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The ORNL Irradiation Creep Facility

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THE ORNL IRRADIATION CREEP FACILITY

T. C. Reiley, R. L. Auble, R. M. Beckers, E. E. Bloom,
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ABSTRACT

A machine has been developed at ORNL to measure the rates of elongation observed under irradiation in stressed materials. The source of radiation is a beam of 60 MeV alpha particles from the Oak Ridge Isochronous Cyclotron (ORIC). This choice allows experiments to be performed which simulate the effects of fast neutrons. A brief review of irradiation creep and experimental constraints associated with each measurement technique is given. Factors are presented which lead to the experimental choices made for the Irradiation Creep Facility (ICF). The ICF consists of a helium-filled chamber which houses a high-precision mechanical testing device. The specimen to be tested must be thermally stabilized with respect to the temperature fluctuations imposed by the particle beam which passes through the specimen. Electrical resistance of the specimen is the temperature control parameter chosen. Very high precision in length measurement and temperature control are required to detect the small elongation rates relevant to irradiation creep in the test periods available (~1 day). The apparatus components and features required for the above are presented in some detail, along with the experimental procedures. The damage processes associated with light ions are discussed and displacement rates are calculated. Recent irradiation creep results are given, demonstrating the suitability of the apparatus for high resolution experiments. Also discussed is the suitability of the ICF for making high precision thermal creep measurements.

INTRODUCTION

When a material is stressed, it undergoes rapid elastic (recoverable) elongation and, if the stress is sufficiently high, plastic (nonrecoverable) elongation. Additional time-dependent elongation occurring under stress is called creep. This deformation is characteristically a highly temperature-dependent process which is significant for engineering applications at temperatures greater than 0.3–0.4 of a material's

melting temperature. Normally, some fraction of the creep deformation is recoverable (the anelastic component), but for the most part creep deformation is nonrecoverable.

Under irradiation, which causes atom displacements, the rate of creep may be increased many orders of magnitude above its "thermal creep rate". This was discovered first in uranium under neutron irradiation by Konobeevski et al.,¹ with the first definitive experiments and mechanistic interpretation reported by Roberts and Cottrell.² The mechanism which was proposed (and is still accepted for uranium) is based on the anisotropic growth under irradiation of individual crystals which, as a stressed aggregate, deform in the stress-biased direction. It was felt that this phenomenon would only be observed in anisotropic materials. However, in the mid-to-late 60's it was discovered that essentially isotropic structural materials such as stainless steel and nickel-based alloys also undergo "irradiation creep".^{3,4} Figure 1 shows the effect of fast reactor irradiation on the creep of type 316 stainless steel. This behavior and the notable limitations of the Cottrell-Roberts theory to describe the irradiation creep behavior of zircaloy,⁵ a moderately anisotropic zirconium-base alloy, have led to the evolution of several other theoretical mechanisms for irradiation creep. Recent reviews of the experimental data and dominant theories are given by Straalsund,⁶ Heald,⁷ Gittus,⁸ and Harries,⁹ (see also Ref. 10).

Initially, the practical consequences of the relatively low rates of deformation under irradiation creep were felt to be of secondary importance to reactor design. However, from experiments reported in 1972¹¹ it was clear that large structural distortions could arise under the swelling and thermal stress gradients in a fast reactor core. Thus, in recent years research has been vigorous on several fronts related to irradiation creep: (1) general design and constitutive behavior, (2) data collection for temperature, flux and composition variations among certain chosen structural alloys, and (3) mechanistic and theoretical studies.

Recently, it has been found¹² that ductility under irradiation creep deformation may be enhanced even beyond that expected for out-of-reactor experiments. The major inference is that the internal stresses

Fig. 1. Thermal and Irradiation Creep Results from Pressurized Tubes (after Gilber et al.⁴⁰).

may be relaxed more easily under irradiation. This has important implications for fusion reactor designs, given the current concern about crack growth and ductility under pulsed conditions; see e.g., Youngdahl and Smith.¹³ As well, the first wall stress state can be strongly affected by irradiation creep.¹⁴

IRRADIATION CREEP MEASUREMENT TECHNIQUES

Several methods of measuring irradiation creep have been used. The dominant techniques have been the fast reactor irradiations of loaded springs (U.K. approach) and pressurized tubes (U.S. approach). In these experiments the spring or tube is periodically removed from the reactor and the length or diameter measured. Several difficulties associated with these approaches are (1) that the stresses are not uniaxial, a factor which restricts interpretation, (2) an intermittent rather than continuous measure of the deformation is made, (3) the limited accuracy

prohibits the detection of fine-detailed response, such as is associated with transients. Other techniques similar in principle to the loaded spring or pressurized tube have been developed, including loaded beams or mandril prebent beams and slit tubes (for stress relaxation measurements). These techniques suffer from the limitations mentioned above.

