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Proton irradiation creep of Inconel 718 at 300°C

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Abstract

Torsional creep tests were conducted on Inconel 718 in the precipitation hardened condition under 17 MeV proton irradiation at 300°C upto a maximum dose of 0.35 dpa. The stress dependence of the irradiation creep rate was linear for the applied shear stresses which ranged from 150 to 450 MPa. The results are discussed in relation to the operating conditions of an ITER-like machine, where Inconel 718 bolts are used to mechanically attach the shielding blanket to the backplate. The irradiation creep induced stress relaxation amounted to about 30% after a dose of 0.35 dpa. © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

In the reference design of ITER, the shielding blanket modules are attached to the backplate by bolts [1]. A high level of preloading of the bolts is required to compensate for the difference in thermal expansion between the two components. The initial preload should be maintained possibly throughout the Basic Performance Phase (BPP) corresponding to a total fluence of 0.3 MWa/m² for the machine and to a total dose of 0.5 dpa for the bolts at a temperature of about 300°C. Inconel 718 in the precipitation hardened condition has been chosen as the bolt material because of its high yield strength, which is higher than 900 MPa at 300°C. Thus, the bolts may sustain high stresses without being plastically deformed. At their position in the reactor, they are exposed to a high-energy neutron flux and irradiation creep may relax the initial pre-load. The question is whether or not the bolts have to be re-tightened before the end of BPP, when the shielding blanket modules would be, in any case, replaced [2].

Most neutron irradiation creep data for Inconel 718 are classified and not available to the ITER project. Initially, the ITER design assumed a stress relaxation of about 10% after a dose of 0.5 dpa. This value results if the relaxation is calculated with a creep constant $\kappa = 10^{-6}$ (MPa dpa)⁻¹ as it has been derived, almost

universally, from in-pile tests for austenitic stainless steel alloys [3]. (Inconel 718 has an austenitic matrix). κ is defined as creep strain divided by dose and the applied stress with the underlying assumption that the irradiation creep process is linear in both stress and displacement rate. For austenitic stainless steels, there is a large irradiation creep database which indicates that, in the absence of swelling, two separate creep regimes exist, described as transient (primary) creep and steady state (secondary) creep. Primary irradiation creep is characterized by an initially high creep rate that declines with continued irradiation [3]. The creep rate, after a dose of about 0.5-1 dpa, approaches a steady state value that may be much smaller than the initial value. Therefore, the magnitude of irradiation creep induced stress relaxation expected for the transient regime cannot be calculated by assuming a creep constant of $\kappa = 10^{-6}$ (MPa $dpa)^{-1}$ that is valid for the steady state regime.

In pile neutron irradiation creep data are very costly and require a rather long testing time. For doses in the order of 0.5 dpa, light ion simulation tests may be used to study the irradiation creep behavior of a material. In comparison to in-pile tests, the experimental parameters stress, temperature and dpa-rate can be controlled more precisely. The data are collected in real time and are available at the end of the irradiation, i.e. in a few days. Among the various simulation techniques for irradiation creep, wire specimens stressed in torsion have special features. The experimental set-up is rather simple and high dose rates may be achieved at a high strain resolution [4]. A torsional system was used to measure the stress dependence of the irradiation creep rate of Inconel

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718 in the precipitation hardended condition for maximum shear stresses in the range 150–450 MPa. The doses accumulated in each of these tests were smaller than 0.07 dpa. Therefore, in order to estimate more precisely the expected irradiation creep-induced stress relaxation after a dose of 0.5 dpa, a single specimen was irradiated up to 0.35 dpa.

