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Work is being submitted to the Proceeding of the Second Topical Meeting on
 Fusion Reactor Materials: Seattle, Washington, August 9-12, 1981.

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IRRADIATION CREEP IN SIMPLE BINARY ALLOYS*

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Creep enhancement during 21-MeV deuteron irradiation was examined at 350°C for two simple binary alloys with representative microstructures, i.e. solid-solution (Ni - 4 at. % Si) and precipitation-hardened (Ni - 12.8 at. % Al) alloys. Coherent precipitates were found to be very effective in suppressing irradiation-enhanced creep. Si solute atoms depressed irradiation creep moderately and caused irradiation hardening via radiation-induced segregation. The stress-dependence of irradiation creep in Ni - 4 at. % Si showed a transition, which seems to reflect a change of mechanism from dislocation climb due to stress-induced preferential absorption (SIPA) to climb-controlled dislocation glide enhanced by irradiation.

1. INTRODUCTION

Evaluation of irradiation creep behavior in anticipated fusion reactor environments by extrapolating data from existing irradiation facilities cannot be done with confidence without a sound understanding of the mechanisms involved in irradiation creep. In-situ creep measurement during bombardment by energetic light ions is quite suitable for this type of mechanistic study because excellent control of temperature, stress and damage rate can be achieved with this technique. However, the major disadvantage of this technique is that experiments are limited to low dose levels. It is, therefore, unsuitable for investigation of the effects of microstructural evolution commonly observed in complicated commercial materials. This point is best demonstrated by a "negative irradiation creep", i.e. deformation against the applied stress, which has been observed in solution treated 316 [1] and 321 [2] stainless steels. This phenomenon is suspected to be associated with microstructural evolution, e.g. a shear modulus change caused by solute segregation [1] or volume contraction resulting from precipitation [2], during the early stage of irradiation.

It would be possible to investigate high dose effects by irradiating specimens to high damage levels in a reactor prior to the in-situ creep experiment with light ion bombardment. This technique would be necessary in order to meaningfully apply the light-ion bombardment technique to complicated commercial alloys. However, the mechanisms of radiation enhancement of creep for representative microstructural conditions can be investigated with ion-irradiation techniques by utilizing model alloys with different microstructures that will remain stable for the duration of the experiment.

This paper will discuss the irradiation creep behavior of solid solution (Ni - 4 at. % Si) and

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precipitation hardened (Ni - 12.8 at. % Al) materials. It was found that the two microstructures exhibit significantly different irradiation creep behavior.

2. EXPERIMENTAL PROCEDURE

Ni - 4 at. % Si (Ni-4 Si) and Ni - 12.8 at. % Al (Ni-12.8 Al) alloys were prepared from high purity nickel, silicon and aluminum by arc-melting, followed by levitation melting to homogenize the composition. These alloys and high purity nickel (99.999%) were processed into 0.75 mm diameter wires by swaging. The nickel wires and Ni-4 Si wires were annealed in an inert environment at 550°C for 10 minutes and at 665°C for 2 hours, respectively. These heat treatments were selected to give a grain size of about 10 μm . Ni-12.8 Al wires were solution annealed at 875°C for 17 minutes followed by aging at 615°C for 1.5 hours. This heat treatment gave a grain size of about 30 μm . The creep specimens were fabricated from these wires by electropolishing a reduced section (~ 0.13 mm diameter and ~ 6.5 mm long).

Creep measurements were made in a torsional-creep apparatus, which is described in detail elsewhere [1, 3]. A 21 MeV deuteron beam from the ANL 60-inch Cyclotron was used for the irradiation experiments. 21 MeV deuterons produce fairly uniform damage throughout the thin wire specimen (the damage rate is only 10% greater at the exit surface than at the entrance surface). All experiments including in-situ creep during bombardment were conducted at 350°C.