An alternative technique involving reactor irradiation is the use of an instrumented testing machine placed in the reactor, (see Ref. 15 for many examples). These machines have the advantage that measurements are continuous, but have the disadvantages that they are quite expensive (~0.5 M dollars per machine of limited lifetime) and that accuracy may still be limited, given the terribly inconvenient and constricted test environment.

Alternative to the above techniques is the use of a well-controlled beam of particles capable of simulating the effects of neutron irradiation. With this approach mechanical measurements may be made on a specimen being bombarded. The particles which have been used range from fission fragments¹⁶ to electrons.¹⁷ The most common approach is to use a beam of light ions ($m < 4$) accelerated by a cyclotron or Van de Graaff accelerator. The first experiment which demonstrated the feasibility of light ions to simulate neutron-induced irradiation creep was that of Harkness et al.¹⁸ Other major experiments have been performed by Hendrick et al.,¹⁹ McElroy et al.,²⁰ Simonen and Hendrick,²¹ Faulkner and McElroy,²² Jung et al.,²³ and Reiley et al.²⁴ General features of these experiments have been reviewed by Reiley and Jung.²⁵

The results from these light ion experiments are similar in phenomenology to in-reactor neutron results with regard to stress, flux, and temperature dependence. Furthermore, the displacement rates under full beam from several of the accelerators used may be >10 times that of a fast reactor, thereby, allowing moderately accelerated testing. This advantage, plus considerably better experimental control, increase the rate at which useful data may be taken in the light ion experiments and increase the available range of parametric variation.

There are several potential difficulties associated with the use of light ions to simulate neutrons. These are described in the following sections.

THE EXPERIMENTAL CONSTRAINTS

Several years ago the development of the Irradiation Creep Facility (ICF) was initiated at Oak Ridge. A statement of the objectives is: to allow the performance of mechanical property measurements under irradiation causing atom displacements, where the displacement processes and their effect on mechanical properties may match or be directly related to the effects of fast neutron ($E > 0.1$ MeV) irradiation, and where the mechanical properties observed may be systematically related to properties of bulk material. It is recognized that fundamental information of quite different sorts than the above constraints would allow may be generated using the ICF; however, the primary goal for completion of this facility has been that it satisfy the above criteria.

There are several experimental factors which make this experiment quite difficult. First, the rate of deformation of materials under fast reactor displacement rates ($\sim 10^{-6}$ dpa/s) and under loads where irradiation creep is important is very small, with typical strain rates being in the range $5 \times 10^{-10} - 5 \times 10^{-8}$ /s. A specimen whose length is limited by the ion beam diameter (~ 20 mm) would have an elongation rate of $0.01 - 1$ nm/s. This is an extremely slow movement to detect in a relatively small amount of time (~ 1 day). The duration of an experiment is in part dictated by the cost per unit time, as well as the availability of the accelerator.

Given such stringency of length resolution it is imperative that thermal expansion fluctuations be minimized. For this experiment specimen temperature control < 1 K is required. The temperature monitoring and control system, which has constituted a major fraction of the overall effort,²⁶ is summarized later. A more obvious requirement is that the length measuring device be very accurate. In this experiment, a capacitance bridge technique, to be discussed later, was used.

