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A study of the effect of titanium on the void swelling behavior of D9 steels by ion beam simulation

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ABSTRACT

The void swelling behavior of D9 with 0.25Ti and D9 with 0.15Ti modified steels was studied using heavy ion irradiation. The cold worked samples were pre-implanted with a uniform helium concentration of 30 appm spanning a width of about 640 nm. This was followed by a 5 MeV nickel ion irradiation to create a peak damage of 100 dpa at a damage rate of 7×10^{-3} dpa s^{-1} at various temperatures between 700 and 970 K. The gross swelling up to the implanted range was measured by step height measurements. It is found that the peak swelling temperatures and the magnitude of swelling are different for the alloys studied. Positron lifetime measurements of the unirradiated alloys annealed at various temperatures show differences in the formation of TiC precipitates. The difference in void swelling behavior in these two alloys with titanium variation is discussed on the basis of the role of titanium on the vacancy migration and TiC precipitate formation.

1. Introduction

D9 steels and their modified versions with phosphorous additions (designated as D9I) are envisaged for use as fuel cladding and wrapper materials in the Indian fast breeder reactor (FBR) programme. In the FBR, the fuel clad tubes would experience temperatures in the range 673–973 K under steady state condition and the maximum neutron dose for target burn up of 100,000 MWd/t is 85 dpa [1]. The displacement damage produced in the material due to high energy neutrons results in point defects and a host of other defects. These point defects recombine, migrate to sinks or evolve into secondary defects like voids and dislocation loops. Voids have a profound influence on the material properties because the material undergoes volumetric swelling when the void forms and grows. The magnitude of this phenomenon would require fuel assemblies to be removed from the reactor before reaching the targeted burn up. Thus, one of the most important effects caused by irradiation in a neutron environment is the void swelling. The dimensional changes introduced in the material due to void swelling limits the lifetime of structural components used in a reactor. Therefore, resistance to void swelling is a major consideration in the choice of materials for the core components.

In order to improve the resistance to irradiation induced void swelling, efficient traps for vacancies and helium such as dislocations and precipitate-matrix interfaces have been introduced by adjusting alloying elements and thermo-mechanical treatments [2,3]. The titanium modified steels exhibited greatly improved swelling resistance under breeder reactor conditions [4] and consequently, have become a prime candidate for structural applications. In the cold worked Ti modified austenitic stainless steels, formation of fine stable precipitates of TiC termed as secondary precipitates have been reported to enhance the resistance against void swelling, helium embrittlement and in-pile creep during irradiation [5–9]. However, introduction of such high number density particles requires a fine tuning of alloy composition, which in turn requires understanding of the synergistic effects of alloying elements, thermal aging, and irradiation on phase stability.

For the improvement of swelling resistance of D9 alloy, one needs to optimize composition of minor alloying elements like P, Si, Ti, etc., which have a major influence on the swelling [10]. This optimization is brought about with a view of to produce microstructures, designed to minimize property degradation during the irradiation. In this paper we set out to explore the effect of titanium concentration on the void swelling behavior in ion irradiated D9 alloy. The role of ion irradiation as a surrogate for neutron irradiation in studying effects of minor elements and for basic studies in radiation damage is well established [7,11]. Accelerated heavy ions possess an inherent advantage of producing high displacement rates by virtue of its high displacement cross-sections compared to neutrons. Displacement rates of $\sim 10^{-2}$ dpa s^{-1} can be produced in comparison to the 10^{-6} dpa s^{-1} produced by its neutron counterpart. Thus ion irradiation can be used as a test

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bed for screening alloys, to decipher the role of minor elements on various irradiation induced property changes to the material, elucidating mechanisms and segregation studies in shorter time than the in-pile irradiation [12]. In the present study the alloys were pre-injected with 30 appm of helium followed by a 5 MeV Ni irradiation to a damage level of 100 dpa in the temperature range of 720–920 K. The swelling in these alloys was measured by surface profilometry.

The effect of precipitates and microstructures produced during aging is important as they minimize the effects of void swelling produced during irradiation. In this regard, positron lifetime measurements were carried out on the unirradiated alloys to monitor the defect recovery and the formation of TiC precipitates during isochronal annealing. Positron annihilation spectroscopy (PAS) is an established technique for studies on vacancy clustering and helium bubbles as well as studies on early stages of solute atom clustering and precipitation [13–16]. The ability of PAS to monitor the TiC precipitation process in D9 alloy has been reported [17]. In the present study the difference in the void swelling behavior of two alloys with different titanium concentration is discussed on the basis of the role of titanium in influencing the vacancy migration and the TiC precipitate formation.