3. RESULTS

Figure 1 shows the creep behavior of pure nickel, Ni-4 Si (solid solution alloy), and Ni-12.8 Al (precipitation hardened alloy) during irradiation by 21 MeV deuterons, at a damage rate of 2×10^{-6} dpa s⁻¹. The experiments were

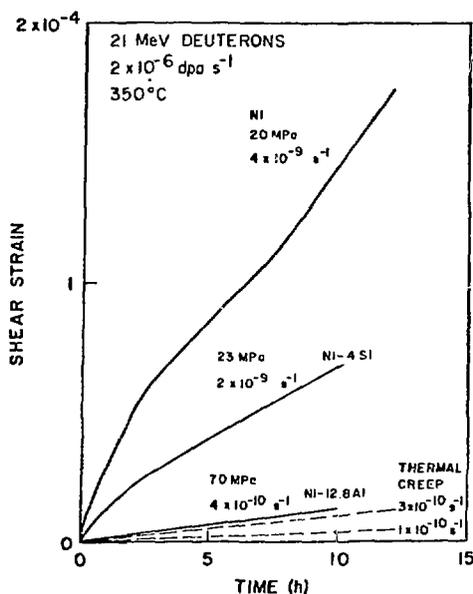


Fig. 1. Creep behavior of pure nickel, Ni - 4 at. % Si (solid solution alloy) and Ni - 12.8 at. % Al (precipitation hardened alloy) at 350°C during irradiation at a damage rate of $2 \times 10^{-6} \text{ dpa s}^{-1}$. Thermal creep rate of $3 \times 10^{-10} \text{ s}^{-1}$ corresponds to the pre and postirradiation behavior of nickel and the preirradiation behavior of Ni-4 Si, $1 \times 10^{-10} \text{ s}^{-1}$ corresponds to the pre and postirradiation behavior of Ni-12.8 Al and postirradiation behavior of Ni-4 Si.

conducted at 350°C and the stress levels were chosen to give comparable pre or postirradiation thermal creep rates (1 to $3 \times 10^{-10} \text{ s}^{-1}$). The steady state portions of the thermal creep curves are also shown in Fig. 1 for reference. For all of the materials, the radiation increases the creep rate. However, the magnitude of the enhancement is quite dependent on the material composition, microstructure and, as will be shown below, the applied stress.

The pre and postirradiation thermal creep rates for pure nickel and Ni-12.8 Al were nearly equal, indicating that no significant hardening has been caused by the irradiation. However, irradiation to 0.07 dpa reduced the thermal creep rate of Ni-4 Si by approximately a factor of 3 at all stress levels investigated (Fig. 2). A study of this hardening effect was carried out and has been reported elsewhere [4]. The results showed that the hardening occurs very rapidly and becomes nearly saturated after a dose of only 0.01 dpa. For this reason, all of the experiments in the study of Ni-4 Si were done on samples that had been irradiated to at least 0.03 dpa.

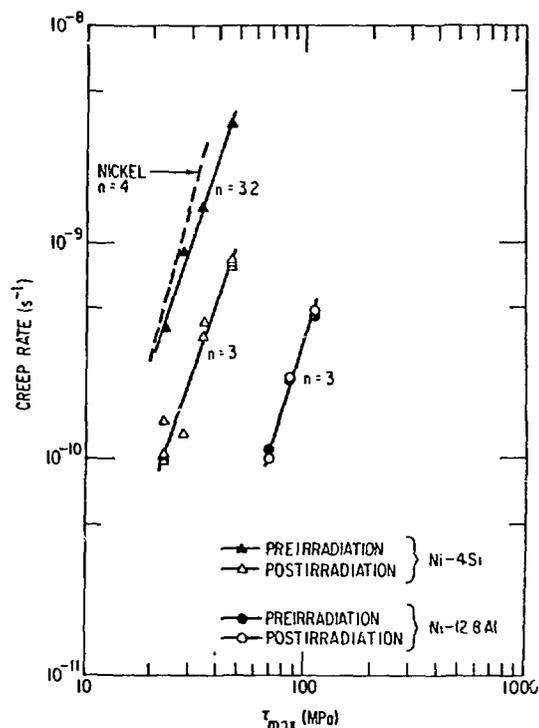


Fig. 2. Comparison of the stress dependence of the thermal creep rate for pure nickel, Ni-4 Si and Ni-12.8 Al alloys in preirradiation and postirradiation ($\sim 0.07 \text{ dpa}$) conditions.

