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A REVIEW OF RECENT IRRADIATION-CREEP RESULTS

by

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TABLE OF CONTENTS

	PAGE
ABSTRACT.	1
INTRODUCTION.	1
IRRADIATION CREEP USING STRESS RELAXATION METHODS	3
CREEP MEASUREMENTS USING PRESSURIZED TUBES.	6
IRRADIATION CREEP UNDER CONSTANT APPLIED LOAD	14
IRRADIATION CREEP EXPERIMENTS USING ACCELERATED PARTICLES	20
SUMMARY OF IMPORTANT RESULTS AND SUGGESTED AREAS FOR RESEARCH .	27
ACKNOWLEDGMENTS	28
REFERENCES.	29

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W. A. Coghlan

ABSTRACT

Materials deform faster under stress in the presence of irradiation by a process known as irradiation creep. This phenomenon is important to reactor design and has been the subject of a large number of experimental and theoretical investigations. The purpose of this work is to review the recent experimental results to obtain a summary of these results and to determine those research areas that require additional information. The investigations have been classified into four subgroups based on the different experimental methods used. These four are: (1) irradiation creep using stress relaxation methods, (2) creep measurements using pressurized tubes, (3) irradiation creep from constant applied load, and (4) irradiation creep experiments using accelerated particles. The similarity and the differences of the results from these methods are discussed and a summary of important results and suggested areas for research is presented. In brief, the important results relate to the dependence of creep on swelling, temperature, stress state and alloying additions. In each of these areas new results have been presented and new questions have arisen which require further research to answer.

1. INTRODUCTION

Irradiation creep is radiation induced plastic deformation of a material. During the last decade or so, there has been considerable effort to measure and understand this phenomenon. Creep rates of materials important to fission reactor designers have been measured as a function of applied stress, temperature, and irradiation fluence. As we shall see, these studies have focused on a few austenitic stainless steels and some zirconium alloys. The purpose of this report is to review and summarize experimental results with emphasis on work published since 1977. Prior to this date, several reviews had been published.

In 1972, Hesker¹ published a broad view of the early irradiation creep results. He included data describing stress, temperature, and fluence dependencies for a broad range of cubic, noncubic, fissile, and nonfissile materials. In addition, he reviewed the theoretical explanation offered at that time. There have been a number of more recent reviews and I call your attention to three of these. Gittus² included a long chapter on irradiation creep in his book. This material includes a discussion of the theoretical models available at that time. In 1977, Straalsund³ collected the available data measured for irradiation creep in stainless steels, the material chosen to be the structural material for breeder reactors. Finally, I refer you to the review of irradiation creep published by Harries⁴ in 1977. In this paper the various methods for measuring irradiation creep are described along with a discussion of the advantages and disadvantages of each. Also Harries has related the experimental measurements to theoretical explanations in an attempt to provide support for or to discredit each of the models. He also presented a list of suggested experiments to answer some of the remaining unanswered questions.

The measurement of irradiation creep has not been easy. At temperatures and stresses where thermal creep rates are very low and irradiation creep can be measured independently, the irradiation creep rates are low and difficult to measure. The radiation environment required for the study is a very hostile environment for materials; and, as a result, it is difficult to design testing machines which will operate reliably and accurately enough to make the measurements. As a result of these difficulties, there have been a wide variety of experimental techniques developed to measure irradiation creep. Each of these has its own advantages and disadvantages, and each has provided useful information. A difficulty develops, however, when results from one technique are compared with results measured using another technique. In some cases these comparisons can be made on reasonable theoretical grounds while in others no direct comparison is possible.

In the following overview, the recent results have been divided into four groups based on the experimental technique used to irradiate the sample and to apply the stress. Data is discussed under the titles — Irradiation Creep Using Stress Relaxation Methods; Creep Measurements Using Pressurized Tubes; Irradiation Creep From Constant Applied Load; and Irradiation Creep Experiments Using Accelerated Particles. In the course of preparing this review, I have attempted to determine what the most important findings have been and what the most important questions left unanswered are. The report will close with a summary of this information.

2. IRRADIATION CREEP USING STRESS RELAXATION METHODS

Various stress relaxation techniques for measuring irradiation creep have been used since irradiation creep was first discovered. The many forms of this technique have several characteristics in common. The samples are loaded to some initial stress and are placed in the reactor environment. The relaxation of this stress occurs through deformation of the sample, and this deformation is observed by measuring the remaining stress in the samples after intervals of irradiation. The continuing relaxation of the stress results from continuing creep strain.

The method is relatively simple, utilizes small samples, and requires minimum experimental sophistication. However, it is not without problems. The most obvious and limiting one is that the strain is limited to an elastic deflection (i.e., strains from about 10^{-6} to 10^{-3}). These small strains often occur during primary creep, and the experimental results are often only measurements of this primary creep. The effect of irradiation on this creep can be obtained by comparison of in-reactor relaxations with out-of-reactor results. From such experiments, it is easy to show qualitatively that irradiation enhances creep but quantitative description is difficult.

Over the last 15 years, several investigators have analyzed stress relaxation experiments. In 1977, Povolo⁵ published an analysis of stress relaxation through creep. To describe the strain rate, he chose

$$\dot{\epsilon} = K(\sigma - \sigma_z)^n f(t) ,$$

where the time dependent primary creep is included in $f(t)$ which is often assumed to be proportional to t^m . Most early results can be derived as special cases of this general one. Povolo showed how the material constants, K , n , and m , can be obtained from analysis of experimental stress relaxation data for tensile, bending, and torsion stress relaxations. There are two obvious limitations, however. First, for this analysis enough data must be taken to evaluate the time derivatives of the relaxed stress. Second, for $f(t) \neq t^m$ more information about the time dependence of primary creep must be known to extract the other creep parameters. In 1978, Povolo and Toscano⁶ derived similar results assuming strain hardening instead of time hardening. Recently,⁷ this last analysis was shown to give a much better fit than the time hardening analysis for thermal relaxation of stress-relieved and cold-worked Zircaloy-4 at 400°C. In spite of these difficulties, a number of recent investigations have been conducted which have provided useful information.