Another important experimental constraint is the suitability of the accelerated particles in terms of penetration distance and damage characteristics. The penetration distances of light ions, heavy ions, and neutrons are shown in Fig. 2 for typically available energies. For our purposes no ex-reactor neutron source exists with sufficient flux for

Fig. 2. Penetration Distance of Various Particles in Nickel.

these experiments. Also, although the localized damage of heavy ions is similar to the damage from primary-knock-on atoms produced by fast neutrons, the penetration of heavy ions is too shallow to allow testing of sufficiently thick specimens which would represent bulk behavior; this thickness was set at 25 μm , a thickness equal to three times a conveniently obtainable grain diameter.²⁵ And, although medium-energy electrons (~ 1 MeV) have large ranges, the damage process is quite different than that of fast neutrons. The possibility of satisfying the major experimental objectives using very high energy electrons ($E > 100$ MeV) remains open, although displacement rates based on the performance of the Oak Ridge Electron Linear Accelerator (ORELA), for example, are too small for our purposes, and the ratio of beam heating to damage is too high for convenient heat removal.

The remaining option, involving light ions, is the approach which was taken. Here the choice of ion and energy becomes a question, on a coarse level, of penetration distance and on a fine level, of damage uniformity. The latter aspect of the choice reflects that the damage cross section is changed as the particle energy is degraded; see Fig. 3.

Fig. 3. Calculated Displacement Rates Caused by Elastic Scattering vs Penetration Depth for Several Particles in Nickel (after Hendrick et al.¹⁹).

Given the limited range and inhomogeneous damage (for specimen thickness which approaches this range) for particles accelerated in a 4-5 MV Van de Graaff, and given the rather small beam currents from tandems of comparable size, it was decided that a larger accelerator, such as the Oak Ridge Isochronous Cyclotron, was required. Once this decision was made, the choice of ion and energy became quite flexible, with the particle and energy being chosen to maximize the displacement rate. The initial beam was 60 MeV α^{++} , which has been the only beam used in our experiments to date, although d^+ or p^+ beams may also be used at some later time.

THE APPARATUS

Shown in Fig. 4 is a schematic of the experimental approach. A beam of particles, 60 MeV α^{++} , (1) enters the helium-filled apparatus through a 6 μ m stainless steel foil; (2) passes through a water-cooled mask which confines the beam to the desired rectangular geometry; (3) passes by a charged-plate ion counter, whose current is directly related to the total beam current; (4) passes through the

Fig. 4. The ORNL Irradiation Creep Facility.

specimen; and (5) comes to rest in the cooling block located less than 0.3 mm downstream from the specimen. The specimen is loaded by a motor-driven ball screw attached to a low force-constant spring (16 kN/m) which is attached to the lower pull rod. The spring is gimbal-type and provides no torque upon a change in length. The load cell is an LVDT which provides an output in response to the elongation of the forcing spring. Several diagrams and photographs of the apparatus components are shown in Figs. 5-10. A summarized list of the major experimental features is given in Table I.

Fig. 5. The Specimen Grip, Length Measurement Configuration for the ICF, Beam View.

Fig. 6. A Mounted Specimen Ready for Testing, 180° to Beam View.

Fig. 7. The Internals of the ICF.

- a. SPECIMEN, GRIPS, PULLRODS
- b. COOLING BLOCK
- c. WATER COOLED PLATES AND SUPPORTS
- d. BEAM MASK AND ION COUNTER
- e. INFRA-RED PYROMETER (MOVABLE)
- f. MOTOF-DRIVEN LOADING DEVICE
- g. HELIUM CIRCULATOR AND AMBIENT TEMPERATURE CONTROLLER
- h. TC OXYGEN METER, GAS PURIFIER
- i. BEAM LINE
- j. CONTAINMENT VESSEL
- k. HELIUM INLET
- l. FARADAY CUP PORT
- m. TC COLD TRAP AND DIFFUSION PUMP
- n. COOLANT FEED AND EXHAUST

Fig. 8. An Overall Schematic of the ICF.

Fig. 9. The Beam Room at the Oak Ridge Isochronous Cyclotron (ORIC) Housing the ICF (cf. Fig. 8).

Fig. 10. The Control Console and Data Acquisition Equipment Located in the Counting Room in ORIC.

