were carried out on creep tested specimens. Figure 8 shows the fracture surfaces of the specimens tested at 200 MPa which corresponds to a ductility minimum. Fracture mode was found to be predominantly intergranular. The fracture in 316 LN SS containing 0.22 wt., % nitrogen appears to be a case of pure intergranular failure.



(a) 0.07 wt. N – 120 h (b) 0.11 wt. N – 344 h

(c) 0.14 wt. N – 537 h (d) 0.22 wt. N – 1262 h

Fig. 8. SEM micrographs showing fracture surface after creep testing at 200 MPa

4. Conclusions

Metallographic studies on creep tested 316LN stainless steel with various nitrogen levels showed that

- The extent of internal and surface creep damage decreased drastically with increase in nitrogen content.
- Area fraction of internal creep damage was measured and it was observed to decrease significantly with increase in nitrogen content.
- The steel containing the highest nitrogen content showed a negligible amount of creep damage irrespective of the stress level.
- The extent of internal and surface creep damage decreased with increasing temperature and decreasing stress.
- Fracture mode was found to predominantly intergranular in the steel containing the higher nitrogen content.

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References

1. S. L. Mannan, S. C. Chetal, Baldev Raj and S. B. Bhoje, Trans. IIM, 56 (2003) 155-178.
2. Valentine G Gavriljuk and Hans Berns, 'High Nitrogen Steels', 135; 1999, New York, Springer-Verlag Berlin Heidelberg.
3. J.R.Kearns in R.A.Lula, 'Proceedings of Conference on New Developments in Stainless Steel Technology', 117; 1985, ASM, Metals Park, OH.
4. Simmons, J.W., Mater. Sci. and Engg. A, 207(1996) 159-169.
5. Valentine G Gavriljuk and Hans Berns, 'High Nitrogen Steels', 135; 1999, New York, Springer-Verlag Berlin Heidelberg.
6. M. D. Mathew and V. S. Srinivasan: in 'Monograph on high nitrogen austenitic steels and stainless steels', (ed. U. K. Mudali and B. Raj), 182–204; 2004, New Delhi, Narosa Publications.
7. V. Ganesan, M.D. Mathew and K. Bhanu Sankara Rao, Mat. Sci. and Tech., 25(2009) 614-618.
8. V. Ganesan, M.D. Mathew and K. Bhanu Sankara Rao, Trans. IIM, 63(2010) 417-421.
9. G.V. Prasad Reddy, R. Sandhya, M. Valsan, K. Bhanu Sankara Rao and S. Sankaran, Trans. IIM, 63(2010) 505-510.
10. G. V. Prasad Reddy, R. Sandhya, K. Bhanu Sankara Rao and S. Sankaran, Procedia Engg. 2 (2010) 2181-2188.
11. J. Ganesh kumar, M. Chowdary, V. Ganesan, R.K. Paretkar, K. Bhanu Sankara Rao and M.D. Mathew, Nucl. Engg. Design, 240(2010) 1363-1370.
12. M. D. Mathew, K. Laha and V. Ganesan, Mater. Sci. Eng. A 535 (2012) 76– 83.
13. G.V. Prasad Reddy, R. Sandhya, M.D. Mathew and S. Sankaran, Advanced Materials Research, 794 (2013) 441-448.
14. K.Oda, N.Kondo and K.Shibata, *ISIJ Intl*., 1990 (30) p. 625.
15. M.L.G.Byrnes, M.Grujici and W.S.Owen, *Acta Metall*., 35 (1987) p. 1853.
16. M.D.Mathew, G.Sasikala, K.B.S.Rao and S.L.Mannan, Materials Science and Engineering, A148 (1991) p. 253-60.