Kenfield et al.⁸ irradiated 78 specimens of 20% CW 316 stainless steel in EBR-II at 370°C. The samples were stressed in four-point bending from 28–345 MPa and were examined after irradiation to 2×10^{25} n/m². Sometime during this exposure, strain occurred which caused a relaxation of from 40–60% of the applied stress. The relaxation strain under irradiation is about four times that measured for a thermal stress relaxation. The results were compared to some predicted by a low fluence design equation, and the measured creep was somewhat less than the predicted creep.

Stress relaxation of loaded springs has been used to measure the irradiation creep of Nimonic 80 (ref. 9) and Inconel X750 (ref. 10). After an irradiation period, the remaining stress on the springs can be measured by deforming the spring slightly and measuring the load required. A significant difference in this method from the bent beam is that the applied stress is nearly pure shear and is uniform across the sample cross section. The results are reported for a very limited

range in temperature and stress, but they show that these materials creep at much the same rate as stainless steel.

In all the results described above, the total strain available to the experiment is small, and so it is not possible to measure irradiation creep at high fluences and high strain rates. A group of investigators¹¹⁻¹⁴ at the Hanford Engineering Development Laboratory (HEDL) have devised two stress-relaxation techniques to overcome this limitation based on the idea of using preirradiated material to make stress-relaxation specimens. In the first method, strips of irradiated type 304 stainless steel duct material were loaded by wrapping them around a mandrel. After re-irradiation, the creep strain was determined by measuring the curvature of unloaded strips. In the second method, a slit was cut in sections of irradiated pressurized tube samples of 20% CW 316 stainless steel. The samples were then loaded elastically by inserting wedges in the slits. The stress on the samples was determined at intervals by measuring the force required to open the slit which decreases as creep causes the elastic stress to relax.

The chief advantage of the mandrel loading technique is that it makes it possible to study irradiation creep in samples of material cut from structural parts having a high irradiation dose. Creep measurements have been reported on type 304 stainless steel^{11,13} samples having nearly the highest neutron exposure of any material. The creep rate appears to have a component that is proportional to the swelling rate of the material. After a neutron exposure of 5×10^{26} n/m², both the swelling rate and the creep rate increased sharply.

The split tube technique makes it possible to study continued irradiation of pressurized tube specimens under a variety of different irradiation conditions. The results reported for continued creep under a change in temperature¹² and under a change in applied stress¹⁴ are of particular importance to fusion reactor design. A temperature change of 40-100°C appears to have very little effect on the measured creep rate of 20% CW AISI 316. The samples continue to creep at much the same rate as they had at the original irradiation temperatures. A change in applied stress, on the other hand, appears to cause immediate transient creep that is independent of prior stress history.

This transient strain involves primarily thermal processes because it does not occur if the samples are annealed for 50 h at the new stress in the absence of irradiation.

Recently, Causey and others¹⁵ have reported some in-reactor stress relaxation results that may also be of interest to fusion reactor designers. They reported in-reactor stress relaxation of bent beams of 11 different materials at 67 and 300°C. In all but one of the cases reported, the creep rate at 67°C was greater than-or-equal-to that at 300°C. These results are consistent with some other early results but are not readily explained using any of the known models.

3. CREEP MEASUREMENTS USING PRESSURIZED TUBES

A second general method for measuring irradiation creep is the measurements of the increase in diameter of pressurized tubes under irradiation. This method has the advantage that the specimens are relatively small and simple to construct. The loading is biaxial and often reflects the actual loading expected for the materials in service. However, this biaxial loading is also a difficulty because it makes it difficult to compare the results with those measured in standard tensile tests.

In his review, Harries⁴ discusses the problems associated with directly applying the Soderberg formalism to convert the results from all types of tests to effective stress and strain. The chief complication arises because of an apparent relationship between swelling and creep. If the sample does not maintain constant volume during creep, straightforward application of the Soderberg formalism is in error. Since the relationship between creep and swelling is the subject of current research, comparison of biaxial, uniaxial, and shear creep results should be regarded with caution. With these advantages and disadvantages in mind, I will now present a summary of the most recent published results.

Walters and co-workers¹⁶⁻²⁰ have analyzed the creep data from 130 pressurized tubes which were irradiated in four subassemblies in EBR-II. The first subassembly was composed of 19 tubes made from solution

annealed 304L stainless steel capsules irradiated at 385°C with the pressure generated hoop stresses from 0-191 MPa. The remaining three subassemblies contained 111 tubes made from 316 stainless steel in several different metallurgical conditions. Two of these subassemblies were irradiated at 400°C and contained helium pressure to generate hoop stresses from 0-345 MPa. The third was irradiated at 550°C with slightly lower stresses. Most of the 316 stainless tubes were in three different metallurgical conditions - 20% cold-worked, solution annealed, and capsules aged for 216 h at 704°C. This last condition, called HTD by the authors, results in the precipitation of large metal carbide particles which causes a large change in swelling and creep behavior. In addition to these tubes, analysis was made of some stainless steel fuel cladding that was irradiated in ERR-II and Rhapsodie at temperatures from 400-600°C.

The analysis of all these samples was directed toward answering questions in two main areas. First, an attempt was made to measure the stress dependence on swelling and to determine the relationship between swelling and creep.¹⁶⁻¹⁸ Second, the authors tried to determine the role that metal carbide precipitation plays in enhancing swelling and creep in these materials.^{19,20} Although we shall see that the questions are not completely answered, several valuable observations were made.

