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# In-reactor creep rupture of 20% cold-worked AISI 316 stainless steel

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Results of an experiment designed to measure in-reactor stress-to-rupture properties of 20% cold-worked AISI 316 stainless steel are reported. The in-reactor rupture data are compared with postirradiation and unirradiated test results. In-reactor rupture lives were found to exceed rupture predictions of postirradiation tests. This longer in-reactor rupture life is attributed to dynamic point defect generation which is absent during post-irradiation testing. The in-reactor stress-to-rupture properties are shown to be equal to or greater than the unirradiated material stress-to-rupture properties for times up to 7000 h.

## 1. Introduction

A primary concern of designers is to guard against component failure or rupture. Since early observations of irradiation-induced embrittlement, [1, 2] numerous programmes have been initiated to investigate stress-to-rupture properties of irradiated materials. Most of these studies concentrate on obtaining stress rupture data by post-irradiation testing of previously irradiated material, i.e. material is irradiated in the unstressed condition, then removed from the reactor and tested. Only limited data from in-reactor rupture testing have been reported [3, 4].

Postirradiation test results on various alloy compositions (AISI 304, AISI 316 and Hastalloy X [4–22]) have demonstrated that irradiation in both thermal and fast reactor energy spectrums result in a loss of material rupture life and ductility. These losses in ductility and rupture life have been attributed to helium embrittlement enhanced by hardening of the matrix during irradiation. A few exceptions to loss of rupture life during postirradiation testing have been documented [23, 24]. However, reduced ductilities were still observed.

Prior to this experiment, in-reactor rupture predictions have been based on postirradiation test results and application of a life or damage fraction rule [25–27]. However, the validity of relating postirradiation ruptures to in-reactor failures is based on several assumptions. The assumptions are: (1) the irradiation-produced microstructure does not recover during a test (irradiation damage is frozen in the microstructure) and (2) irradiation damage and deformation damage are commutative processes (irradiation during deformation does not alter the micro-structural processes responsible for deformation and ultimate failure).

During analysis of thermal and in-reactor creep of 20% cold-worked AISI 316 stainless steel (316 CW) it was realized that deformation processes, especially at the higher temperatures, were influenced by the tendency of the material to recover [28, 29]. Results of in-reactor tests [29] and theoretical modelling [30–32] also suggested that irradiation dynamically affects the deformation process. These observations indicated a need to evaluate stress to rupture, not only through postirradiation test methods, but also during irradiation.

Therefore, a programme was initiated to obtain in-reactor failures and high temperature creep measurements on 316 CW in Experimental Breeder Reactor II (EBR-II). The results reported here include observations from the first four interim examinations of this continuing experiment.

## 2. Test procedures

The stress rupture specimen consists of a pressurized piece of tubing, 2.82 cm long with welded end caps. The main advantage of this pressurized


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tube specimen is its size. A great variety of stress and temperature conditions can be simultaneously tested in a limited amount of reactor space. Testing of unirradiated tubular specimens of various lengths indicate short lengths of tubes can be used to obtain creep and creep rupture data without interference due to end constraints. Length to diameter ratios as low as 4 have been successfully used to determine irradiated and unirradiated creep and creep rupture properties of a wide range of materials [33, 34].

After the end caps were electron-beam welded to the tube, the specimen was placed in a chamber for pressurization. Desired stress levels were obtained by pressurizing the specimens to a predetermined level based on specimen geometry and target irradiation temperature. Stresses were calculated using the method of Gilbert and Blackburn [28]. Gas entered the specimen through a small hole in one end cap and this hole was sealed with a laser welder. A high-purity Ar–2% He gas mixture was used to pressurize the specimen. Specimen weights were measured before and after pressurization to ensure the proper volume of gas had been sealed in the sample. Weight change was also used to determine specimen rupture. A decrease in weight is proportional to the amount of gas lost. The He in the gas mixture allowed mass spectrometer vacuum leak detection with sensitivities greater than $1 \times 10^{-8}$ $cm^3$ $sec^{-1}$.

