



Time–temperature-sensitization diagrams and critical cooling rates of different nitrogen containing austenitic stainless steels

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ARTICLE INFO

Article history:

Received 29 July 2009

Accepted 1 January 2010

Keywords:

Austenitic stainless steel

Sensitization

TTS diagrams

Critical cooling rates

ABSTRACT

Nitrogen-alloyed 316L stainless steel is being used as structural material for high temperature fast breeder reactor components with a design life of 40 years. With a view to increase the design life to 60 years and beyond, high nitrogen stainless steels are being considered for certain critical components which may be used at high temperatures. Since carbon and nitrogen have major influence on the sensitization kinetics, investigations were carried out to establish the sensitization behaviour of four heats of 316L SS containing (i) 0.07%N and 0.035%C, (ii) 0.120%N and 0.030%C, (iii) 0.150%N and 0.025%C and (iv) 0.22%N and 0.035%C. These stainless steels were subjected to heat treatments in the temperature range of 823–1023 K for various durations ranging from 1 h to 500 h. Using ASTM standard A262 Practice A and E tests, time–temperature-sensitization diagrams were constructed and from these diagrams, critical cooling rate above which there is no risk of sensitization was calculated. The data established in this work can be used to select optimum heat treatment parameters during heat treatments of fabricated components for fast reactors.

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1. Introduction

It is well known that when austenitic stainless steels are extensively heated or slowly cooled in the temperature range of 773–1123 K, chromium rich $M_{23}C_6$ carbides precipitate in the vicinity of the grain boundaries with the concomitant chromium depletion along the grain boundaries. When chromium level near the grain boundaries falls below 12%, the material is said to be ‘sensitized’. When the sensitized material is exposed to corrosive media, intergranular corrosion takes place. Fabricated components are often exposed to the sensitization range during solution annealing, stress-relieving or dimensional stabilization heat treatments. During these heat treatments if cooling rates slower than the critical cooling rates are used, the component may get sensitized and become prone to intergranular corrosion and intergranular stress corrosion cracking. Although sensitization and intergranular corrosion are well understood, such failures still occur when the effect of sensitization is not taken into account during fabrication, pre-commissioning stages and service period [1–4]. Several components fail in service due to sensitization because of improper heat treatment or improper welding conditions.

Type 316L(N) stainless steel is the structural material in the primary side of 500 MWe Prototype Fast Breeder Reactor which is being built at Kalpakkam, Tamilnadu, India. In order to avoid sensitization at the heat affected zone during welding and to avoid the risk of chloride stress corrosion cracking during storage of fabricated components in coastal site of Kalpakkam, low carbon (0.03%) stainless steels are used. Since low carbon grades have lower strength than normal grades, nitrogen is specified as an alloying element to improve the mechanical properties so that the strength is comparable to 316 SS. The authors have studied the effect of nitrogen through establishing time–temperature-sensitization (TTS) diagrams and continuous cooling sensitization (CCS) diagrams for type 316 SS with various weight % of carbon and nitrogen [5,6]. From these studies it can be inferred that as nitrogen content increases, the time required for sensitization at nose temperature increases. The authors have reported that the critical cooling rate above which there is no risk of sensitization is 0.43 K/h for 316LN SS containing 0.03% carbon and 0.089% nitrogen. Therefore, the cooling rates encountered during welding will not result in sensitization at the heat affected zone. Nitrogen retards both formation and coarsening rates of $M_{23}C_6$ because it reduces the diffusivity of chromium and carbon. In addition to that it improves passivity [7–12] and hence greater extent of chromium depletion can be tolerated without the risk of sensitization. More recent investigations indicate that nitrogen actually enhances the diffu-

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sion of substitutional elements by increasing the formation of vacancies but delays the nucleation of $M_{23}C_6$, the structure of which is destabilized when carbon is partially substituted by nitrogen [13].