The 304L stainless steel capsules were irradiated the longest; hence, the information is known to higher doses. In addition, the tubes in this experiment are long enough to sample a neutron flux variation of a factor of 60 and to experience a variation in average neutron energy from 0.077 to 0.61 MeV.¹⁶ No effect on deformation, either creep or swelling, was measured for these variations in flux and energy. However, both the creep rate and swelling rate increased with fluence. The stress dependence of the creep for stresses up to 191 MPa is linear, and at the highest stress the total deformation is approximately one third swelling and two thirds creep. Figure 1 has been replotted from Ref. 18 and shows the total deformation at 191 MPa along with the deformation measured in an unpressurized tube. The dashed curve is the irradiation creep determined by

ORNL - DWG 81-12118

Fig. 1. Creep, swelling and total diameter changes as a function of neutron fluence for type 304L stainless steel capsules.¹⁸

subtraction of the swelling strain from the total and assuming that swelling is independent of stress. Destruction of a stress free capsule and also one stressed at 157 MPa for immersion density measurements^{17,18} showed no effect of applied stress on swelling for capsules irradiated at 400°C. This result is not universal, however, since comparison of the swelling in fuel cladding with that in unstressed samples at 530°C shows that the cladding, which is stressed, swells much faster. The value of the cladding stress can be measured by determining the residual stress after irradiation by measuring the displacement of a slit cut in the tube. This measurement raised another question, however, because the values measured were smaller than expected. This disagreement suggests that swelling under this stress may be faster, or perhaps there is a chemical change in the cladding that leads to higher swelling which relaxes the internal stress. This question was not resolved by the authors.

One interesting comment was made in the discussion of the residual stress measurements, however. Transmission electron microscopy showed that a swelling gradient as measured by void size and number density did not occur. The authors correctly point out that the measurement of voids does not necessarily measure swelling. Swelling occurs in the region where the excess interstitials precipitate. It appears that if the interstitials diffuse along the stress gradient, the result will be stress enhanced swelling. If the excess interstitials move along the stress gradient by climb and glide of dislocations, the resulting creep relaxes the stress. These effects would be very difficult to distinguish from each other.

As the reader can see from above, the important question relating applied stress and swelling has not been answered. Recently, Garner et al.²¹ collected the available data and attempted to rationalize the contradictory information. Briefly, they concluded that more than one mechanism is involved and much more work understanding stress-affected precipitation and chemical evolution is needed before the link between stress and swelling can be resolved.

Results from the 316 stainless steel studies add another complication to the problem.¹⁶ At 400°C the results show very similar stress and temperature variation to that reported for 304L stainless steel; but, in general, the deformation rates are less for 316 stainless. One exception to this similarity, shown in Fig. 2, is that the 20% cold-worked and solution annealed samples shrink for the first 10 dpa of irradiation as a result of precipitation. Also analysis of the swelling shows that the HTD samples begin to swell immediately while the 20% cold-worked and solution annealed samples initially shrink. Then, after 10 dpa the solution annealed samples begin to swell rapidly while the cold-worked samples swell slowly to at least 40 dpa.

At 550°C the behavior of 316 stainless, shown in Fig. 3, is even more complicated with a nonlinear stress dependence and may also show a decrease in the dose required before rapid deformation begins. The analysis of the metal carbide concentration offers a strong circumstantial argument to explain these observations. The HTD samples have

ORNL-DWG 81-11667

Fig. 2. The diameter change of AISI 316 stainless steel pressurized tubes as a function of neutron dose¹⁶ at 400°C and a hoop stress of 276 MPa. (a) Aged for 216 h at 704°C and (b) combination of solution annealed and 20% cold-worked samples.

about 15 mg of precipitates per gram of sample before and after irradiation. Under irradiation at 400°C the precipitate content of the 20% cold-worked and solution-annealed samples increases from nearly none to more than 0.5 mg/g of sample, and the deformation rate increases accordingly. At 550°C, the precipitation increases to about one half of that found for the HTD materials, and the deformation rate is nearly as fast. Recently, there has been significant progress in understanding the precipitation behavior in irradiated stainless steel.²² It is likely that the amount of precipitates not only increase as a function of temperature and fluence but also change in crystal structure and metal ion content. Further research may well shed some light on the mechanisms for the deformation increases observed.

Recently, creep has been measured in samples undergoing a gradual temperature decrease.²³ In 20% cold-worked AISI 316 cladding, larger

Fig. 3. Stress normalized creep strain as a function of neutron dose for pressurized AISI 316 stainless steel tubes¹⁶ which had been aged for 216 h at 704°C; (a) creep was measured at 550°C and $\sigma_{\text{hoop}} = 207$ MPa and (b) based on data measured at 550°C with $\sigma_{\text{hoop}} = 103$ MPa and at 400°C with $\sigma_{\text{hoop}} = 276$ MPa.

creep deformations have been measured in those samples that have undergone a temperature decrease. Further analysis of these data have been presented along with a proposed model.²⁴ It is observed that γ' precipitates form during low temperature excursions. This precipitation is believed to lead to faster microchemical changes in the material.

Bergmann, et al.²⁵ irradiated pressurized tubes made from two different heats of 14% cold-worked DIN 1.4981 austenitic stainless steel. This steel is similar to AISI 316 and the results were also very similar. For irradiation temperatures from 410–480°C and hoop stresses from 0–237 MPa, the deformation begins slowly and then increases rapidly at about 20 dpa. The in-pile creep rate is proportional to stress except for the highest temperature, 480°C where a higher stress dependence is observed. No heat-to-heat differences were found for the two heats.