2. Experimental details

2.1. Specimen preparation

D9 alloy samples of size 15 mm $\times$ 15 mm $\times$ 1.5 mm thick and 5 mm $\times$ 12 mm $\times$ 1.5 mm thick are spark cut from 18% cold worked hexagonal wrappers supplied by M/s. Valinox, France (designated as sample A) and 20% cold worked wrapper tubes obtained from NFC, Hyderabad (designated as sample B). Prior to cold working the wrapper materials were initially solution treated at 1343 K for 30 min. The samples were further mechanically polished and finished with a diamond-lapping compound to obtain a smooth surface. The thickness of the samples after polishing was $\sim$1.0 mm and the roughness of the polished surface was measured to be $\sim$100 Å. The chemical analysis of samples A and B using a direct reading spark emission spectrometer is given in Table 1.

Table 1
Chemical analysis of the D9 alloys obtained from spark emission spectrometry

Elements	Sample ID	
	Sample A (Concentration in wt%)	Sample B (Concentration in wt%)
Carbon	0.037 ± 0.005	0.037 ± 0.005
Chromium	15.0 ± 0.5	15.0 ± 0.5
Nickel	14.0 ± 0.5	14.0 ± 0.5
Manganese	1.90 ± 0.05	1.42 ± 0.05
Molybdenum	2.20 ± 0.05	2.10 ± 0.05
Phosphorus	<0.014	<0.014
Sulphur	<0.005	<0.005
Silicon	0.75 ± 0.05	0.88 ± 0.05
Cobalt	0.020 ± 0.005	0.020 ± 0.005
Copper	<0.050	<0.050
Titanium	0.250 ± 0.005	0.150 ± 0.005
Vanadium	0.045 ± 0.003	0.045 ± 0.003
Tungsten	<0.055	<0.055
Aluminum	<0.050	<0.050
Arsenic	<0.034	<0.034
Niobium	<0.016	<0.016
Lead	<0.070	<0.070
Tin	<0.006	<0.006
Cold work	18%	20%

2.2. Irradiation procedure

The samples of sizes 5 mm $\times$ 12 mm $\times$ 1.5 mm thick were used for ion irradiation experiments. The samples were first implanted with helium in order to simulate the effects of helium obtained by transmutation reactions in reactor conditions. The theoretical helium concentration per dpa for the PFBR has been computed to be $\sim$0.1 appm dpa^{-1}. Helium plays a crucial role in void nucleation in titanium modified steels and the amount of swelling increases with increasing helium concentration [18]. Therefore, in order to obtain discernable swelling which can be measured by step height measurements we have pre-injected a higher amount of helium $\sim$0.3 appm dpa^{-1}. The helium implantation scheme involves a series of implantation from 200 keV to 700 keV in steps of 100 keV. The fluence of each helium implantation is adjusted so as to yield a resultant constant profile of 30 appm spanning a width of $\sim$640 nm (refer to Fig. 1). Thus a uniform concentration of helium exists in the peak damage region of 5.0 MeV Ni^{2+} ions. All samples used in this study were irradiated with helium to specific fluence according to the irradiation scheme described above. Therefore, within the limits of error in charge integration ($\sim$<1%) they all have a similar concentration of helium. No analytical technique was used to measure the concentration of implanted helium in the samples. The specimens which were pre-injected with helium were subsequently implanted with 5.0 MeV Ni^{2+} ions to a fluence of about 9.0×10^{16} ions cm^{-2} using a 1.7 MV Tandetron accelerator. This irradiation produces $\sim$100 dpa at the damage peak as calculated by the TRIM program [19] invoking modified Kinchin-Pease analytic solution for the calculation of displacement damage. The range and straggling of 5.0 MeV Ni^{2+} ions in stainless steels as calculated from the TRIM program are 1140 nm and 118 nm, respectively. The scheme of irradiation is shown in Fig. 1. The nickel ion irradiation was carried out at temperatures ranging from 720 to 970 K. The sample during irradiation was mounted on a UHV manipulator equipped with a high temperature heating attachment. The temperature stability during irradiation was ±2 K. During implantation the irradiation chamber vacuum was maintained at 4×10^{-7} mbar. The sample after the high temperature irradiation was quenched to the ambient temperature using a jet of helium gas cooled by liquid nitrogen. For both samples A and B only one irradiation was carried out at each temperature.