Table 1. General Features of the ICF

1. Test Environment

Circulating, H₂O Trapped, Purified Helium

Helium Pressure = 3×10^4 – 10^5 Pa

Helium Temperature = 300 ± 0.5 K

2. Specimen Dimensions

Gage Length = 20 mm

Gage Width = 2.0 mm

Gage Thickness = 75–125 μ m

3. Temperature Monitoring and Control

Control: Specimen Resistance

Monitoring: Thermocouples, Translatable Infrared Pyrometer

4. Mechanical Systems

Length: Capacitance Transducers

Load: Spring plus LVDT

5. Alpha Beam Characteristics

Energy In = 60 MeV

Energy Out = 54 MeV (for 75 μ m Ni)

Current Density ≤ 0.25 A/m²

Displacement Rate $\leq 3 \times 10^{-6}$ dpa/s

6. Range and Response under Beam

Temperature ~500–1000 K

Stress ≤ 580 MPa

Length Resolution ≈ 20 nm

Elongation Rate Resolution ~ 0.05 nm/s (in 4 h)

Strain Rate Resolution $\sim 10^{-9}$ s⁻¹ (in 4 h)

Time for Change in Stress ~ 10 s

Time for Change in Temperature ~ 1 s

The Mechanical Testing Equipment

Figures 5 and 6 show the arrangement of specimen, grips, and length measuring devices. The grips are hydraulically operated at 12 MPa which provides a gripping frictional force of 80–160 N (coef. of friction – 0.25), depending on the specimen grip length (12–25 mm at each end). The relative motion of the grips is measured by two capacitance transducers manufactured by Automatic Systems Laboratory (ASL), (model number 1072 SLVC Transducers and 1050.6 bridge). This system provides length resolution ~20 nm, with 0.01% linearity over a measuring range of 10 mm. The components for the transducers were machined under ORNL supervision from Invar (Unispan LR-35 from Universal Cyclops Steel) having a thermal coefficient of expansion of $0.1 \times 10^{-6}/K$. The transducers were specially assembled and calibrated by ASL. Thermal radiation shields protect these transducers from excessive heating.

The pull rods are made of Cr-plated Invar and are set in precision bearings (~10 μ m clearance) which are housed in a line-bored frame (tolerance: <25 μ m misalignment over a distance of 0.45 m). The frame, pullrods, and loading system are balanced with varying thermal expansion coefficients to cancel any thermal expansion caused by an overall temperature shift. All major structural components are water cooled from a constant temperature bath (Lauda Inc. Model K-4/R) controlled to ± 0.1 K. The entire apparatus has vibration isolators and/or bellows attachments to every external device or support point. Equipment vibrations have presented no problems.

Figures 8 and 9 show the orientation of the other major components of the apparatus. A fan-driven helium circulation system is shown which has a helium purifier (Precision Gas Products, PGP-8100-ST01), an oxygen analyzer (Applied Electrochemistry S-3A, N-22, R1), a temperature controller (Valley Industries, Inc.), a heat exchanger using the primary flow of the constant temperature water bath, and a liquid nitrogen cold-trap for water removal. Another helium circulation loop using a bellows pump, a surge tank and another heat exchanger allows the flow of helium in two jets which impinge upon a point slightly upstream of the specimen. The system may be evacuated with a diffusion pump to pressures $\sim 10^{-3}$ Pa, and the system is designed for overpressures of one atmosphere.

The Temperature Control System

The temperature control system is based on the variation of specimen resistance with temperature. A variable AC ohmic heating current which supplies a significant amount of the specimen heat input is used to compensate for heat input fluctuations. The specimen is treated as a leg of a modified double Kelvin bridge and a fast control circuit is used to keep specimen resistance within narrow limits. This system is described at great length elsewhere,²⁶ and will only be reviewed here. An AC system was chosen to avoid temperature-dependent DC emfs which may occur at any point along the electrical path. The frequency was chosen to be 750 s^{-1} to allow rapid response, but not so high as to encounter stray reactances. The real difficulty in perfecting the measurement and control system was the development of a sufficiently accurate AC error voltage detector. It was decided to use a phase-insensitive rectified signal approach, one which avoids reactance problems. The error voltage signal must be rectified to allow positive and negative control.