Creep deformation of pressured tubes made from 20% cold-worked 316 stainless steel at higher temperatures has been reported by two different groups. Gilbert and Lovell²⁶ extended the earlier measurements of Gilbert and Bates²⁷ to 720°C. The high temperature results are for low fluence and stresses that are high enough to cause significant thermal creep. From their data, creep behavior in the region where both thermal and irradiation creep occur shows complicated temperature dependence with a possible decrease in creep rate when the samples are irradiated. More recently, Wassilew et al.²⁸ conducted creep tests on 316 stainless steel samples before, during, and after irradiation at 712°C. In all their measurements the in-reactor creep rates were the highest.

Pressurized tubes have been used to study irradiation creep in other alloys. These include the relatively high temperature measurements by Paxton et al.^{29,30} of creep in a ferritic steel containing 12% Cr, three commercial austenitic stainless steels, one experimental titanium-modified austenitic stainless steel, and five precipitation hardened Fe-Ni-based alloys. In addition, recent measurements of creep in a 17% Cr ferritic steel at lower temperatures have been reported by Erler et al.³¹ Finally, the method has been used to study creep and growth of zirconium alloys.³²⁻³⁴

Measurements of creep under the same conditions for ten different alloys, described above, gives a very powerful method to compare the behavior of different materials. Paxton et al.^{29,30} measured the creep of these ten alloys in a total of 17 metallurgical conditions, under four stresses, and at two temperatures, nominally 540 and 605°C. The five precipitation hardening alloys, all of which depend on γ' and/or γ'' precipitates for hardening, are the most creep resistant and show a linear stress dependence although the exact meaning of the results are subject to interpretation. Swelling contributes to the diametral strain and is measured to be a function of stress in one of the austenitic materials, cold-worked AISI 316 at 540°C. When this swelling is removed from the total strain, the resulting creep curve is much more linear in stress. Also for another material, AISI 330, the creep rate is the same both in and out of reactor at 605°C. It is

likely, therefore, that much of the nonlinear stress dependence is a result of thermal creep.

One last complication is observed for two of the precipitation hardening materials, M-813 and PE-16. An inverse temperature dependence is measured for creep. This decrease in creep rate for an increase in temperature is accompanied by a very strong decrease in swelling which offers further support for the direct relationship between creep and swelling.

The creep and swelling of ferritic materials at lower temperatures, 400-550°C, was reported by Erler et al.³¹ The results are very simple to report in that void swelling is confined to the α -phase particles formed during some heat treatments. The creep rate that accompanied the very low swelling rate is correspondingly low. Very few measurements of creep greater than 0.2% were measured for irradiation out to 150 dpa.

Pressurized tubes of another form have been used to study creep in zirconium alloys. The tubes used for the study were the large pressure tubes used in a number of heavy water reactors. The ends of the tubes were constrained; therefore, the stress state was more complicated than a simple pressurized tube. Zirconium is an anisotropic material, and the observed strain is a combination of anisotropic growth and creep. The deformation mechanisms are complicated and depend on metallurgical variables that are difficult to control. Kolstad³⁴ has recently made a comparative evaluation of some experimental data using ten different creep correlations, and the predicted strain using these equations varied by an order of magnitude. This disparity is a good illustration of the difficulty in understanding irradiation creep in these anisotropic materials.

As an example of a method to improve understanding of creep and growth in irradiated Zircaloy tubes, we would like to mention the model developed by Holt.³² He based the model on a quantitative correlation describing both the elongation and diametral strain of Zircaloy pressure tubes which were manufactured in eight different procedures and irradiated in four power reactors. The results of the correlation show that the growth rate is approximately proportional to

dislocation density, increases in the direction of elongating grains, and also increases in the direction that has a decreasing number of basal plane normals. The creep rate, on the other hand, is insensitive to dislocation density, but is related instead to the texture of the tube. The model successfully predicts the diametral and longitudinal strain rates for tubes experiencing end loads during operation. In a later paper,³³ the model is used to define ideal textures and grain sizes for reactor pressure tubes.

4. IRRADIATION CREEP UNDER CONSTANT APPLIED LOAD

Although pressurized tubes have been useful in measuring irradiation creep data, experiments which have a simpler applied stress are often easier to interpret and compare. In addition, the study of initial strain transients or transients in load or temperature requires a fully instrumented creep machine which can record data continuously. The first of these requirements was achieved early in the study of irradiation creep.

Mosedale and co-workers³⁵⁻³⁷ used a technique of applying a constant load to helical spring samples immersed in liquid sodium. The strain was determined by measuring X-radiographs taken in a hot cell after irradiation. This method has the advantages that the applied loads do not change during the test and the applied stress is nearly pure shear. It has the disadvantages, however, that the strain can be determined only at intervals when the capsules are removed from the reactor. In their earlier results³⁵ irradiation induced creep was measured at 300°C in a variety of materials including Nimonic PE16, Nimonic 80A, 316 stainless steel, molybdenum, and nickel. More recent results^{36,37} are focused on materials for fast reactor cores, namely five different austenitic stainless steel alloys. The results for all the alloys are similar with linear stress and dose dependence. The temperature dependence is weak and appears to depend on the material. In some cases the creep rate increases with increasing temperature and in others it decreases. There appears to be strong evidence to support a damage rate dependence. The strain per dpa tends

to decrease with increasing damage rate. Two curves have been replotted in Fig. 4. The normalized creep strain/dpa shows a linear dependence on the inverse square root of the damage rate. This dependence is expected from theory³⁸ when defect recombination is the dominant mode of loss. Although microstructural evidence is not available, this condition is likely. The decreasing creep rate with increased damage rate may lead to lower creep rate in pulsed systems because these systems will have higher damage rates for the same average damage. No mention of swelling was made and although the total damage was as high as 15.5 dpa for 316 stainless steel and up to 36 dpa for some of the other alloys, no indication of a rapid increase in creep rate was observed. The creep rates were nearly constant from 1 dpa until the tests were terminated. An attempt to compare the results with other data from pressurized tubes and tensile creep tests tended to uncover the complex aspects of irradiation creep behavior but "...no simple generalization..." appears to be possible at the moment.³⁷

Fig. 4. The normalized creep strain per dpa plotted against the square root of the damage rate for two austenitic stainless steels. The data are replotted from Ref. 37.