2.3. Step height measurements

For carrying out the step height measurements, the specimen was partially masked with a stainless steel mask with a knife edge

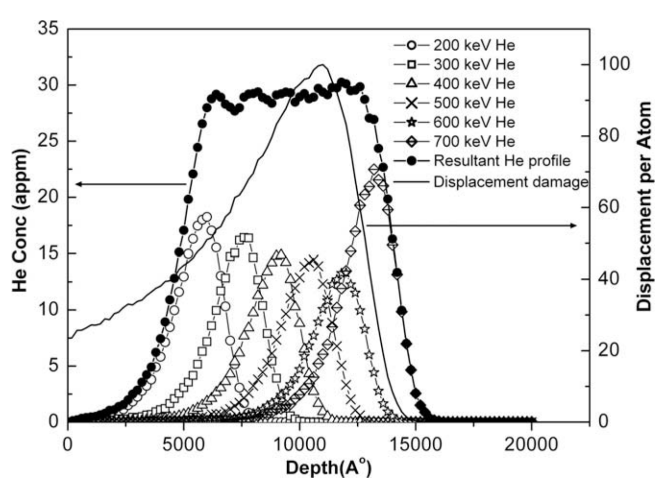

Fig. 1. Implantation scheme showing helium concentration profile resulting from an irradiation scheme and 5 MeV nickel damage profile simulated using the TRIM program.

during nickel ion irradiation, so that only the unmasked region was irradiated. As the bombarded region swells, the surface becomes elevated and a step forms at the interface between the masked and unmasked regions. A measurement of this step height provides the total integrated swelling that has occurred along the path of the bombarding ion [20]. The step height was measured using a surface profilometer. In order to arrive at the quantitative estimate of volumetric swelling, suitable corrections were made for the changes in step height due to injected nickel ions and for the sputtering of the sample by the nickel ions [20]. As the range of the 5 MeV nickel ions is much less than 1% of the specimen thickness, the swelling in this thin layer is laterally constrained by the bulk of the material underneath. Thus the total volume change was accommodated by a movement of mass perpendicular to the sample surface. The height 'h' of the plateau elevated from the initial surface could be given by a rough estimation [21] as,

$$h = \frac{S_A}{1 + S_A} \Delta R, \tag{1}$$

where h = step height (Å), ΔR = visible void spreading width (Å), S_A = the average void swelling (%) and ΔR was assumed to be $R_p + \frac{\Delta R_p}{2}$, where R_p and ΔR_p are the range (Å) and straggling (Å) of the nickel ions. From the measurements of h, the void swelling S_A was estimated.

2.4. Positron life time measurements

Isochronal annealing treatments were carried out from 300 to 1273 K in steps of 50 K on unirradiated samples belonging to the same stock as the irradiated samples in a vacuum of 10^{-6} mbar. The annealing time for each temperature was fixed at 30 min based on previous studies [17,30]. Since we are following the different stages which evolve during annealing from the initial defect state, decreasing or increasing the annealing time will respectively postpone or advance the onset of the subsequent stages [22]. In other words, the onset of the subsequent stages will occur either at a lower temperature or higher temperature depending upon the time of annealing. Thus, while making a comparison of alloys with respect to their defect evolution it is imperative to carry out isochronal annealing for the same time. The positron lifetime measurements were carried out at room temperature after each isochronal annealing step. The positron lifetime measurements were measured with a fast-fast coincidence system coupled with a pair of BaF_2 detectors having a time resolution of 260 ps. ^{22}Na of 10 μCi activity evaporated on a thin nickel foil was used as the source. The lifetime measurements were performed using the standard sample-source sandwich geometry. Samples from the same stock as the irradiated samples but with sizes 15 mm × 15 mm × 1.5 mm thick were used. The samples of larger dimensions were used for positron lifetime measurements in order to enable efficient sandwiching of the source. The measured life time spectra were analysed into different lifetime components and their intensities using programs RESOLUTION and POSITRONFIT [23].

