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# Improvement in Creep Damage Tolerance of 14Cr-15Ni-Ti Modified Stainless Steel by Addition of Minor Elements

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# Abstract

Creep properties of 14Cr-15Ni-Ti modified steel, alloyed with phosphorus and silicon were investigated at 973 K in the stress range 175-250 MPa. The phosphorus content in the alloys was 0.025 and 0.04 wt.%, silicon 0.75 and 0.95 wt.% and titanium in the range 0.16-0.3wt.%. The variation between minimum creep rate and rupture life for these alloys was found to follow the Modified Monkman Grant relationship. The inverse of Modified Monkman Grant relationship which is defined as the damage tolerance parameter was found to be above ten for these alloys, indicating that these alloys can withstand high strain concentrations. Optical microscopic investigations revealed extensive matrix deformation and precipitation. Creep damage in the form of cracks or cavities was not observed in these alloys corroborating high tolerance for creep damage. Addition of boron was found to prevent grain boundary damage.

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Keywords: Monkman grant relationship; creep damage tolerance parameter; matrix deformation; strain concentration; minimum creep rate

# 1. Introduction

The relation between minimum creep rate,  $\dot{\mathcal{E}}_m$  and rupture life,  $t_r$  is generally expressed as

$$\dot{\mathcal{E}}_m t_r = C_{MG} \tag{1}$$

where  $C_{MG}$  is the Monkman Grant constant, lies in the range 0.05-0.5 [1]. Dobes and Milicka [2] noted that the scatter of experimental values obtained when using equation (1) can be considerably reduced by using a modified equation of the form

$$\dot{\mathcal{E}}_m t_r / \mathcal{E}_T = C_{MMG}$$

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where  $\mathcal{E}_T$  is the fracture strain and  $C_{MMG}$  is the modified Monkman-Grant constant. Numerous investigators have consistently found that Eq. (2) provides a better fit of the data than Eq.(1) [2-7]. The inverse of Modified Monkman Grant relationship,  $\mathcal{E}_T / \dot{\mathcal{E}}_m t_r$  is the damage tolerance parameter  $\lambda$ . The magnitude of the creep damage tolerance parameter is important in practical situations when high strains develop in regions, say, where a change in component cross-section leads to stress concentrations. Values of  $\lambda$  equal to 5 or more are then thought to ensure that the localised strain levels typically encountered during service will not lead to premature cracking. In addition, for theoretical studies, the  $\lambda$  value may provide an insight for the damage mechanisms responsible for tertiary creep and eventual fracture [8].

In this investigation, creep studies have been conducted on various heats of 14Cr-15Ni-Ti modified steel alloyed with phosphorus (P) and silicon (Si), (a candidate material for the core applications of Proto Type Fast Breeder Reactor, which is in the advanced stage of construction at Kalpakkam) at 973 K in the stress range 175-250 MPa. The relation between minimum creep rate, rupture life and rupture ductility was established using Modified Monkmann Grant relationship and the inverse of Modified Monkmann Grant relationship was evaluated to identify the damage mechanism.

## 2. Experimental

Laboratory heats of 14Cr-15Ni-Ti modified stainless steel were prepared by vacuum induction melting (VIM) of high purity charge of electrolytic grades of ferrochrome, followed by vacuum arc remelting (VAR) process. The VAR ingots were hot forged and then hot rolled into plates of 12 mm thickness, subsequently solution annealed in the temperature range of 1323-1423 K, pickled and passivated. Creep properties of these heats were investigated at 973 K for the heats in 20% cold worked condition. The chemical composition of the various heats of the material investigated is given in Table 1. The average grain size of the material was 40  $\pm$  7 microns. Optical, scanning and electron microscopic studies were conducted to characterise the microstructure, fracture behaviour.