The method of loading helical springs has also been used to study irradiation creep of nickel alloys³⁹ for temperatures from 270 to 360°C. Three types of nickel alloys were studied: solid solution strengthened with molybdenum, precipitation hardened with aluminum, and dispersion strengthened with the additions of Al_2O_3 . Some of the results are shown in Fig. 5. In general, alloys which showed the greatest reduction in thermal creep also showed the greatest reduction in irradiation creep. A strong dose rate effect was encountered which made direct comparisons difficult. At a low damage rate ($\approx 2.5 \times 10^{-8}$ dpa/s) the alloys containing aluminum and molybdenum crept at about the same rate as pure nickel while at a higher damage rate ($\approx 4 \times 10^{-7}$ dpa/s) the alloys crept about ten times slower than pure nickel. A recent report⁴⁰ of creep rates in modified AISI 316 also supports strong alloying effects on the creep rate. In particular, additions of silicon and molybdenum decrease the creep rate. In general, the same elements that reduce swelling also reduce creep.

Fig. 5. The shear creep strain as a function of dose for three different alloys of nickel (replotted from Ref. 39).

Up to this point all the experimental techniques discussed involve measurement of the creep strain at intervals when the samples are out of reactor. In order to study strain transients and creep at low fluence, it is necessary to measure strain continuously. There are data reported from at least five different fully instrumented creep test assemblies. Although the number of reported experiments is small and the results are inconclusive, these experiments will now be discussed.

In 1972, Gilbert et al.⁴¹ reported a partially successful experiment to measure in-reactor creep continuously. A specimen of 20% cold-worked 316 stainless steel was crept under uniaxial stress of 138 MPa at 454°C. The measurements terminated prematurely when the machine failed. The measured steady-state creep rate, $1.2 \times 10^{-10} \text{ s}^{-1}$ is higher than values measured using pressurized tubes but is about 30% lower than the values reported below for type 321 stainless steel.

Two specimens of 20% cold-worked Er58B (type 321) stainless steel were irradiated in a tension creep machine in DFR.³⁶ The first sample was irradiated at 500°C for 960 h at 93.1 MPa followed by 275 h at 41.0 MPa. The observed steady state creep rates were 1.6×10^{-10} and $5.8 \times 10^{-11} \text{ s}^{-1}$, respectively. The second specimen was irradiated at 500°C unstressed until a dose of 11.1 dpa and was then creep tested at 500, 450, and 400°C at 93.1 MPa and also at 400°C at 13.8 and 48 MPa. Under irradiation at these temperatures this steel undergoes some swelling and also shows some phase instability which complicates the interpretation of the creep data. Several observations are possible, however. Strain transients which lasted from 1 to 2 dpa were seen when the stress was increased. These transients did not appear when the stress was reduced. The pre-irradiation of the second sample did not seem to be important in influencing creep rate; the average measured creep rate at 93.1 MPa was nearly the same in the two samples. The creep rate increased about a factor of 4 as the temperature decreased from 450 to 400°C. This trend was not expected to continue since the creep rate was higher at these temperatures than that measured on helical springs at 280°C. No satisfactory explanation was offered for this negative temperature dependence.

Based on published results, perhaps the most successful design for an in-reactor creep machine is that based on the resonant cavity strain measurement developed at Grenoble. Creep machines have been built for both the Rapsodie and BR2 reactors. Dupouy et al.⁴² reported results from three experiments made in Rapsodie. All three specimens were 316 stainless steel — the first was solution annealed and the other two were 20% cold worked. The equipment failed during the first experiment and the data were incomplete. The other two experiments were carried out at 450°C and at a high applied stress of 400 MPa. After little if any transient creep, the measured creep rates were 1.7 and $1.4 \times 10^{-9} \text{ s}^{-1}$, respectively. The second sample was irradiated to 36 dpa and the creep rate remained linear with dose throughout the experiment.

Several successful experiments using a creep machine in the BR2 reactor have been reported.⁴³ Three specimens of DIN 1.4981 stainless steel (16 Cr, 16 Ni, Nb) were irradiated at stresses of from 100 to 200 MPa. The irradiation temperature for the three samples was initially 420, 450, and 500°C; however, a heater failure in the second experiment caused the temperature to decrease to 350°C. During this temperature decrease, which lasted about 2000 h, no change in the creep rate was measured. The observed temperature dependence is not strong with the maximum creep rate at 420°C and the minimum at 450°C. The stress dependence based on stress-change measurements is linear, but an unusual observation was made. After creeping to a dose of about 12 dpa, the 420°C specimen was irradiated at zero stress for some time and then reloaded to 100 MPa. No deformation was observed for two reactor cycles and then the creep began at a rate double that measured previously at 100 dpa. We believe that it is possible that rapid swelling began at that dose and the measured enhanced creep rate reflects swelling-driven creep. No density measurements have been made.

The creep behavior of ferritic steel DIN 1.6770 (2.2 Cr, 1.01 Mo, 0.8 Ni) has also been measured using the resonant cavity apparatus.⁴⁴ For irradiations out to a fluence of 4.5 dpa and stress to 200 MPa at 400°C, the creep rate was less than $2.2 \times 10^{-11} \text{ s}^{-1}$. At 500°C

and 100 MPa the strain rate increases with time (like tertiary creep) and is about five times as fast as in a typical austenitic stainless steel. Continuation of the 400°C sample at 500°C reproduced the same result. The 500°C result is about ten times as fast as thermal creep in the same material, but has the same general behavior.