3. Results and discussion

Fig. 2 shows the variation of void swelling as a function of irradiation temperature for the samples A and B. The experimentally measured values of void swelling at different temperatures are enlisted in Table 2. For both alloys the swelling increases as a function of temperature to a peak value and thereafter decreases at higher temperature. At low temperatures the swelling is low because the defect migration is low and hence resulting in high steady-state concentrations of interstitials and vacancies [12,24]. This leads to an enhanced recombination and only few defects migrate to biased sinks to cause swelling. As the temperature is increased, recombination is no more a dominant process as the vacancies become mobile and the defects migrate to sinks. At such temperatures in the presence of a dislocation bias (i.e., the dislocations absorb more interstitials compared to vacancies) the void growth takes place [24]. The drop in swelling at temperatures beyond the peak swelling temperatures is due to the thermal emission of vacancies from the voids. The temperature versus swelling relationship of both the alloys (as shown in Fig. 2) is in agreement with the physical picture of void formation. However the magnitude of swelling at the peak swelling temperature and the peak swelling temperatures are different for the two alloys. While the alloy with 0.15% Ti displayed a swelling of ∼15% at the peak swelling temperature of 923 K, the alloy with 0.25% Ti has a swelling maximum of ∼4% at the peak swelling temperature of 823 K. With similar thermo-mechanical treatment effected on both the samples the difference in void swelling behavior can be attributed to the difference in their titanium content. The role of titanium in influencing swelling behavior in titanium modified steels is in several ways: (a) The residual gases are strongly gettered and are thus unavailable for stabilization of void nuclei; (b) the cold-work dislocation structure is stabilized (c) the interaction between helium atoms and the TiC particle-matrix interface strongly modifies the helium bubble distribution [4]. Considering the various precipitates formed during thermal aging in Fe–Cr–Ni alloys with titanium, molybdenum and carbon additions one finds that there is a presence of TiC and $M_{23}C_6$ precipitates apart from the sluggish laves and eta (η) phases [25]. The $M_{23}C_6$ precipitates are mostly formed along the grain boundaries and deformation bands as large precipitates. In

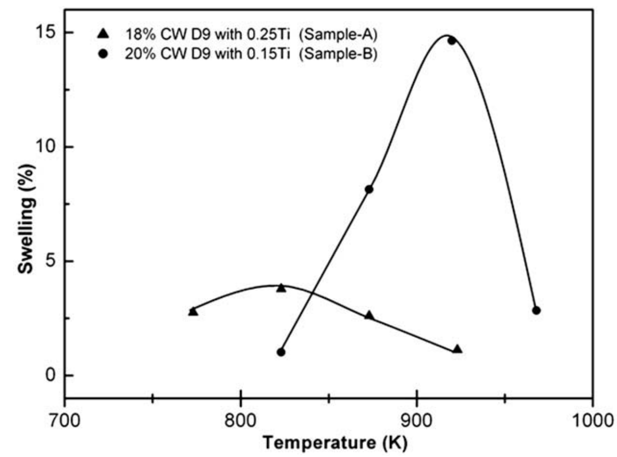

Fig. 2. Temperature dependence of void swelling measured by surface profilometry for the D9 alloys with different titanium concentration.

Table 2
The magnitude of swelling for the D9 alloys irradiated at different temperatures

Temperature (K)	Step height (Å)		Void swelling (%)	
	Sample A	Sample B	Sample A	Sample B
773	336.83	ND	2.76	ND
823	432	124.75	3.78	1.02
873	280.7	930	2.60	8.15
920	134.17	1577.5	1.12	14.65
968	ND	341.83	ND	2.85

ND: The irradiation was not carried out at this temperature.
The tabulated values for step heights are the net step heights i.e. with corrections made for height decrease due to sputtered material (55 Å, calculated from the TRIM program) and height increase due to the injected ions (105 Å).