As an additional check on proper pressurization, the diameter of the specimen was measured before and after filling. This allowed the dimensional change due to elastic expansion of the tubing caused by pressurization to be monitored. Diameters were measured by a laser interferometer measurement machine [35] which accurately measured diameter changes as small as 0.01%. After pressurization and measurement, the specimens were loaded into an instrumented sub-assembly for irradiation in EBR-II.

The instrumented sub-assembly consisted of three canisters operating at nominal temperatures of 590, 650 and 700° C. The temperature in each canister was maintained by a balance between the nuclear heat generation within each canister and heat transfer through a gas annulus to flowing sodium coolant. Temperature was controlled by adjustment of an inlet flow valve which varied the flow rate of sodium past the canisters. Sample temperatures were measured every 10 sec by six thermocouples which were positioned axially

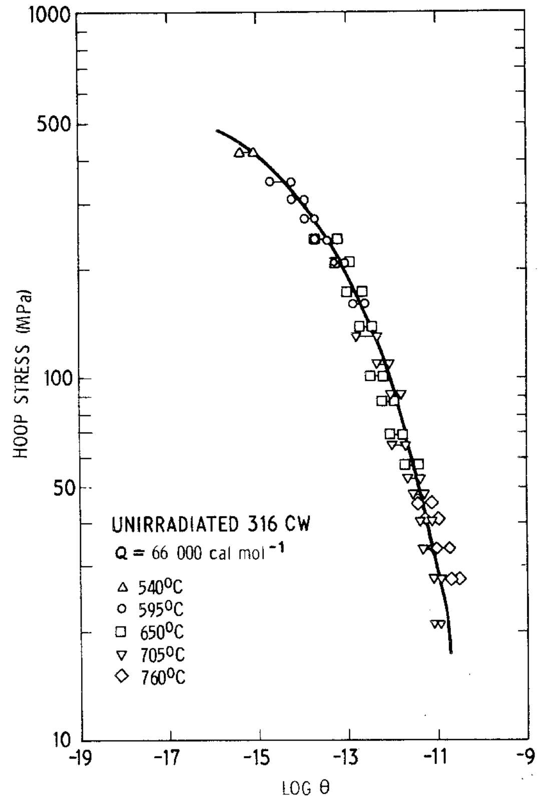


*Figure 1* Sherby–Dorn temperature compensated rupture time plot for unirradiated 316 CW.

along the length of the experiment. Periodic adjustment of the valve was made to maintain a constant irradiation temperature.

At specified time intervals, the specimens were removed from the reactor for diameter and length change measurements. During these interim examination periods, the specimens were reweighed to determine whether the gas had leaked out and the diameter profile examined for specimen failure. In this manner a range of rupture time was determined for each specimen.

In addition to the in-reactor specimens, a thermal control pressurized tube matrix was thermally soaked at 5 temperatures (540, 595, 650, 705 and 760° C) for time intervals of 1, 10, 30, 100, 300, 600, 1000, 2000, 3000, 5400 and 7800 h. At each time interval, diameters were measured to determine creep and creep rupture information.

## 3. Experimental results

The unirradiated stress-to-rupture data is plotted in Fig. 1 using the Sherby–Dorn temperature compensated rupture time parameters [36]