High-nitrogen austenitic stainless steels are being considered for the next generation fast reactors. For certain critical high temperature components requiring high strength, nitrogen alloying in the range of 0.11–0.22% is being proposed. These high-nitrogen austenitic stainless steels are more favoured compared to the conventional alloys because of their high yield and tensile strength and ductility, high strength–fracture toughness combination, high strain hardening potential, resistance to deformation-induced martensite formation, low magnetic permeability, improved wear, cavitation and erosion resistance, sensitization and localized corrosion resistance. The excellent mechanical and corrosion properties of high nitrogen SS may allow them to be substituted for more expensive materials such as nickel based alloys or super alloys for certain applications. The beneficial effects of nitrogen are appreciable only if the solubility limit of the nitrogen is not exceeded. Compared to carbon, nitrogen is less effective in causing sensitization. Extent of chromium depletion caused by 0.08% C is more than that caused by 0.2% nitrogen [14]. This implies that nitrogen is not as detrimental as carbon even when the solubility limit is exceeded.

However, in high nitrogen stainless steels, extensive ageing at elevated temperature leads to the precipitation of Cr_2N . Although Cr_2N leads to less Cr depletion compared to $Cr_{23}C_6$, this also contributes to increase in DOS. Controversial opinions exist among various authors on the threshold nitrogen % up to which the beneficial effect with respect to sensitization is observed [15–19]. In other words, in austenitic stainless steels, the effect of nitrogen on the sensitization process is not clearly understood. It has been reported that nitrogen enlarges the Cr_2N precipitation zone and increase the sensitivity to IGC in low carbon (0.01%) steels containing nitrogen in the range of (0.02–0.54%) [17]. Another study of the sensitization behaviour of AISI 304 SS containing 0.045% C and nitrogen in range of 0.04–0.25% reveals that nitrogen up to 0.16% has a retarding effect on the growth of carbides [15,17]. In 316 SS, it has been reported that steels with carbon content greater than 0.02% and nitrogen greater than 0.13% are susceptible to both intergranular stress corrosion cracking (IGSCC) and intergranular corrosion (IGC) due to the precipitation of carbides and nitride, respectively at grain boundaries [18]. In a study of IGSCC behaviour of 18–10 SS containing up to 0.22% nitrogen in (i) O_2 and (ii) O_2/Cl^- containing water under boiling water condition, nitrogen alloyed stainless steels was found to be less susceptible [17].

In order to use these recently developed high nitrogen stainless steels in service without the risk of failure, complete understanding of the microstructural stability and corrosion resistance is essential. Since nitrogen addition also leads to sensitization, its influence on kinetics of sensitization has to be established. In this paper, the sensitization behaviour of four heats of 316L SS containing (i) 0.07% N and 0.035% C, (ii) 0.12% N and 0.030% C, (iii) 0.15% N and 0.025% C (iv) 0.22% N and 0.035% C was established using ASTM standard A262 Practice A and E tests [20]. time–temperature–sensitization (TTS) diagrams were established to understand isothermal sensitization behaviour. From the TTS diagrams, critical cooling rate above which there is no risk of sensitization was established using a mathematical method developed by one of the authors [21].

2. Experimental

316L SS with four different nitrogen contents were used in these studies. The chemical composition and heat number are pre-

Table 1
Chemical composition.

Elements	Weight (%)			
	Plate 1 (heat no.: 8334)	Plate 2 (heat no.: 8344)	Plate 3 (heat no.: 8345)	Plate 4 (heat no.: 8335)
N	0.15	0.07	0.22	0.12
C	0.025	0.035	0.035	0.03
Cr	17.5	17.5	18.5	17.5
Ni	12.5	12.5	12.5	12
Mo	2.35	2.4	2.4	2.3
Mn	1.5	1.45	1.4	1.5
Si	0.26	0.3	0.26	0.3
S	<0.005	<0.005	<0.005	<0.005
P	0.015	0.015	0.015	0.015
Co	0.05	0.04	0.048	0.04
Cu	0.08	0.053	0.08	0.06
Ti	<0.08	<0.08	<0.08	<0.08
Nb	<0.07	<0.07	<0.07	<0.07

Table 2

Results obtained in ASTM A262 Practice A test and E test for plate 1 (heat no.: 8334 – 0.15%N, 0.025%C).