2. Experimental details

2.1. Experimental set-up

The irradiation creep tests were performed with the torsional creep machine installed at the Ispra cyclotron. A controlled torque is applied to a cylindrical specimen by an electromagnetic system which consists of a coil mounted inside a permanent magnet. The twist angle of the specimen resulting from an applied torque is measured by an optically coupled system. The heat generated by the interaction of the particle beam and the specimen is removed by a high purity helium gas jet directed perpendicularly to the specimen axis. Under thermal conditions, the specimens were heated to their creep temperature by a direct current. Under irradiation conditions, the heating current was reduced while keeping the specimen temperature constant. The specimens were irradiated with 17 MeV protons at a dose rate of $3.5-4.2 \times 10^{-6}$ dpa/s. The intensity of the beam, *j*, which hit the specimen was calculated from the difference in electric heating power input for beam off/on conditions at a fixed specimen temperature. The resulting magnitude of *j* was used to calculate the dpa-rate with the aid of the TRIM code [5]. The specimen temperature was measured with an infrared pyrometer.

2.2. Specimen fabrication

Light ion irradiation creep tests require the use of mini specimens for damage homogeneity and cooling reasons. The material was processed into wires of about 156 µm diameter, with three intermediate solution annealings at 1000°C in an inert atmosphere for 20 min. Thereafter, the recommended heat treatment was applied: solution annealing at 1000°C for 0.5 h in inert atmosphere, air cooling to $718 \pm 14^{\circ}$ C, hold 8 h, furnace cooling down to $621 \pm 14^{\circ}$ C, hold 18 h, and subsequent air cooling to room temperature [6]. After the heat treatment, both ends of a ca. 8-cm long wire segment were covered with a nickel layer of 1-mm thickness by an electroplating process. The central wire segment of 8 mm, the gage length over which the rotation occurs when a torque is applied to the specimen, remained unplated. The Ni-plated parts of the specimen were tightly fixed in the grips of the creep machine such that

high stresses could be applied without causing slippage of the specimens at the grip position.

3. Results and discussion

3.1. Stress dependence

The stress dependency of the irradiation creep rate was measured for a temperaure of 300°C. The maximum shear stresses ranged from 150-450 MPa. (tensile equivalents 260-778 MPa, calculated with the relationship $\varepsilon/\sigma = \gamma/3\tau$ where ε and σ are tensile strain and stress, γ and τ shear strain and shear stress). Five specimens were used, each for one stress. Before the irradiation, the specimens were exposed to thermal creep conditions for 15-20 h in order to exhaust strain transients of thermal origin. Stress and temperature values were equal to those imposed during irradiation. Fig. 1 shows an example: the creep strain is plotted versus time for a specimen subjected to a constant maximum shear stress of 350 MPa first under thermal, thereafter under irradiation conditions (dpa-rate = 3.5×10^{-6} (dpa/s)) at a temperature of 300°C. At the end of the thermal creep period, the creep rate was almost zero and increased significantly as soon as the specimens were exposed to irradiation. At the beginning of the irradiation, there was a small strain transient during which the creep rates decreased before reaching approximately constant values after a dose of 0.01 dpa. However, a small negative curvature remained also at higher doses.

In Fig. 2, the quantity s = creep rate divided by dparate is plotted versus the applied maximum shear stress, τ_{max} , where the creep rates correspond to the mean slope of the irradiation creep curves in the dose interval 0.02– 0.07 dpa. Fig. 2 shows that the creep rate is a linear



Fig. 1. Creep under thermal and irradiation conditions at 300°C for a maximum shear stress of 350 MPa.



Fig. 2. Stress dependence of the irradiation creep rate.

function of stress in the whole stress interval 150-450 MPa, in contrast to results of torsional creep tests conducted on 20% cold worked (cw) 316 stainless steel samples under light ion irradiation at 300°C. For this material, s was a linear function of τ_{max} up to a limiting stress of about 150 MPa, for higher stresses s grew faster than linear, in a first approximation, guadratic [7]. A similar observation was made for 20% cw 316 stainless steel samples tested in tension under proton irradiation at 300°C [8]. This difference in stress dependence for Inconel 718 and cw type 316 stainless steel indicates that, at high stresses, the underlying creep mechanisms may be different for the two materials. A linear stress dependence of the irradiation creep rate is attributed to a SIPA mechanism (stress induced preferential absorption), whereas a quadratic stress dependence may be attributed to a climb and glide of dislocations [9]. It is plausible that the latter process is suppressed to a great extent in a precipitation hardened material containing a high number of obstacles for dislocation glide. Thus, the stress dependence of the irradiation creep rate stays linear.