$$\theta = t_r \exp(-Q/RT),$$

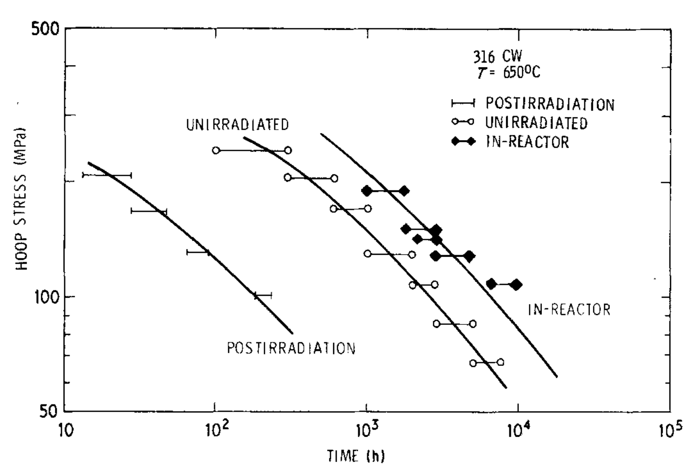


*Figure 2* Comparison of the rupture behaviour of postirradiation, unirradiated, and in-reactor tested specimens at 650° C.

where $t_r$ is the rupture time in hours, $Q$ is the activation energy, $R$ is the gas constant and, $T$ is the temperature in K.

By using an activation energy of 66 kcal $mol^{-1}$, the different temperature data coalesced to a single line. The wide range of stresses, temperatures and times produced an accurate description of the unirradiated time-to-rupture properties of 316 CW.

In Fig. 2, the unirradiated thermal prediction is compared with postirradiation [37] and in-reactor stress-to-rupture data at 650° C. As expected, the postirradiation test data indicates reduced rupture times of a factor of approximately 10. However, in-reactor stress-to-rupture life is shown to be equal to or greater than the stress-to-rupture life of the unirradiated material. These results demonstrate that irradiation during deformation has a large effect on stress-to-rupture properties. This is more dramatically illustrated in Fig. 3 for the full in-reactor data set encompassing temperatures, 560 to 760° C, and accumulated fluences as high as $4.5 \times 10^{22}$ neutrons $cm^{-2}$.

Although the rupture life of 316 CW is approximately the same for unirradiated and in-reactor specimens, Fig. 4 demonstrates reduced ductility of the in-reactor tested material. In-reactor ductilities one half of the unirradiated material ductilities were observed. This reduction in irradiated material ductility has been extensively investigated in postirradiation tested materials. A number of investigations [38, 39] have shown that embrittlement can be associated with He produced by $^{10}B(n, \alpha)^7Li$ reactions with neutrons. This He is insoluble in austenite and often preferentially precipitates as bubbles at grain boundaries. Waddington and Lofthouse [40, 41] and Gittins [42] have shown that the growth of He bubbles in irradiated specimens leads to accelerated crack propagation once a crack has been nucleated. Similar behaviour caused by precipitation of He is expected to occur in in-reactor tested materials.

Both unirradiated and irradiated material ductility increases with decreasing stress at constant temperature. To investigate this increasing ductility

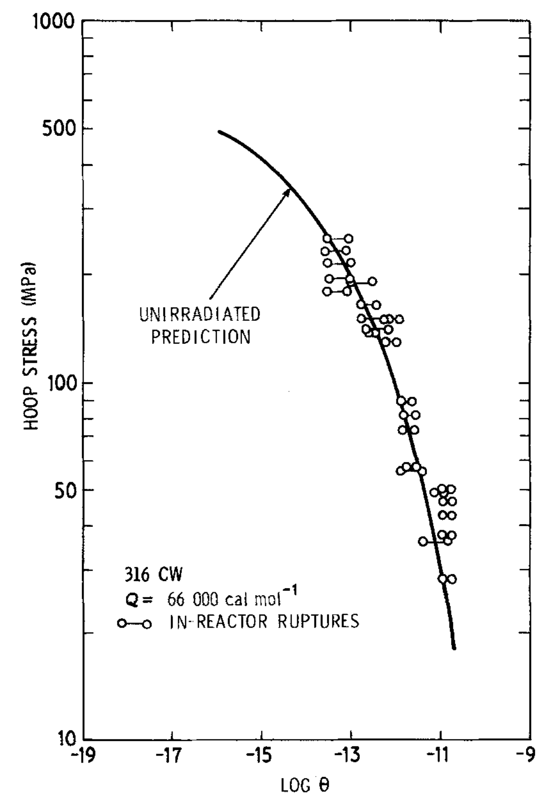


*Figure 3* Comparison of unirradiated and in-reactor ruptures on a Sherby–Dorn temperature compensated rupture time plot.