	Р	Si	Ti	Ni	Cr	Mn	Mo	С	Ν	В	S
Heat No.											
0.025 P 0.75 Si 0.16 Ti	0.026	0.76	0.18	15.53	14.38	1.71	2.36	0.047	0.008	0.0053	0.004
0.025 P 0.75 Si 0.2 Ti	0.025	0.75	0.23	15.49	14.39	1.64	2.35	0.039	0.008	0.0050	0.004
0.025 P 0.75 Si 0.24 Ti	0.026	0.77	0.25	15.48	14.37	1.51	2.33	0.040	0.009	0.0050	0.004
0.025 P 0.75 Si 0.3 Ti	0.025	0.73	0.36	15.43	14.29	1.63	2.35	0.042	0.008	0.0056	0.004
0.04 P 0.75Si 0.24Ti	0.034	0.74	0.25	15.38	14.35	2.36	2.40	0.046	0.01	0.0050	0.005
0.25P 0.95Si 0.24Ti	0.022	0.91	0.29	15.36	14.24	1.76	2.35	0.054	0.008	0.0055	0.006
0.04P 0.95Si 0.16Ti	0.039	1.01	0.20	15.48	14.27	1.92	2.37	0.038	0.007	0.0051	< .01
0.04P 0.95Si 0.20Ti	0.040	1.02	0.25	15.37	14.29	2.22	2.39	0.042	0.008	0.0050	0.003
0.04P 0.95Si 0.24Ti	0.038	1.00	0.26	15.25	14.14	2.33	2.34	0.043	0.007	0.0053	0.004
0.04P 0.95Si 0.3Ti	0.041	0.97	0.29	15.45	14.27	1.80	2.33	0.040	0.008	0.0052	0.003

Table 1.Chemical composition of various heats of 14Cr-15Ni-Ti modified steel (in wt.%).

# 3. Results and discussion

#### 3.1. Creep curves

The creep curves (strain vs. time) for various heats are shown in figure1. Generally, all the three regions of the creep curve were observed for some of the alloys. The variation of creep rate with creep strain (Fig.2) exhibited a primary and a short secondary. Figure 3 shows a plot between minimum creep rate and applied stress for the alloys containing nearly the same amount of titanium.



Fig.1. Creep curves at a stress level of 175 MPa for various heats of 14Cr-15Ni-Ti modified steel.

Fig.2. Variation of creep strain rate with creep strain for various heats of 14Cr-15Ni-Ti modified steel.

A power law relationship was found to be obeyed between minimum creep rate and applied stress. The stress exponent values were 9 for the alloy containing phosphorus 0.025, silicon 0.75 wt.%, 5 for the alloy containing P 0.025 Si 0.95 wt.%, 7 for the alloy containing P 0.04 Si 0.75 wt.% and 3 for the alloy containing P 0.04 Si 0.95 wt.%.



Fig.3. Variation of minimum creep rate with applied stress for various heats of 14Cr-15Ni-Ti modified steel.

These values indicate that the material deforms by dislocation creep. As can be seen from figure 1 in most of the alloys, strain gets accumulated in the tertiary stage. Many different processes can start tertiary acceleration, including grain boundary cavity and crack development, neck formation and particle coarsening in precipitation

hardened alloys [8].

#### 3.2. Damage tolerance parameter

Figure 4 shows a plot of variation of minimum creep rate with rupture life (normalized by rupture strain) indicating the validity of Modified Monkman Grant constant. The inverse of modified Monkman Grant relationship is the damage tolerance parameter,  $\lambda$ . Figure 5 shows a plot of damage tolerance parameter with rupture life for all the heats. From these figures it can be concluded that the damage tolerance parameter values are more than 10 indicating that the material can tolerate every large strain concentrations. The large values of  $\lambda$ , arises from the higher ductility exhibited by this material (more than 10). In an earlier study [9] on 15Cr-15Ni-Ti modified steel, the ductility was low, less than 10% and the damage tolerance parameter also was less than 10. Modelling exercises [8] have predicted that  $\lambda < 2.5$  when tertiary creep and fracture are attributable to cavitation, with higher values expected when the tertiary stage begins as a consequence of necking (with or precipitate coarsening with  $\lambda > 5$ ). The increase in ductility of the alloys, in the present study, can be attributed to the addition of boron, around 50ppm. In 15Cr-15Ni–Ti modified steel, studied earlier; the boron content was around 10 ppm. Addition of Boron in ppm levels was found to improve creep ductility in 304, 321 and 347 stainless steels [10]. The higher damage tolerance parameter values exhibited by 14Cr-15Ni–Ti steel are correlated with the metallographic investigations. This aspect is discussed in the following section.