Heat treatment	Results obtained in	
	Practice A	Practice E
As-received	Step	No attack
1023 K – 16 h-AC	Step	No attack
1023 K – 24 h-AC	Step	No attack
973 K – 10 h-AC	Step	No attack
973 K – 16 h-AC	Ditch	Attack
973 K – 24 h-AC	Ditch	Attack
973 K – 40 h-AC	Ditch	Attack
948 K – 8 h-AC	Step	No attack
948 K – 10 h-AC	Step	No attack
948 K – 16 h-AC	Ditch	Light attack
923 K – 10 h-AC	Step	No attack
923 K – 16 h-AC	Ditch	Attack
898 K – 16 h-AC	Step	No attack
898 K – 24 h-AC	Ditch	Attack
873 K – 24 h-AC	Step	No attack
873 K – 40 h-AC	Ditch	Attack
823 K – 100 h-AC	Step	No attack
823 K – 240 h-AC	Step	No attack
823 K – 500 h-AC	Ditch	Attack

sented in Table 1. The as received materials were in the mill-annealed condition. From all the plates, 100 mm × 10 mm × *t* mm (thickness) specimens were cut. The details of the heat treatments carried out are presented in Tables 2–5. From each heat treated specimen, 10 mm × 10 mm × *t* mm (thickness) specimen was cut for ASTM A262 Practice A test and rest of the specimen was kept for ASTM A262 Practice E test. For ASTM A262 Practice A test, specimens were mounted in epoxy resin (Araldite) and the mounted specimens were polished up to fine diamond (1 μm) finish. For ASTM A262 Practice E test, the specimens were polished up to 400 grit. As-received specimen as well as all the heat treated specimens were etched in 10 wt.% ammonium persulphate solution at a current density of 1 A/cm² for 5 min according to ASTM standard A262 Practice A test. The etched structures are classified as step (absence of chromium carbides), dual (discontinuous carbide precipitation – no single grain completely surrounded by carbides), ditch (one or more grains completely surrounded by carbides).

As received specimen as well as all the heat treated specimens were subjected to ASTM standard A262 Practice E test, to determine whether the specimens were sensitized or not. Practice E test consisted of exposing the specimens embedded in copper turnings in boiling 10% $CuSO_4$ + 10% H_2SO_4 solutions for 24 h and then subjecting them to 180° bend test. The specimens were bent through

Table 3

Results obtained in ASTM A262 Practice A test and E test for plate 2 (heat no.: 8344 – 0.07%N, 0.035%C).

Heat treatment	Results obtained in	
	Practice A	Practice E
As-received	Step	No attack
1023 K – 16 h-AC	Step	No attack
1023 K – 24 h-AC	Step	No attack
973 K – 10 h-AC	Step	No attack
973 K – 16 h-AC	Ditch	Attack
973 K – 24 h-AC	Ditch	Attack
973 K – 40 h-AC	Ditch	Attack
948 K – 6 h-AC	Step	No attack
948 K – 8 h-AC	Ditch	Attack
948 K – 10 h-AC	Ditch	Light attack
948 K – 16 h-AC	Ditch	Light attack
923 K – 10 h-AC	Step	No attack
923 K – 16 h-AC	Ditch	Attack
898 K – 16 h-AC	Step	No attack
898 K – 24 h-AC	Ditch	Attack
873 K – 24 h-AC	Step	No attack
873 K – 40 h-AC	Step	No attack
873 K – 50 h-AC	Ditch	Attack
823 K – 100 h-AC	Step	No attack
823 K – 240 h-AC	Step	No attack
823 K – 500 h-AC	Ditch	Attack

Table 4

Results obtained in ASTM A262 Practice A test and E test for plate 3 (heat no.: 8345 – 0.22%N, 0.035%C).