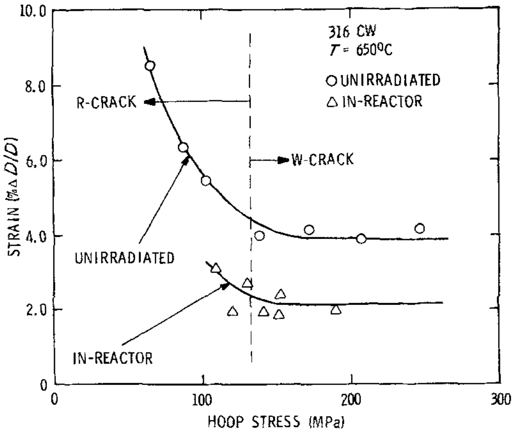


*Figure 4* Decreased ductility of in-reactor tested material. Both in-reactor and unirradiated ruptures show increasing ductility with decreasing stress.

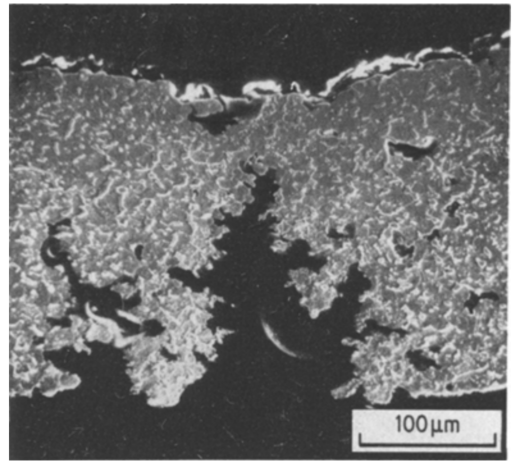


*Figure 6* Failure of a pressurized tube by r-crack linking. This type of failure was typical of low strain rate ruptures.

with decreasing stress, the failed unirradiated samples were metallographically examined. Examination revealed two classical types of failure cracks: w-cracks (triple-point wedge cracks, see Fig. 5) and r-cracks (spherical voids at grain boundary surfaces, see Fig. 6). In accordance with Garafalo [43] the w-cracks were found predominantly in the high strain rate (high stress–low temperature) ruptures while r-cracks were found in low strain rate (high temperature–low stress) ruptures. The transition from predominantly w-cracks to r-cracks at constant temperature was marked by a large increase in strain (Fig. 4). This increase in strain is caused by transition from a fast wedge crack propagation region to a slower diffusion-controlled growth of voids and cavities failure mechanism. Fig. 7, which was generated from postirradiation examination of 25 specimens, maps this transition as a function of stress and temperature. For decreasing temperature, the hoop stress at which r-cracks dominate increases. This is the result of irradiation creep and swelling processes which dominate the deformation behaviour at low temperatures.

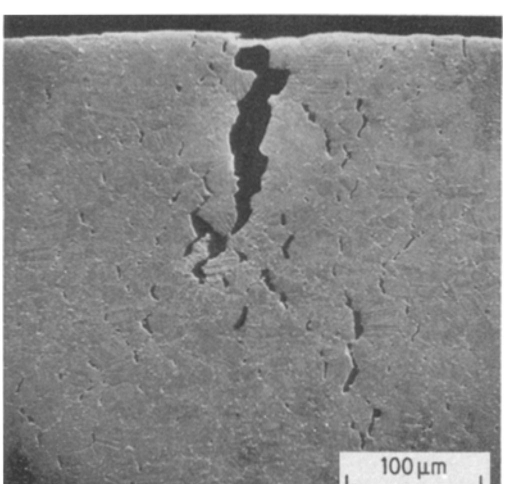


*Figure 5* Failure of a pressurized tube by w-crack propagation. This type of failure was typical of high strain rate ruptures.