contrast, the TiC precipitates which are formed in high number density along the dislocations are fine precipitates (20–30 nm) [25]. Moreover, the $M_{23}C_6$ precipitates reduced in number when titanium is present in the alloy because carbon was depleted to form TiC precipitates [25]. In relevance to swelling $M_{23}C_6$, laves and eta (η) phases are undesirable as they form in low number density and large sizes. In such cases both helium atoms and vacancies are shared by a small number of precipitates thus leading to earlier formation of critical cavity size [25]. On the other hand presence of TiC precipitates in large number density disperses the helium atoms resulting in delayed formation of critical sized void nuclei. Besides, fine precipitates of TiC are strong neutral sinks for vacancies and interstitials and hence act as sites for increased recombination of vacancies and interstitials. Coherent precipitates like TiC act as traps for vacancies and interstitials. The coherent precipitate is of a structure in which the lattice planes are continuous with those of the matrix but however there is a strain field at the interface of the precipitate and matrix [12]. The TiC precipitates have a face-centered cubic structure with an extremely high lattice mismatch with the austenitic matrix. The difference in lattice parameter ranges from 19% to 21% resulting in large strain at the TiC matrix interface [26]. The source of attraction of vacancies and interstitials to the trap is the relief of the strain field and the trap strength is only limited by the capacity of the interface to hold the defect until the anti-defect arrives [27]. The TiC precipitates which are fine sized have large precipitate matrix interface area which facilitates increased recombination of point defects. Void swelling studies on proton irradiated Japanese PCA alloy steels (Fe–16.2Ni–14.6Cr–2.37Mo–1.79Mn–0.53Si–0.24Ti–0.06C) have shown a qualitative dependence of decrease in swelling with increased formation of TiC precipitates [28]. Thus a comparison of TiC precipitates formation in the alloys would resolve the difference in their influence on the swelling behavior.

Positron life time measurements were carried out on the samples A and B to monitor the defect recovery during isochronal annealing. Fig. 3 shows the variation of positron lifetime τ, with annealing temperature for the samples A and B. The lifetime corresponding to the solution annealed state is 110 ps and is indicated in the figure. The observed variation of lifetime τ displays distinct stages viz., a monotonic decrease in τ from the initial cold worked state up to ~900 K in sample B and ~ 800 K in sample A. This is

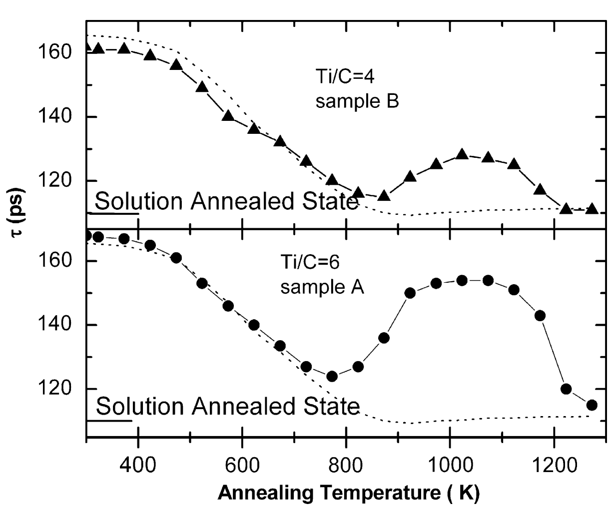

Fig. 3. Variation of positron life time with annealing temperature for the cold-worked D9 alloys with different titanium concentration, sample A 18% CW (●) and sample B 20% CW (▲). The dashed lines in both graphs correspond to the recovery curve for cold worked Ti-free steel samples. The lifetime corresponding to the solution annealed state is 110 ps and is indicated by a solid line.

followed by a stage where there is an increase in lifetime to saturation, followed by a decrease in lifetime. The first stage corresponds to point defect recovery and it is due to the migration of vacancies to sinks such as cold worked dislocations thus resulting in the annihilation of dislocations [17]. The subsequent stage where there is an increase in lifetime τ is the result of positron trapping at the TiC precipitate-austenitic matrix interface [17]. Earlier TEM studies reported [29] the formation of TiC precipitates in cold worked titanium modified stainless steels in the temperature range of 923 to 1073 K. These studies showed that the process of precipitation is dislocation controlled. Large strains arising from the lattice mismatch of the TiC and the austenite matrix results in generation of misfit dislocations at the TiC-matrix interface region [31]. These misfit dislocations are effective traps for positrons [17]. Thus an increase in lifetime indicates an increase in positron trapping rate arising from the increase in number density of TiC precipitates [17]. Such an increase reaches a peak value at ~1050 K suggesting that the TiC precipitation is complete by this temperature. This observation is in accordance with the reported TEM studies [29]. The dashed lines in Fig. 3 correspond to the recovery curve for cold worked Ti-free steel samples. And quite evidently there is no increase in lifetime in the 800 – 1100 K regime. This further confirms that the observed increase in lifetime in titanium containing cold worked samples is due positrons annihilating in the TiC precipitate matrix interface [17]. Another feature that we observe in Fig. 3 is that the lifetime value for the sample A is more than that of sample B. This increase in positron trapping rate can arise either due to change in the nature of the positron traps or due to the increase in number density of traps [30]. Since the only traps for positrons are misfit dislocations at these temperatures, the increase in positron trapping rate is attributed to the increase in number density of misfit dislocations. The increase in number density of misfit dislocations can arise only from an increased concentration of TiC precipitates [30]. Thus the increase in average lifetime of positrons trapped in the sample A in comparison to sample B is due to the increase in the number density of TiC precipitates formed in the former. Thus the increase in number density of TiC precipitates formed in sample A in comparison to sample B has resulted in a decrease in the magnitude of swelling at the peak swelling temperature in sample A. The decrease in lifetime beyond ~1200 K in both alloys is ascribed to the decrease in the number density of TiC precipitates due to coarsening of the TiC precipitates [17]. Also one finds that the onset of TiC precipitate formation has occurred at a lower temperature in sample A than in sample B. This may be due to the difference in kinetics of formation and growth of TiC precipitates as a result of varying titanium concentration. The excess amounts of titanium in sample A has resulted in increased nucleation sites for the formation of TiC precipitates. Therefore at a given temperature it is likely that more number of TiC precipitates have nucleated in sample A than in sample B.