Heat treatment	Results obtained in	
	Practice A	Practice E
As-received	Step	No attack
1023 K – 1 h-AC	Dual	No attack
1023 K – 3 h-AC	Dual	No attack
1023 K – 6 h-AC	Ditch	No attack
1023 K – 10 h-AC	Ditch	No attack
1023 K – 24 h-AC	Ditch	No attack
973 K – 1 h-AC	Step	No attack
973 K – 3 h-AC	Dual	No attack
973 K – 6 h-AC	Ditch	No attack
973 K – 10 h-AC	Ditch	Light attack
973 K – 24 h-AC	Ditch	Attack
923 K – 7 h-AC	Dual	No attack
923 K – 10 h-AC	Dual	Light attack
923 K – 16 h-AC	Dual	Attack
923 K – 50 h-AC	Ditch	Attack
873 K – 24 h-AC	Dual	No attack
873 K – 70 h-AC	Ditch	Attack

Table 5

Results obtained in ASTM A262 Practice A test and E test for plate 4 (heat no.: 8335 – 0.12%N, 0.030%C).

Heat treatment	Results obtained in	
	Practice A	Practice E
As-received	Step	No attack
1023 K – 10 h-AC	Dual	No attack
1023 K – 24 h-AC	Ditch	No attack
973 K – 7 h-AC	Dual	No attack
973 K – 10 h-AC	Dual	Very light attack
973 K – 24 h-AC	Ditch	Attack
923 K – 10 h-AC	Dual	No attack
923 K – 16 h-AC	Dual	No attack
923 K – 24 h-AC	Dual	No attack
923 K – 50 h-AC	Ditch	Attack
873 K – 24 h-AC	Dual	No attack
873 K – 70 h-AC	Ditch	Attack

180° and over a diameter equal to the thickness of the specimen being bent. The bent specimens were examined under low magnification (20×). The appearance of cracks or fissures on the bend re-

gion indicates susceptibility to IGC. As per ASTM A262-02a Practice E test (Section 29.1.3), specimens must be exposed to a minimum period of 15 h. Longer time exposure increases the sensitivity of the results. Since the authors have reported [5,6,22–30] sensitization data for various austenitic stainless steels using the previous version of this standard (ASTM A262-85 Practice E: 24 h exposure), for the current materials also same exposure time was used for comparison purpose.

3. Results

3.1. ASTM standard A262 Practice A test

Optical microscopic examination was carried out for mill annealed as well as heat treated specimens which were etched as per ASTM standard A262 Practice A test. All the four plates in as received (mill-annealed) condition showed ‘step’ structure (Fig. 1a–d) indicating the absence of secondary phases like carbides. Examination of the various heat treated specimens indicated that at each ageing temperature, as the ageing time increased, the microstructure changes from “step” to “ditch” structure as expected (Fig. 2a–d Tables 2–5). When all the 4 plates were subjected to identical heat treatments, not much difference was observed in their respective optical micrographs, as indicated in Fig. 3a–d.

3.2. ASTM standard A262 Practice E test

The results obtained in ASTM A262 Practice E test for plate 1 (heat no.: 8334 – 0.025%C, 0.15%N) and for plate 2 (heat no.: 8344 – 0.035%C, 0.07%N), plate 3 (heat no.: 8345 – 0.035%C, 0.22%N), plate 4 (heat no.: 8335 – 0.030%C, 0.12%N) are collectively presented in Tables 2–5, respectively.

3.2.1. Construction of TTS diagram

These diagrams were obtained by plotting IGC test results on a temperature vs. log soaking time axis and drawing a line which demarcates the sensitized and non sensitized region. Figs. 4–7 represent the TTS diagrams for plate 1 (heat no.: 8334 – 0.025%C, 0.15%N) and for plate 2 (heat no.: 8344 – 0.035%C, 0.07%N), plate 3 (heat no.: 8345 – 0.035%C, 0.22%N), plate 4 (heat no.: 8335 – 0.030%C, 0.12%N), respectively. These diagrams can be used for selecting optimum heat treatment parameters under isothermal conditions.