Figure 3 shows the stress dependence of the irradiation creep rate (the total creep rate minus the postirradiation thermal creep rate) of Ni-4 Si. The creep rate has a linear dependence on the applied stress below 60 MPa. Above this stress, the stress exponent increases smoothly to 3 which is the same as that for thermal creep. In the high stress regime, the irradiation creep rate for a damage rate of $1.3 \times 10^{-6} \text{ dpa s}^{-1}$ is somewhat less than the thermal creep rate. This means that the total creep rate is increased by less than a factor of 2 under these conditions. In the low stress regime, at 20 MPa, the irradiation increases the creep rate by an order of magnitude.

Figure 4 shows the effect of damage rate on the irradiation creep rate. At 34.5 MPa, the irradiation creep rate is clearly linear in damage rate. Two experimental points at 23 MPa are in good agreement with a linear dependence. Three points for a stress of 103 MPa, which is well into the high stress regime, indicate a less than linear damage rate dependence, but additional experiments would be required to verify this result.

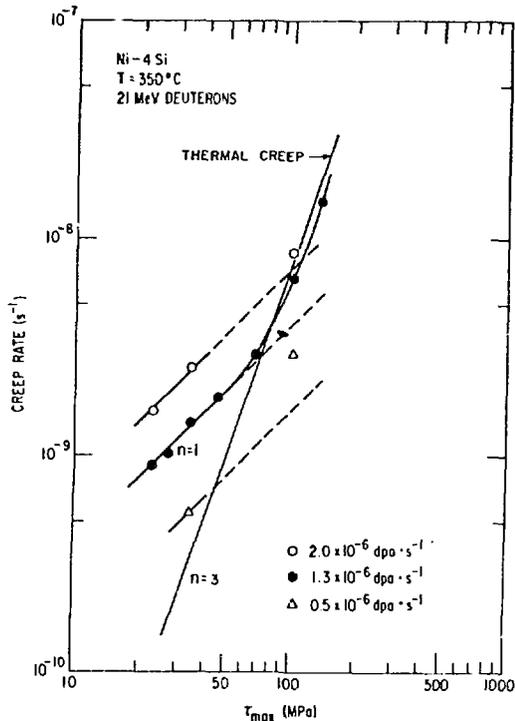


Fig. 3. Stress dependence of the irradiation creep rate (irradiation-enhanced component) for Ni-4 Si. Thermal creep rate is also shown for comparison.

4. DISCUSSION

A number of mechanisms have been proposed for irradiation enhanced creep. The most favored among these mechanisms are [5] stress-induced preferred nucleation (SIPN) of point defect aggregates, dislocation climb due to stress-induced preferential absorption (SIPA) of point defects, and climb-controlled dislocation glide (CG) enhanced by irradiation. The first two mechanisms predict a linear stress dependence of the irradiation creep rate, whereas the CG model is expected to give a stress dependence that is at least quadratic [6] and possibly as large as that for thermal creep. The stress dependence found for irradiation creep of Ni-4 Si showed a transition from linear to a higher order at 60 MPa. Therefore, in the low stress regime, SIPN, SIPA or some variation of these mechanisms appear to be responsible for the irradiation produced component of the creep. Since the SIPN mechanism should prevail only at a very early stage of irradiation [7], SIPA climb of dislocations (both network dislocations and Frank loops) should be the major mechanism. The fact that the irradiation creep rate vs stress

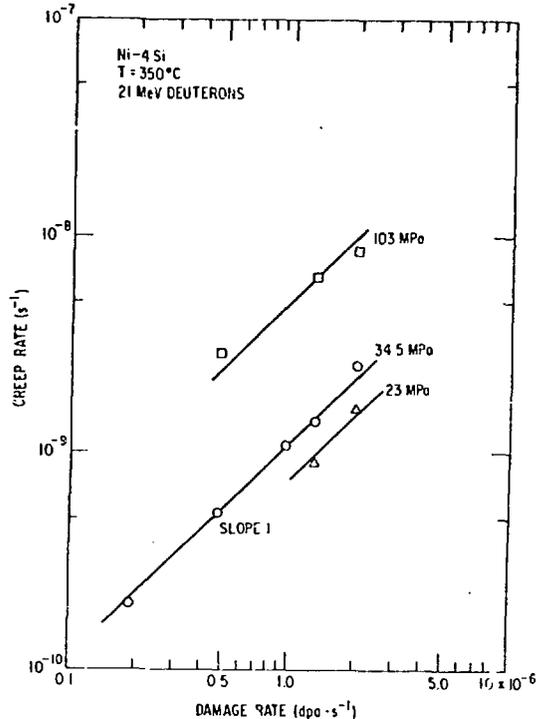


Fig. 4. Damage rate dependence of the irradiation creep rate (irradiation-enhanced component) for Ni-4 Si at 23 MPa, 34.5 MPa and 103 MPa.

behavior at high stress becomes parallel to the thermal creep behavior strongly suggests that this regime is controlled by a radiation enhancement of the thermal creep mechanism. The most probable mechanism in this case is climb controlled glide.