Another successful creep machine has been used to study in-reactor creep of zirconium alloys. Two results published by MacEwen and Fidleris will be reviewed. First, in an early report⁴⁵ the creep of Zircaloy-2 was measured in and out of reactor at 335 K and 25 MPa. After an initial transient of about 50 h, a steady state creep rate of $5.6 \times 10^{-10} \text{ s}^{-1}$ was measured. Then, when the reactor was shut down, a very large initial increase in the creep rate was observed. This increase was only a 1 to 2 h transient, however, during which the creep rate dropped to an insignificant value. The authors offered convincing arguments to support the idea that the increase in creep rate occurs through a climb-glide process driven by the excess vacancies in the material. This explanation was discarded later, however, when this same phenomenon was observed in other experiments.⁴⁶ Continued irradiation showed that the "transient strain" was primarily due to thermal expansion and contraction of the creep machine. One test on a zirconium single crystal was continued for 5800 h at 300°C and 25 MPa and the final observed strain rate was $3.9 \times 10^{-10} \text{ s}^{-1}$. This compared to less than $1.4 \times 10^{-10} \text{ s}^{-1}$ during reactor shutdown periods. A second crystal was irradiated at a 70% higher flux at 5.8 MPa and 300°C. Then, during a long reactor shutdown, the stress was raised to 25 MPa. The creep rate was initially about half of the steady state value of the first crystal but slowly increased to a value 2.4 times the value of the other crystal. The microstructure of the first specimen was studied, and the visible damage was seen to be almost entirely small loops with a density of $4.5 \times 10^{20} \text{ m}^{-3}$ and an average size of 13.5 nm. A low magnification micrograph revealed swaths through the loops probably cleared by glide of network dislocations. We believe that the majority of the observed strain occurs from the glide of these dislocations which removes the loops. A model was suggested for this flux-enhanced swath formation.

5. IRRADIATION CREEP EXPERIMENTS USING ACCELERATED PARTICLES

The final class of experiments designed to study irradiation-induced creep phenomena are those which use beams of accelerated particles to cause the irradiation damage. During the last ten years there have been a variety of experimental efforts utilizing many different particles including electrons, neutrons, protons, deuterons, and alpha particles. In all cases, thin metal samples are loaded in complex devices on the target end of a large accelerator. For reasonable irradiation times the total irradiation dose is small, and so great care must be taken to minimize thermal transients both in the specimen and the machine. Small temperature changes result in large strain errors. In addition, the particle beams produce radically different primary damage energy spectra which makes comparisons with reactor data difficult. The major advantage of these systems is that the experimental variables — stress, temperatures, and fluence — are known more precisely, and analysis of the data produces important information about the underlying physical mechanisms.

There have been only a few electron and neutron experiments so they will be briefly considered first. The range of electrons with energy of the order of 1 MeV in metals is short so that the sample thickness is limited to a few microns. This severe restriction eliminates the use of electron beams from producing bulk material creep data. On the other hand, the very thin foil is an advantage, in that, by using a high voltage transmission electron microscope it is possible to observe the changes in the internal structure during irradiation. Buckley and Manthorpe⁴⁷ irradiated aluminum-silicon alloys in an HVEM and were able to calculate the local stress on network dislocations by measuring dislocation bowing. The outcome of these measurements is that during an hour of irradiation the local stress on individual dislocations changed by orders of magnitude and even sign. The authors write, "one affects all and all affects one." This somewhat obvious conclusion has been almost completely ignored in theoretical models for irradiation creep.

Another in-situ irradiation creep experiment under HVEM irradiation has been reported on annealed 316 stainless steel⁴⁸ along with some observations of thermal creep. Thermal creep is characterized by heterogeneous planar glide of network dislocations leading to rapid hardening and decreasing creep rates. Irradiation creep shows nucleation, growth, and unfauling of Frank loops. These unfaulted loops glide to join the network. Applied stress leads to preferential nucleation and unfauling of those loops whose Burgers vectors make the smallest angle to the tensile stress. This work shows a definite difference in mechanisms between thermal and irradiation creep.

Studying creep with electrons and light ions offer a partial solution to the problem of obtaining information under better known experimental conditions, but in reactors almost all the damage occurs through interactions of neutrons. Because of the availability of neutrons from fission reactors, the damage from fission neutrons has been studied. For fusion reactors, which produce a high fraction of 14 MeV neutrons, nothing is known. Two high energy neutron sources have been used to study differences that high energy neutrons might make. One of these sources, the Be(d,n) source at the University of California at Davis has been used to study irradiation creep.⁴⁹ A sample of annealed niobium was irradiated at 600°C under a stress of 76 MPa after steady state thermal creep was established at $1.4 \times 10^{-6} \text{ s}^{-1}$. Then, a series of 12 short beam-on, beam-off cycles were conducted. When the beam was turned on the creep rate immediately increased about 30%. Then the creep rate decayed during the next 6000 s to a value less than 1/10 of the thermal value. When the beam was turned off the creep rate decreased immediately and then slowly returned to near the thermal value during the next 6000 s. The authors believed that the short transients were due to first an increase and then a decrease on the interstitial concentration while the long transients resulted from vacancy clusters.