3.2.2. Method to calculate critical cooling rate (CCR)

Sensitization may also result from cooling through the sensitization temperature range. The sensitization that results from continuous cooling is of great practical importance since it is this type of thermal exposure that occurs in slow cooling after high temperature annealing or in the cooling of a weld or weld heat affected zone. TTS diagrams cannot be used directly to determine the extent of sensitization that can occur when the material is continuously cooled. The intersection of a superimposed cooling curve with the isothermal TTS diagram will not indicate whether the steel is sensitized or not, because it does not take into account the effect of time spent in the different temperature regions. Dayal and Gnanamoorthy have reported a method to predict the extent of sensitization during continuous cooling/heating of the material [21]. The cooling curve is divided into small segments (ΔT) from the highest temperature (T_H) to lowest temperature (T_L) of the relevant TTS diagram. The time of transit Δt is determined for each segment of the cooling curve and is divided by the sensitization time t_s at the mean temperature T of this segment from the TTS diagram. The cumulative fraction (α) of the resident time in successive segments from T_H to T_L is calculated and is defined as

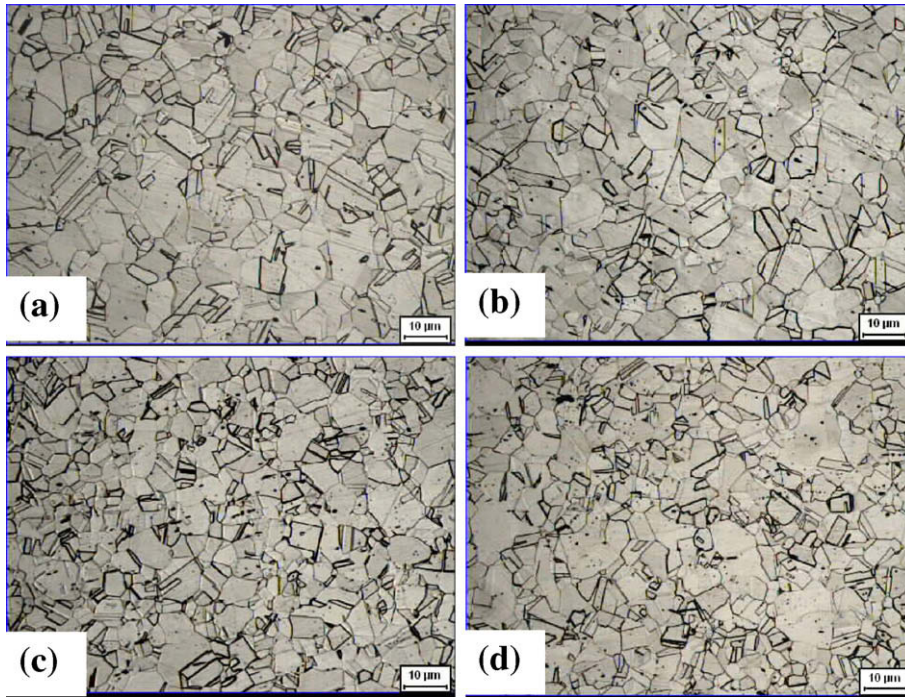


Fig. 1. Optical micrographs obtained in ASTM A262 Practice A test for (a) plate 1, (b) plate 2, (c) plate 3 and (d) plate 4 in the as-received (mill-annealed) condition.