Since the ductility of the in-reactor material was less than the ductility of unirradiated material, the relatively unaffected rupture time of the in-reactor tested material must lie in the creep-rate behaviour. Comparison of the unirradiated, post-irradiation and in-reactor creep curves reveal the reason for the observed difference in stress to rupture lives. Fig. 8 shows there is little difference for temperatures above 620° C in the primary and

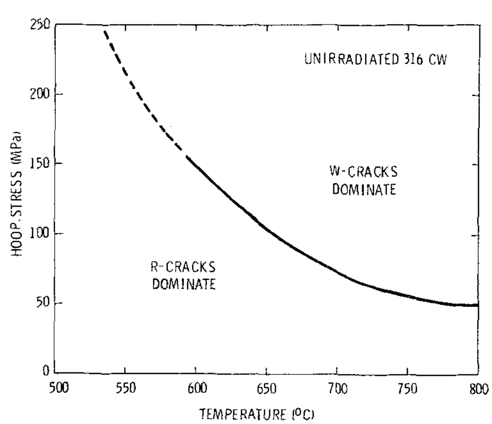


*Figure 7* Transition from predominately w-crack failure to r-crack failure as a function of stress and temperature.

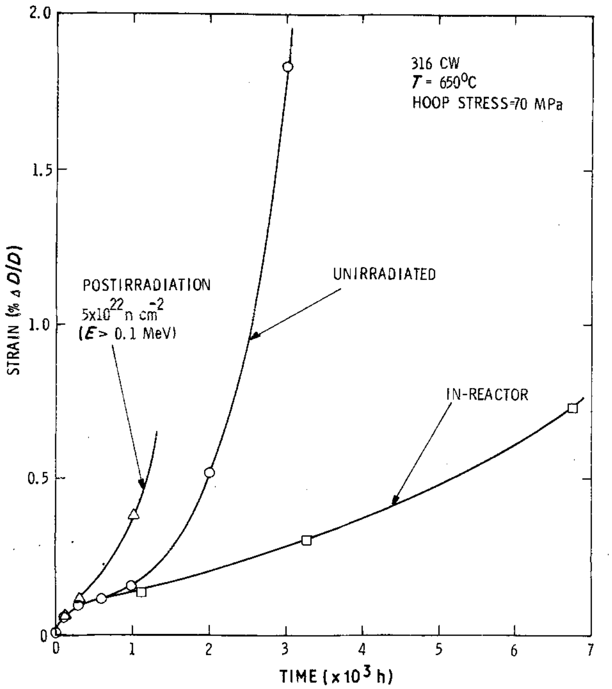


*Figure 8* Comparison of strain-time curves for postirradiation, unirradiated and in-reactor tested specimens. Tertiary creep is delayed in the in-reactor tested material.

secondary creep rates of the unirradiated and in-reactor tested material. Only the duration of the secondary or steady-state creep regime is different. Although the in-reactor tested material has reduced ductility, irradiation during deformation delays the onset of tertiary creep resulting in a stress rupture life equal to or greater than the unirradiated properties. This retardation of tertiary creep (Figs 8 and 9) has previously been observed by Gilbert and Lovell [44] for temperatures above 620° C.