Fig.4. Validity of modified Monkman Grant relationship for various heats of 14Cr-15Ni-Ti modified steel.

Fig.5. Variation of damage tolerance parameter with rupture life.

# 3.3. Microstructural correlations

Figure 6 shows an optical micrograph of the alloy containing P 0.025 Si 0.75 and titanium 0.25 wt.%. It can be seen from the figures that the material contains well deformed grains and precipitates on the deformation bands. Creep damage in the form of cracks was not observed in all the heats. Boron addition in high temperature alloys has been reported to increase creep rupture strength [11-13]. It is widely believed that the addition of boron increases creep rupture life and ductility through the increase in creep cavitation resistance of the steel by grain boundary strengthening. The reason for strengthening of the grain boundary is not precisely known. In most instances, it is thought that boron is concentrated on grain boundaries, where it enters into the precipitates and alters the character of the grain boundary/precipitates or matrix precipitates in such a way to suppress microcavity formation. Figure 7 shows the scanning electron micrograph of the heat containing P 0.025 Si 0.75 wt.% and titanium 0.25 wt.%. The fracture surface of this alloy is characterised by predominantly ductile dimples with little cavitation indicating ductile failure. Addition of copper (Cu), boron (B) and cerium (Ce) was found to suppress cavity growth rate significantly in 347 stainless steels [14]. The significant decrease

in cavity growth rate in 347CuBCe steel is believed to be associated with the segregation of boron instead of sulphur on cavity surface. Segregation of elements on interfaces is reported to decrease interface diffusivities. On segregation of high melting point (2353 K) boron instead of low melting point (386 K) sulphur on the creep cavity surface, the surface diffusivity might have reduced significantly to retard cavity growth. In the present study the addition of 50 ppm of boron reduces creep damage and cavitation and improves ductility. Boron being an interstitial element is known to decrease the solubility of carbon and nitrogen in austenitic stainless steel [15] and accelerate metal–carbonitride precipitation. Boron replaces some carbon in  $M_{23}C_6$  carbide to form  $M_{23}(C, B)_6$  and increases its stability. In an austenitic stainless steel, Fujiwara et al. [11] attributed the increase in creep strength upon boron addition to the enhanced precipitation of  $M_{23}C_6$  carbides on the dislocations. Increased precipitation as well as better stability of the precipitates, might be the reasons for the enhanced creep deformation and grain boundary sliding resistances of the steels on boron addition.



Fig.6. Microstructure of the alloy containing P 0.025 Si 0.75 and Ti 0.23 wt.% creep tested at 175 MPa at 973 K showing well deformed grains, deformation bands and precipitation.



Fig.7. Fracture surface of the alloy containing P 0.025 Si 0.75 and Ti0.25 wt.% creep tested at 200 MPa at 973 K exhibiting excess dimples.

# 4. Conclusions

- The three stages of creep curve, primary secondary and tertiary were observed for some of the alloys.
- The deformation behaviour of the steels is identified as dislocation creep aided by diffusion of vacancies.
- Modified Monkman Grant relationship was found to establish the relation between minimum creep rate, rupture life and rupture strain.
- The inverse of modified Monkman Grant relationship, the damage tolerance parameter was high above ten for all the alloys indicating that the steel can withstand high strain concentrations. The higher values of damage tolerance parameter are due to increased ductility, caused by the addition of Boron which restricts creep cavity growth.

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