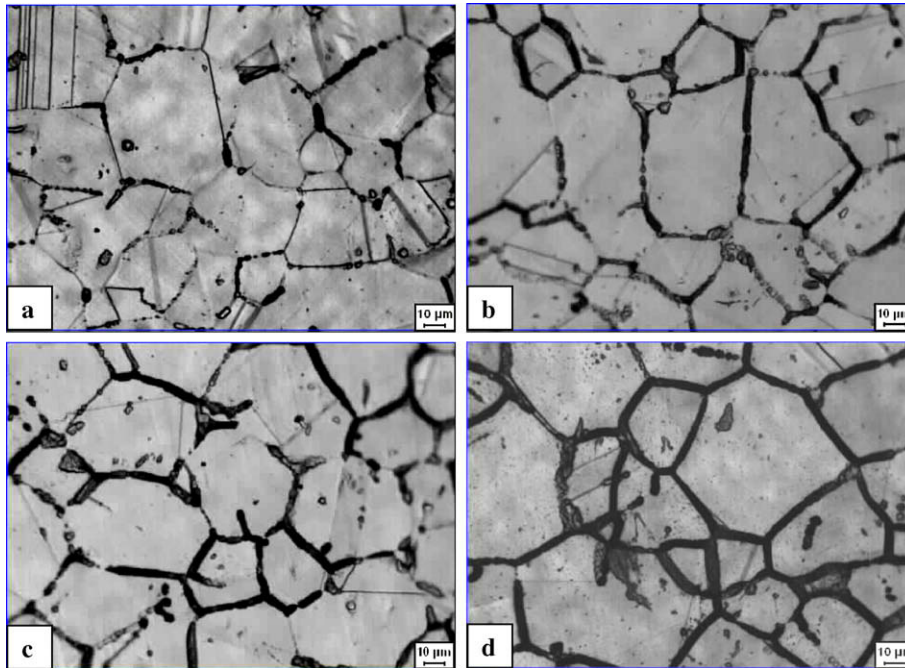


Fig. 2. Optical micrographs obtained in ASTM A262 Practice A test for plate 3 (heat no.: 8345) (a) 923 K – 7 h; (b) 923 K – 10 h; (c) 923 K – 16 h and (d) 923 K – 50 h.

$$\alpha = \sum_{T_L}^{T_H} \Delta t / t_s$$

Sensitization takes place only when $\alpha \geq 1$.

With the help of this equation, a critical linear cooling rate (CCR) to cause sensitization is calculated and is given by the equation

$$R = \Delta T \sum_{T_L}^{T_H} 1/t_s$$

where ΔT is the temperature difference in the small temperature steps considered.

Using this method, Dayal et al. have calculated the critical linear cooling rate for a type 316 SS, in annealed condition, as 234 K/h and they have verified the validity of these results experimentally. They have also shown that the value of α is not dependent on the magnitude of ΔT and hence no great advantage can be obtained by taking very small ΔT values [21]. In the present work, the time required for sensitization

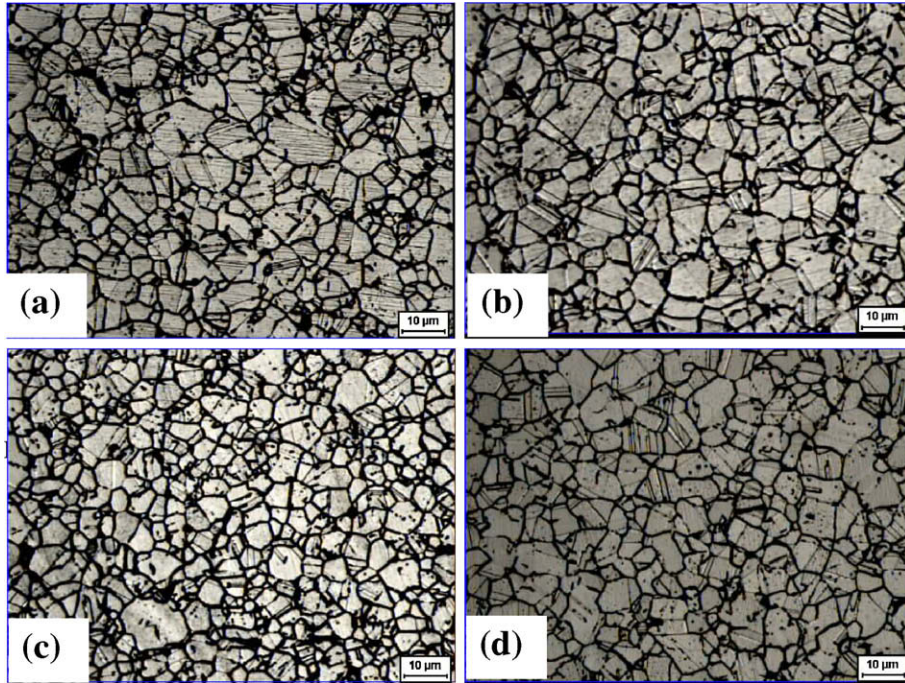


Fig. 3. Optical micrographs obtained in ASTM A262 Practice A test for (a) plate 1, (b) plate 2, (c) plate 3 and (d) plate 4 in the heat treated (973 K – 24 h) condition.