A block diagram of the temperature control system is shown in Fig. 11. The resistances R_x , R_y , and R_s are, respectively, the specimen, cable, and standard resistances. Since the normal resistance for the specimen dimensions chosen is between 0.05 and 0.1Ω , the cable resistances become quite important, thus, the need for a double Kelvin bridge. Also, since the heating current must be rather large (our power amplifier may generate up to 30 A), the standard resistor must have small resistance so that heat is primarily generated in the specimen, and the resistor must be thermally stable. The standard resistor is an enclosed manganin strip resistor with 4 taps, having a total resistance of 0.120466Ω at 23°C . Its temperature is maintained constant by a closed circulation loop of butanol flowing through a Lauda K-2 cooling bath. The instrumentation amplifiers and the rectifiers are the critical features of the entire system. These provide an error voltage proportional to $I \cdot \Delta R_x$, which has divided into it a voltage proportional to the current to linearize the control. This error signal is amplified and becomes a 750 s^{-1} control signal whose amplitude determines the heating current. The temperature controller has performed quite well

under actual operating conditions, maintaining the specimen resistance

excursions to less than that associated with temperature excursions of 1 K. Under conditions of constant heating current, the beam has been shown to cause temperature fluctuations of greater than 20 K. Without beam on the specimen, the temperature control is better than 0.1 K.

One important capability of the temperature controller is that it may compensate for resistance offsets caused by annealing, radiation damage, precipitation, or other microstructural changes. This is done by balancing the specimen resistance at a controlled ambient temperature using a backup bridge; then, if a change occurs under test conditions, the specimen resistance may be rebalanced at this ambient via a $\Delta R_{\text{correction}}$. This $\Delta R_{\text{correction}}$ is simultaneously included in the set resistance for the primary bridge, as well as the back-up bridge. For this correction to be perfect, a resistance change associated with structural changes must have the same value regardless of temperature. Although this has not been investigated sufficiently to be proven, there is a similar precedent associated with changes in resistance caused by

changes in composition of solid solutions, the so-called Mathieson's Rule. To date, we have been satisfied with our ability to compensate for small intrinsic resistance changes.

The temperature gradients along the length and width of the specimen are monitored by a movable infrared pyrometer having a resolution of ± 1 K (Raytek, Inc., Model S/L-300). The spot size at the 0.3 m focal distance is 1.1 mm. The infrared wavelength is 2.1–2.3 μm which is nearly completely transmitted through the special quartz viewing window (G.E. type 125 fused quartz). A serious complication in using the IR pyrometer for absolute measurements is the variability of emissivity with materials and with time. Our experience with type 316 stainless steel has shown that emissivities between 0.2 and 0.6 may be obtained depending on the length of exposure at temperature. A very thin graphite coating increases the emissivity to 0.9. Although the emissivity does stabilize with time, more characterization experiments and/or surface treatments (e.g., preoxidation or graphitization, see McElroy et al.²⁰) will be necessary before the IR information may be used as a more precise absolute temperature measuring device. For our initial irradiation creep experiments the specimens were oxidized 2 h at 825 K in air.

However, at this stage the pyrometer serves as a device for monitoring gradients or changes in gradients. See Fig. 12 for an example of the temperature profiles obtained during an experiment.

Extensive heat transfer calculations were performed at ORNL by K. K. Chipley, which were instrumental in designing the gripping arrangement. It was shown that temperature peaks could occur at points outside the gage section, depending on the proximity of the grips (heatsinks). Figure 13 gives an example of possible temperature profiles, with one in which the grips are too widely separated and one with correct grip spacing. This optimum grip spacing is somewhat temperature dependent. A pair of cartridge heaters (Watlow Elec., C1E14, 25 watts) are used to control (± 1 K) the temperature in each grip and can provide an extra degree of control over the specimen temperature and temperature profile. The spacing between the specimen and the cooling block is another degree of freedom which can affect the temperature; and this may be varied quickly by a stepping motor.

Fig. 12. Temperature Profile for a Type 316 Stainless Steel Specimen During Irradiation; Surface Emissivity Assumed Constant at 0.33.

Fig. 13. (a) Temperature Profile Calculated for a Type 316 Stainless Steel Specimen Absorbing 12 J/s Beam Power in Gage Section, with a 31.6 mm Grip Spacing, without Grip Heating. The ohmic heating is 4.35 J/s. (b) The calculated temperature profile for the same conditions as in (a), but with grip spacing of 27.6 mm and a grip heating of 1.4×10^9 J/s-m³.

Thermocouples are also used to provide temperature information. Pt and Pt-10% Rh wires of 50 μm thickness are individually spot-welded to the specimen at adjacent points across the specimen. (Care must be taken to minimize the longitudinal distance between the wires to minimize the rms voltage error caused by the 750 s^{-1} heating current passing through the specimen.) These thermocouples are used primarily to assist in the measurement of the specimen resistance at ambient temperature, although they also provide reference information for temperature fluctuation during the experiment. No evidence has yet been observed that the alpha beam irradiation causes decalibration of the thermocouples.