The single experiment on Ni-12.8 Al showed a relatively small enhancement of the creep rate due to irradiation. This may be a result of the relatively high stress used in the experiment. Because irradiation creep often has a smaller stress dependence than thermal creep, the ratio of the irradiation creep rate to the thermal creep rate increases as the stress decreases, e.g. the low stress regime for Ni-4 Si in Fig. 3. The conditions chosen for the experiment on Ni-12.8 Al may correspond to the transition region of the Ni-4 Si behavior where the enhancement of the creep rate by irradiation is small. The work of Jones and Crossland [8] supports the idea of a greater effect of radiation on creep rate at lower stress. Jones and Crossland measured the creep rate in reactor of a Ni-12 at. % Al alloy with applied stresses ranging from 14 to 30 MPa. The only major

difference between their material and the material used in this study was that theirs had been aged for a longer time. They found a substantial effect of irradiation on the creep rates in the stress regime investigated; thermal creep rates were undetectable whereas, creep in reactor was easily measurable. Both this work and the Jones and Crossland data for 18.5 MPa with $4.4 \times 10^{-9} \text{dpa s}^{-1}$ give a creep rate per unit stress per unit damage rate of $2 \times 10^{-12} \text{Pa}^{-1} \text{dpa}^{-1}$. This may indicate that the radiation creep rate is linear in both stress and flux in the regime bounded by the two experiments. However, Jones and Crossland do not show linear stress and flux dependencies for their experiments, so the correlation with their data for other stresses or damage rates is not as good. For Ni-4 Si in the low stress regime, the creep rate per unit stress per unit damage rate is $3.4 \times 10^{-11} \text{Pa}^{-1} \text{dpa}^{-1}$. If the irradiation creep rate of Ni-12.8 Al has a linear stress and flux dependence below 70 MPa, then the irradiation creep vs stress curve for the precipitation hardened alloy is about a factor of 17 lower in strain rate than the low stress regime for Ni-4 Si solution hardened material. Further experiments would be required to clarify this point, but it does seem that the precipitation hardened structure is effective in reducing the irradiation creep rate.

It is difficult to make a clear comparison of the irradiation creep of different materials when they exhibit the complex stress dependence found for Ni-4 Si, because the result is dependent on the regime in which the comparison is made. If one compares the behavior of Ni, Ni-4 Si and Ni-12.8 Al at a given stress, it is found that both the thermal and irradiation creep rates are modestly decreased in the silicon alloy and strongly suppressed in the aluminum alloy. Comparison, as in Fig. 1, at stresses that give the same thermal creep rate leads to the same qualitative conclusion. A number of mechanisms may be operating to produce these effects. Solute atoms are expected to affect irradiation creep by the following mechanisms: i) Point defects may be trapped at the solute atoms thus increasing vacancy-interstitial recombination and reducing the flow of point defects to sinks where they can contribute to creep [9, 10]. ii) If the solute atom is undersized and can form a mobile solute-interstitial complex, the bias factors for the sinks can be changed because the complex has a different size mismatch or modulus defect than the free interstitial [9]. iii) Composition gradients or precipitation at defect sinks through radiation-induced segregation may modify the bias factors [10]. Any of these mechanisms or all of them simultaneously may be operating in the Ni-4 Si alloy. The effects of the γ' precipitates in the Ni-12.8 Al may be to enhance recombination of vacancies and interstitials by trapping them at the coherent particle interfaces [11] or by retarding the climb of the dislocations [12]. It is possible that the

suppression of irradiation creep in the Ni-12.8 Al results from the residual aluminum atoms in solution rather than from the precipitates, but it is difficult to see why the oversized aluminum atoms would be so much more effective than the undersized silicon atoms.

5. ACKNOWLEDGMENT

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