The early experiments using ion beams uncovered the difficulty encountered in maintaining very precise temperature control, but they also demonstrated the basic usefulness of the approach. Buckley and Manthorpe⁴⁷ irradiated pure copper with 4 MeV protons at $2 \times 10^{-6} \text{ s}^{-1}$, 17°C, and with applied stresses of 10 and 50 MPa. The experiment

showed considerable enhancement in the creep rate but quantitative values were not possible. Harkness et al.⁵⁰ reported results on three stainless steel samples irradiated with 22.4 MeV deuterons. The creep rate of a sample of solution annealed 304 stainless steel, irradiated at 416°C under a stress of 414 MPa, was found to be about nine times higher than the thermal creep rate of $4.2 \times 10^{-9} \text{ s}^{-1}$. The observed weak temperature dependence reflected an activation energy of $34 \text{ kJ mol}^{-1} \text{ }^{\circ}\text{C}^{-1}$. This same steel pre-irradiated in EBR-II to a fluence of $5 \times 10^{25} \text{ n/m}^2$ showed a factor of 15 decrease in the proton induced irradiation creep. After adjustment for different irradiation temperatures and stress, the measured value showed good agreement with in-reactor creep rates measured using pressurized tubes. Creep of 20% cold-worked 316 was also measured at 505°C. A 1-2 power stress dependence was found, which compares to a thermal creep stress dependence of about 9.

Irradiation creep of high purity nickel has been investigated for several years using both deuteron and alpha particle beams. In 1976, Hendricks et al.⁵¹ reported a creep rate of $7.5 \times 10^{-9} \text{ s}^{-1}$ in nickel irradiated with 22 MeV deuterons at 224°C. The creep rate was several orders of magnitude higher than that expected for thermal creep. Other measurements were made with this same creep machine⁵² using both 22 MeV deuterons and also 70 MeV alpha particles. If the damage functions are correct, about 20% fewer defects contributed to irradiation creep from alpha particles than for deuterons, (i.e., deuterons were about 25% more effective in causing irradiation creep). In this work the initial transient appeared to saturate after 12 h of irradiation. Stress change experiments with most stresses above 200 MPa give an applied stress exponent of from 1.67 to 2.00. The results are consistent, however, with a linear stress dependence for stresses less than 200 MPa. Microstructural observations showed an increased loop size with increased stress. This work was continued⁵³ with emphasis on the microstructure during the initial transient. After 8 h of irradiation the initial dislocation density of $2 \times 10^{15} \text{ m}^{-2}$ decreased to $3.2 \times 10^{14} \text{ m}^{-2}$. During this same period the defect cluster density increased from less than $1 \times 10^{19} \text{ m}^{-3}$ to $1.4 \times 10^{22} \text{ m}^{-3}$, and the average size of the clusters changed from 2.9 nm to 4.3 nm. The creep

rate divided by the flux decreased from 4.3 to 0.05 dpa⁻¹. The study of nickel continued on another accelerator using 17 MeV deuterons.⁵⁴ For a specimen of nickel loaded to 215 MPa at 220°C, the steady state irradiation creep rate was found to be $2.6 \times 10^{-8} \text{ s}^{-1}$ for a damage rate of $5.4 \times 10^{-7} \text{ dpa/s}$.

Before the test, the thermal creep rate was about 70% of this value; but after a total dose of 0.055 dpa, the thermal creep rate was reduced to 10% of irradiation creep values. The same machine was used to evaluate the stress and temperature dependence of irradiation creep in pure, cold-worked nickel.⁵⁵ For a stress range of 205 to 250 MPa the steady state creep stress exponent was 2.5. An increase of 40°C elevated the creep rate by a factor of 1.44 which was less than expected. In all these studies the creep rates were greater than $2.0 \times 10^{-9} \text{ s}^{-1}$ and the stresses were higher than 200 MPa. Most previously discussed results, showing linear stress dependence, were measured at lower applied stress.

McElroy and co-workers^{36,56,57} have reported creep measurements on pure nickel and 321 stainless steel using thin samples ($\approx 25 \text{ }\mu\text{m}$) and irradiating with 4 MeV protons. In the initial report,⁵⁶ irradiation and thermal creep of pure nickel was measured for temperatures from 400–600°C and stresses from 20–200 MPa. Thermal creep agrees with that reported for bulk specimens with an activation energy of 2.8 eV. Under irradiation the thermal creep rate decreases while the irradiation creep rate continues at a rate in excess of the thermal rate. Below 125 MPa, irradiation creep shows a linear stress dependence. Electron microscopy reveals more dislocation line segments for stressed specimens while unstressed specimens show more loops.

The work was continued with emphasis on creep in commercial 321 stainless.⁵⁷ The measured irradiation creep rate on 60% cold-worked samples shows a linear stress dependence for stresses less than $\sim 150 \text{ MPa}$, a logarithmic time dependence, and a small temperature dependence below 520°C ($Q \approx 0.9 \text{ eV}$). Above this temperature the temperature dependence is about the same as self diffusion. Electron microscopy after 0.22 dpa at 150 MPa revealed a very high dislocation density (10^{15} m^{-2}) and showed very few signs of irradiation damage. This

microstructure is in contrast to that seen for a solution annealed sample which had almost no network dislocations and an unfaulted loop density of $2 \times 10^{22} \text{ m}^{-3}$ with an average diameter of 8.5 nm. Since the total dose that can be achieved in these devices is low, an attempt was made to pre-irradiate some samples under stress and then measure the irradiation creep.³⁶ For a pre-irradiation to 7 dpa at 440°C under a stress of 50 MPa, the subsequent creep rates were nearly the same as those measured before the pre-irradiation. Only the swelling strain was subtracted. No stress effect on swelling was seen.

Recently several investigators have published results on irradiation creep in AISI 316 stainless steels. Blanchard et al.⁵⁸ irradiated 50- μm -thick specimens with 29.4 MeV alpha particles at temperatures from 300 to 450°C. A total dose of 1 dpa was achieved with a damage rate of $10^{-5} \text{ dpa s}^{-1}$. One significant new result is that addition of 0.25 wt % Ti to this steel decreases the creep rate by a factor of from 2-3.