In addition to the above mentioned effects the TiC precipitates also seem to influence swelling beyond the peak swelling temperatures. The decrease in swelling at temperatures above the peak swelling temperature is usually ascribed to the difficulty in nucleating voids (due to the decrease in vacancy supersaturation) and thermal emission of vacancies from voids hence discouraging void growth [12,24]. The presence of TiC precipitates has resulted in increased recombination of point defects at its interface. Thus there is a decrease in vacancy supersaturation and this is more likely at higher temperatures where the equilibrium concentration of vacancies is high. Also one finds that the onset of TiC precipitation has occurred at a lower temperatures in sample A in comparison to sample B (refer to Fig. 3). Thus the decrease in vacancy supersaturation should occur at lower temperatures in sample A than in sample B. And which is why one observes a low amount of swelling in sample A at temperatures corresponding to the peak swelling temperature of sample B.

While evaluating the effect of titanium additions on swelling it should be mentioned that titanium being an oversized atom in austenitic alloys compared with the other major alloying elements interacts with the vacancies and hence diffuses via vacancy migration being subject to the inverse Kirkendall effect. Titanium is a fast diffusing species diffusing faster in austenite by a factor of 7.6 times than Fe atoms [32]. Therefore, the effective diffusion coefficient of vacancy migration will be elevated by titanium additions. Recently, Okita et al. [32] have compared the swelling behavior in Fe–15Cr–16Ni and Fe–15Cr–16Ni–0.25Ti and have observed that the peak swelling temperature of the ternary alloy is greater than that of ternary alloy with 0.25% titanium addition by $\sim$100 K. This effect of titanium in solution is consistent with our results (refer to Fig. 2) where the alloy with higher titanium concentration has a peak swelling temperature lower than the other. The increase in vacancy migration has resulted in greater recombination of the point defects and migration of vacancies to sinks at lower temperatures. Therefore, in sample A the recombination dominated regime is shortened and void growth regime is attained at lower temperatures compared to sample B. This has resulted in the reduction of peak swelling temperature in sample A by 100 K in comparison to sample B. Therefore, we conjecture that the presence of greater concentration of titanium in sample A in comparison to sample B has resulted in increased effective vacancy diffusion coefficient and hence a decrease in peak swelling temperature of sample A.

Thus from this study one can conclude that the increase in titanium concentration in D9 alloy from 0.15% to 0.25% has resulted in lowering of the peak swelling temperature by 100 K due to the increase in vacancy migration. The increase in titanium concentration also has led to an enhanced production of TiC precipitates thus resulting in restrained swelling in the alloys with higher Ti content.

4. Conclusion

Void swelling behavior has been investigated by step height measurements in Ni-ion irradiated titanium modified D9 steels with varying titanium concentrations. The swelling at the peak swelling temperature of the alloys with 0.25% of titanium and 0.15% of titanium are found to be $\sim$4% and $\sim$15% respectively. The TiC precipitate formation in these two alloys was studied by positron lifetime measurements of the unirradiated alloys which are annealed at various temperatures. The reduced swelling in the alloy with 0.25% titanium is due to the greater number density of TiC precipitates formed. The difference in peak swelling temperatures between the two alloys has been ascribed to the effect of titanium in solution in increasing the effective vacancy diffusion coefficient.

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