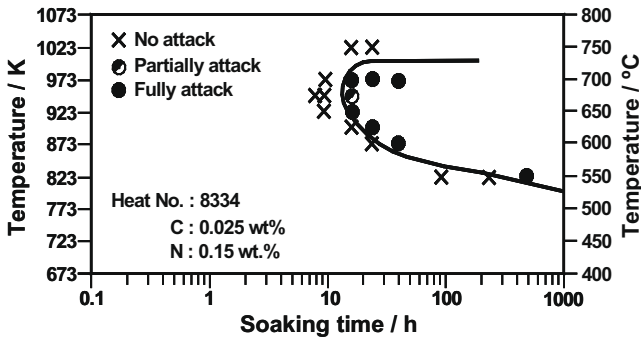


Fig. 4. Time-temperature-sensitization diagram for plate 1 (heat no.: 8334) established as per ASTM standard A262 Practice E test.

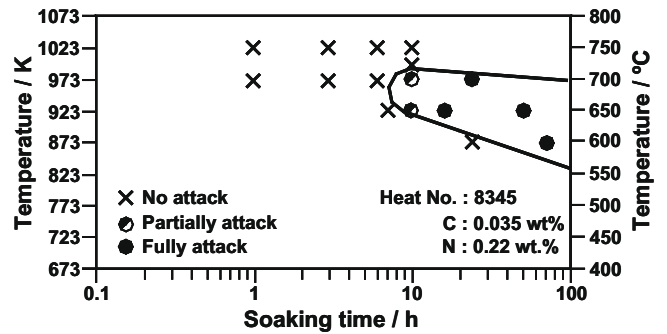


Fig. 6. Time-temperature-sensitization diagram for plate 3 (heat no.: 8345) established as per ASTM standard A262 Practice E test.

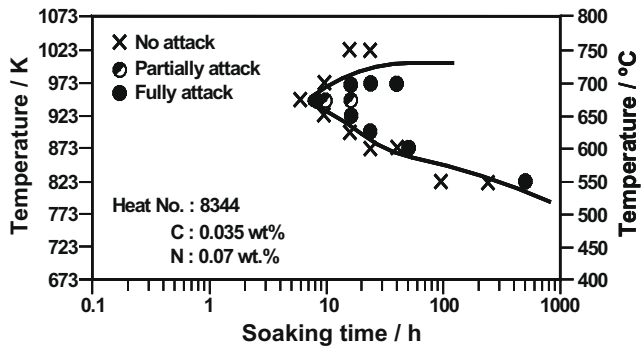


Fig. 5. Time-temperature-sensitization diagram for plate 2 (heat no.: 8344) established as per ASTM standard A262 Practice E test.

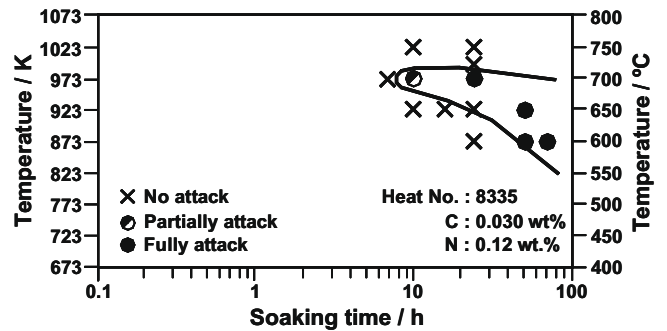


Fig. 7. Time-temperature-sensitization diagram for plate 4 (heat no.: 8335) established as per ASTM standard A262 Practice E test.

4. Discussion

(t_s) at each temperature was measured from the TTS diagram and critical cooling rates were calculated and are collectively presented in Table 6.

From the TTS diagrams (Figs. 4–7), it can be seen that for all the four stainless steels, the sensitization temperature range is

Table 6
Critical cooling rates.