In another approach Jung et al.^{59,60} have reported creep results on 50- μm -thick foils of 20% cold-worked AISI 316 stainless steels bombarded with 10 MeV deuterons and 7 MeV protons. For temperatures between 300 and 400°C the irradiation creep increases about the same fraction as that seen from neutron data. The stress dependence is quadratic above 250 MPa. Samples of a very low impurity content heat of this steel creep much faster than the commercial heat. Very careful analysis of the damage functions for the deuterons and protons shows no difference in creep behavior between the two particles. In a separate experiment Reiley et al.⁶¹ report creep rates in agreement with these results using a beam of 60 MeV alpha particles. These alpha particles produce primary recoils having 0.1 to 1.0 MeV; and therefore, the damage spectrum should be more like that found in fast reactors. The agreement between these last results using beams of three different particles is in contrast to the difference between deuterons and alpha-particles mentioned previously.⁵² All the reported ion irradiation creep rates are from 2 to 10 times higher than that found for neutron irradiation. It appears that questions concerning defect production and cascade structures still do not have

completely satisfactory answers. Also, very recent measurements⁶² of swelling in deuteron bombarded high purity stainless steel have shown a decrease in incubation dose with applied stress. It is likely that an accompanying change in creep will occur.

Torsion creep under 21 MeV deuteron irradiation of solution annealed and aged (6 h at 800°C) AISI 316 stainless steel has been measured at 400°C at a maximum stress of 110 MPa.⁶³ The stress is lower than the previously reported creep experiments on this steel and the results are in sharp contrast. For the first 0.15 dpa, the observed creep rate is negative, (i.e., the creep strain opposed the applied stress). The explanation for this phenomenon is based on an increase in shear modulus for the material as a function of irradiation resulting from the precipitation of Ni_2Si particles which decreases the matrix concentration of these elements. No microstructural information was presented. In the torsion experiment, an increase in shear modulus will result in a negative strain to maintain a constant applied stress.

Figure 6 is a plot of these results for cold-worked 316 stainless steel along with some results using protons and deuterons. The solid symbols refer to measurements made on specimens which had been pre-crept. Note that no significant difference is observed.

In another set of experiments using this same torsional creep apparatus,⁶⁴ pure nickel and Ni-4 at. % Si were studied at 350°C. In all the experiments pre- and postirradiation creep rates were measured, and significant irradiation-induced hardening was seen. The torsional machine allows measurements down to a creep rate of $2 \times 10^{-10} \text{ s}^{-1}$ which is lower than the rates reported using other machines. This capability allows creep studies at low stresses, 23 to 110 MPa, which makes the data more comparable to that measured in-reactor. In light of this, a linear stress dependence was measured for stresses less than 70 MPa. Above this stress the creep rate increases faster. The creep rate was linear in dose and the observed radiation hardening saturates for doses greater than 0.01 dpa. The silicon alloy shows more hardening and microstructural observations show a higher density of small Frank loops decorated with Ni_3Si .

ORNL-DWG 80-16450R

Fig. 6. A summary plot of irradiation creep of 316 stainless steel under charged particle irradiation. The solid points designate samples that were pre-crept (PC) thermally before irradiation.

6. SUMMARY OF IMPORTANT RESULTS AND SUGGESTED AREAS FOR RESEARCH

Many of the main features of irradiation creep behavior had been reported prior to 1977. Such properties as strain increasing with fluence, linear with applied stress, and only weakly dependent on temperature had been reported. However, since 1977 there have been several surprising results, and these have been summarized in this report.

During this time there have been several reports relating creep and swelling. Data have been reported showing that large increases in swelling are accompanied by large increases in creep. In addition, those alloying additions that depress swelling also tend to reduce the creep. The connections become even more complicated with the observation that swelling depends on the applied stress — at least in some alloys at high temperatures. Finally, there is a role played by irradiation-induced or -enhanced precipitation. Each of these phenomena have been observed and reported recently along with the connection to irradiation creep, but the mechanisms connecting them were not understood. Ongoing work is addressing some of these questions.⁶⁵

The effect of temperature on irradiation creep has also been explored and some surprising observations have been made. Formerly, irradiation creep had been thought to be nearly independent of temperature, and there have been a large number of observations to support this weak dependence. The temperature range for these measurements was limited, however, and more recent measurements at lower temperatures have shown an inverse temperature dependence. The creep rate increases considerably as the temperature decreases. Further, it appears that temperature decreases during creep can result in increased strain. Many additional creep tests are needed, especially those using the same measurement techniques, over the entire temperature range. Accompanying microstructural observations are also needed to determine the mechanisms establishing the complicated temperature dependence. It appears that radiation-induced nonequilibrium precipitation plays a role.

The stress dependence of irradiation creep appears to increase at high stresses and temperatures. It appears that this increase results from the same processes leading to thermal creep, but it occurs in the radiation damage structure. The resulting creep rate is faster than thermal creep of a thermal creep structure. Also some nonlinear stress dependence appears to be related to stress dependent swelling, and this is not understood.

Another somewhat surprising result is the apparent difference in the fluence dependence for samples stressed in torsion and for pressurized tubes which are biaxially stressed. A linear creep rate as a function of fluence has been seen in dead-weight loaded springs for fluences from 1 to 40 dpa, whereas the pressurized tubes show an increasing creep rate for the first several dpa. Also, some measurements using charged particle creep apparatus show a dependence of incubation dose on applied stress. Understanding these observations would aid in the development of a more realistic creep equation.

In conclusion, the observations reported during the last few years continue to challenge our understanding of the physical processes governing irradiation creep behavior. It continues to be necessary to couple measurements of creep with those of swelling and also microstructural observations. This last involves not only the dislocation structure that leads to creep but also the alloy segregation, precipitate formation, and cavity behavior. Each of these processes plays an important role in controlling the flow of defects that determines radiation damage processes.

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