3.2. Dose dependence of the irradiation creep rate

The irradiation creep curves measured on Inconel samples at 300°C for doses up to 0.07 dpa had a slight negative curvature. In order to examine whether or not this trend continues for higher doses, a single specimen was irradiated in three runs up to a total dose of 0.35 dpa. The dose rate was 4.2×10^{-6} (dpa s⁻¹). Between the irradiation runs the specimen was kept at room temperature at the same max. shear stress of 165 MPa as maintained during irradiation. This procedure was chosen in order to 'freeze in' the microstructure developed during the irradiation. The shear strain stayed almost constant between the irradiation runs. Fig. 3 shows the shear strain accumulated during the three irradiation



Fig. 3. The irradiation creep strain is plotted as a function of dose for a specimen irradiated in three runs. The creep rate decreased with dose.

periods, distinguished by different symbols, as a function of dose. A continuous drop in creep rate is evident. After a dose of 0.3 dpa, the creep rate has decreased to about 30% of the value determined at 0.05 dpa.

3.3. Irradiation creep induced stress relaxation

Irradiation creep induced stress relaxation will affect the stress/strain condition of the pre-stressed bolts and the surrounding material, and, in addition, the fatigue life of the bolts. An ITER type reactor will have a cyclic operation. A stress relaxation increases the stress range in the fatigue cycle and, thus, reduces the fatigue life. The irradiation creep curve shown in Fig. 3, was converted into a stress relaxation curve: if the total strain is kept constant, the stress can change only if the elastic strain is converted into plastic strain: $\Delta \tau = -G\Delta \gamma_c$. $\Delta \tau$ is the shear stress relaxation which corresponds to the creep strain $\Delta \gamma_c$, where G is the shear modulus (G = 70 GPa for Inconel at 300°C). In Fig. 4, the resulting stress relaxation, τ/τ_0 , is plotted as a function of dose, where τ is the actual and τ_0 the initial shear stress. The dashed line is an extrapolation of the measured stress relaxation up to a dose of 1 dpa with the assumption that the creep rate stays constant at the value measured at a dose of 0.35 dpa. Fig. 4 shows that the stress relaxation is fast at the beginning of the irradiation approaching a saturation at higher doses. After a dose of 0.5 dpa, the extrapolated stress relaxation amounts to about $40 \pm 20\%$ assuming an error of 50%. The hollow circle represents a bend stress relaxation test result after in pile irradiation at 315°C ([10], assuming 6.25×10^{20} n/cm² correspond to 1 dpa). The data indicate that the irradiation creep induced stress relaxation after a dose of 0.5 dpa may be higher than 17%, assumed as an upper limit according to



Fig. 4. Stress relaxation for Inconel 718. The hollow circle represents bend stress relaxation measured after in-pile irradiation at 315° C [10].

the ITER design. However, the magnitude of primary creep may depend on stress state and irradiation conditions [3]. In addition, non-creep related phenomena such as radiation induced densification of the material may affect the strain measurement depending on the stress mode applied. For example: an irradiation induced reduction in volume, as it has been observed for Inconel 718 [11], would have the opposite effect for strain measurements in torsion and tension at constant loading conditions: in torsion, the angle of twist would increase, in tension, the specimen length would decrease, thus, masking, at least partly, the irradiation creep strain.

4. Conclusions

In the reference design of ITER, the shielding blanket modules are attached to the backplate by bolts. Inconel 718 in the precipitation hardened condition has been chosen as the bolt material. In order to estimate the magnitude of the irradiation creep induced stress relaxation, proton irradiation creep tests have been conducted in torsion on this material at a temperature of 300°C for shear stresses in the range 150–450 MPa, (tensile equivalent 260–778 MPa). The tests show that:

- 1. there is a linear stress dependence of the irradiation creep rate for the whole stress range applied;
- the irradiation creep rate continuously decreased with dose in the range 0–0.35 dpa;
- 3. the irradiation creep induced stress relaxation may be higher than 17%, considered as an upper limit in the ITER design after a dose of 0.5 dpa.

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