Heat no.	Plate no.	Composition (wt.%)	CCR (K/h)
8345	3	0.22%N; 0.035%C	14
8344	2	0.07%N; 0.035%C	11
8334	1	0.15%N; 0.025%C	9
8335	4	0.12%N; 0.03%C	9

between 823 K and 973 K. The nose temperature (temperature at which time required for sensitization is minimum) is about 923 K. Among the four plates investigated, plates 2 and 3 have almost identical chemical composition but nitrogen content was 0.07% in plate 2 and 0.22% in plate 3. The plate containing higher nitrogen showed higher susceptibility to sensitization and IGC. For example, 10 h exposure at 923 K and 973 K resulted in sensitization in plate 3 containing 0.22% nitrogen whereas plate 2 containing 0.07% nitrogen is free from sensitization. This can be attributed to the fact that the beneficial effects of nitrogen are appreciable only if the solubility limit of the nitrogen is not exceeded. Nitrogen gets precipitated as Cr_2N when solubility limit is exceeded. However compared to carbon, nitrogen is less effective in causing sensitization and the extent of chromium depletion caused by 0.08%C is more than that caused by 0.2% nitrogen.

The CCR for these nitrogen containing stainless steels varies between 9 and 14 K/h. Carbon and nitrogen are the most important compositional variables influencing the kinetics of sensitization. If the kinetics of carbide precipitation is faster, the material is said to be more susceptible to sensitization and hence faster cooling rates have to be adapted to avoid sensitization that may result during continuous cooling. In other words, higher the value of CCR, more is the susceptibility of the material to sensitization and intergranular corrosion. It can be seen from Table 6, the CCR of plate 3 is the highest. When the carbon content decreases, then the CCR also decreases. However, from industrial point of view, it can be concluded that there is not much difference in the CCR between the four plates. Comparing plates 2 and 3 containing identical chemical composition except nitrogen content, our results indicate when nitrogen content is 0.22 wt.% (plate 3 – 8345), the CCR is 14 K/h, whereas the CCR is 11 K/h when the nitrogen content is lower (0.07 wt.%). This agrees very well with the previous results reported in literature where above 0.16 wt.%, nitrogen addition is detrimental from sensitization point of view [15,18].

The critical cooling rates of plate 1 (H8334 – 0.025%C, 0.15%N) and plate 4 (H8335 – 0.03%C, 0.12%N) were 9 K/h which is exactly same. In these two plates, the nitrogen content is below 0.16 wt.% and nitrogen remains in solid solution and renders the solid solution strengthening as well as retards sensitization kinetics. In plate 1, reduction in carbon content (0.025%) is compensated by addition of 0.15 wt.% nitrogen, where as plate 4 contain higher carbon (0.03%) and lower nitrogen (0.12%) renders the required mechanical strength and sensitization resistance. Hence it can be concluded that CCR established for these materials quantitatively explains the influence of carbon and nitrogen. When nitrogen content is 0.22 wt.%, Cr_2N gets precipitated and CCR is marginally higher than the other materials with lower % of nitrogen.

5. Conclusions

In this paper, the TTS diagrams of four different nitrogen containing 316 SS (C-0.025–0.035% and N-0.07–0.22%) established as per ASTM standard A262 Practice A and E are presented. The sen-

sitization temperature range and nose temperature were same for all the four materials. For the material containing 0.22% nitrogen, the kinetics of sensitization was faster. The critical cooling rate of these plates were in the range of 9–14 K/h. The data presented in this paper is expected to be useful in selecting optimum soaking time and cooling rates while performing solution annealing, stress-relieving and dimensional stabilization heat treatments of certain critical components for the fast reactors.

Acknowledgements

The authors are grateful to Dr. Baldev Raj (Director, Indira Gandhi Centre for Atomic Research), Dr. P.R. Vasudeva Rao (Former Director, Metallurgy and Materials Group) and Dr. K. Bhanu Sankara Rao (Former Associate Director, Materials Development and Characterisation Group) Dr. T. Jayakumar (Director, Metallurgy and Materials Group) for their keen interest and support during the course of the investigation.

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