## 4. Discussion

Several mechanisms or combinations of mechanisms may explain this retardation of tertiary creep by irradiation during deformation. Lovell has suggested that the initial stages of tertiary creep are caused by recovery of the cold work structure. Based on this assumption, Gilbert and Lovell [44] have hypothesized that the retardation arises from short-lived obstacles that are generated during irradiation. They have coined the phrase "dynamic hardening" to describe the process. This dynamic

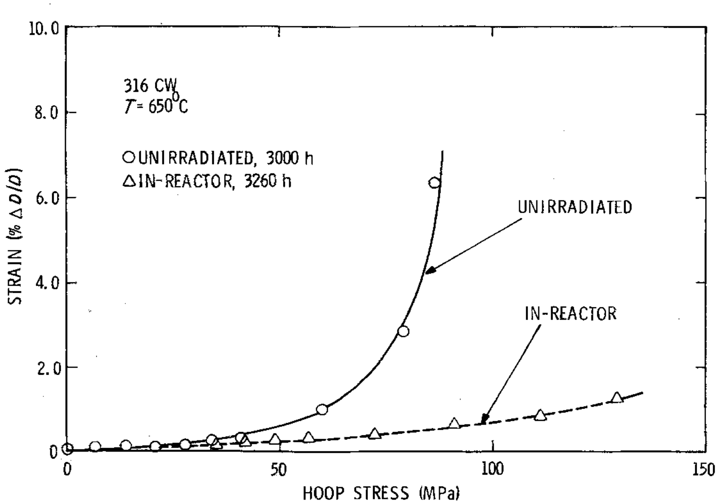


*Figure 9* Delay of the onset of tertiary creep observed in in-reactor tested material.

hardening caused by obstacles such as vacancy clusters, small interstitial loops and irradiation-produced jogs in dislocation lines counter recovery effects responsible for the onset of tertiary creep. Because these obstacles are unstable at high temperatures and quickly anneal-out prior to post-irradiation testing, the onset of tertiary creep is not delayed in postirradiation tests.

An alternate mechanism is the delay of w- and r-crack nucleation and growth by irradiation. Nemy and Rhines [45] have shown in Al–2.6% Mg that the inception of tertiary creep can be ascribed to nucleation of intergranular cracks and/or necking. If the onset of tertiary creep in 316 CW occurs concurrently with the nucleation of cracks, then irradiation-induced creep may plastically relax stresses at w-crack tips, delaying the onset of tertiary creep and failure. Alternately for r-crack formation, Mancuso, *et al.* [46] have suggested that irradiation, indeed point defect annihilation, can control reaction rates at sinks and reduce the rate of cavity growth. This mechanism again requires irradiation during deformation for the delay of tertiary creep onset which is consistent with the absence of this phenomena in postirradiation tested materials.

A third probable mechanism for the delay of tertiary creep is irradiation-induced precipitation. Garafalo [43] and Davies and Evans [47] have shown in austenitic stainless steel and Nimonic 80A that grain-boundary precipitate structures can delay the onset of tertiary creep. Irradiation may similarly produce an altered precipitate morphology which may lead to a longer secondary creep region. Irradiation-induced precipitation in 316CW has been reported by several investigators [48–50]. For this mechanism to be consistent with post-irradiation tests, where no delay in tertiary creep is displayed, the irradiation-induced precipitate must be stable and quickly disappear prior to postirradiation testing.

It is highly probable that a combination of the above mechanisms is responsible for the delay by irradiation of the onset of tertiary creep. Regardless of the exact mechanism operating, irradiation during deformation is responsible for the enhanced rupture life of in-reactor tested materials over postirradiation tested materials. This result indicates that the use of postirradiation test data in conjunction with a life or damage fraction rule is an invalid method of predicting in-reactor failures.

## 5. Conclusions

The following conclusions resulted from this work.

(1) In-reactor rupture life of 316 CW stainless steel is greater than or equal to the unirradiated material stress to rupture properties for times up to 7000 h.

(2) Ductilities of one half that of the unirradiated material can be expected for in-reactor tests.

(3) Enhanced in-reactor rupture life results from delay of the onset of tertiary creep.

(4) Retardation of the tertiary creep may be caused by a combination of dynamic irradiation hardening, delayed crack nucleation and growth, or irradiation-induced precipitation.

(5) Significant recovery during slow rate post-irradiation testing, combined with dynamic effects of irradiation during deformation, suggest slow-rate postirradiation testing is an invalid method of predicting in-reactor failures.

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