One of the more important parts of the temperature control system for these experiments is the technique for specimen heat removal. The beam provides a great deal of heat input; e.g., a 60 MeV alpha beam passing through a 75 μm stainless steel specimen leads to the generation of $7.6 \times 10^8\text{ w/m}^3$ per μA of particles. A normal beam current on the specimen would be 0-5 μA ; therefore, to maintain a constant specimen temperature, a heat removal rate of up to 30 w is required. A significant amount of heat is conducted across a helium-filled gap of about 300 μm to the nose-plate of the cooling block. The position of the cooling block may be adjusted with several degrees of freedom in relation to the specimen. (Also, since during certain measurements it is necessary to move the cooling block out of the beam path, this position must be quite reproducible.) The cooling block is filled with a circulating mixture of ethylene glycol and water, controlled at $\sim 270\text{ K} \pm 0.5\text{ K}$ using a refrigerator system. The cooling block was originally designed to be cooled with liquid nitrogen, given the projected inability of beam heating to be removed by convective rather than boiling heat transfer within the cooling block. This proved not to be necessary; i.e., convective heat transfer was capable of removing sufficient heat. The other heat loss paths are to the grips or to the helium surrounding the specimen. This latter loss mechanism can be greatly enhanced using the available helium jets. The specimen thermal time constant is about 0.2 s.

To promote long-term thermal stability, the enclosing vacuum jar is insulated and both it and the main base plate have heating elements

imbedded in them. The apparatus ambient is maintained at a temperature a few degrees above room temperature to improve ambient control. The various elements of the helium temperature control system give an overall control of ± 0.5 K for the helium temperature, although the apparatus structural members are regulated even more closely by the constant temperature water bath. Numerous copper-constantan thermocouples are used to monitor the temperature throughout the apparatus.

Beam Measurement and Characteristics

Extensive calculation for the beam transport system, shown in Fig. 14, were made using the code OPTIK in an effort to optimize the transmission of the 60 MeV alpha particles from ORIC to the creep facility, while maintaining the desired spatial distribution on the specimen. Several preliminary experiments were performed to verify the predicted quadrupole focusing element settings and resultant beam intensity profiles. The measured settings were found to agree within 10% with the predicted settings, which is considered reasonable in view of the uncertainty in the beam emittance from ORIC.

Fig. 14. The Beam Transport System from ORIC to the ICF.

Beam profiles are measured using a scanning plate containing a slanted slit (for vertical scan) and a narrow vertical slit (for horizontal scan). A typical beam intensity profile during an irradiation creep experiment is shown in Fig. 15. The central region of the beam is seen to be reasonably well represented by a Gaussian distribution. Since it is important to minimize the variation in damage over the length and width of the specimen, it is desired that the variation in beam intensity over the specimen be limited to about $\pm 10\%$. For a Gaussian intensity distribution, this restriction limits the allowable transmission to approximately 50% in both the horizontal and vertical dimensions. The expected net transmission is approximately 40%, based on measurement of type shown in Fig. 15.

The actual beam profile, and hence transmission achieved during a given experiment, depends on the emittance of the beam from ORIC. Since this varies from one run to the next, it is necessary to measure the beam profile before each experiment. The beam is first aligned and the

Fig. 15. A Measured Beam Profile using a Diagonal Slit and a Faraday Cup. The beam stop is located before the switching magnet (see Fig. 14). The mask is the final beam-shaping aperture located 150 mms from the specimen.

approximate profile determined by means of a phosphor, using a TV camera to view the illuminated spot. The profile is then measured with a scanning plate located approximately 50 mm downstream from the specimen; the transmitted beam is measured in an evacuated, electron-suppressed Faraday cup. The vacuum must be better than $\sim 10^{-2}$ Pa to avoid spurious readings induced by ion currents. Final positioning of the beam is accomplished using the specimen temperature control unit by adjusting the beam position to minimize the amount of Ohmic heating required to maintain a given temperature. Fine-tuning of the beam profile is performed using the temperature gradient information from the infrared pyrometer.

During an irradiation creep measurement, the beam is intercepted by the cooling block, thus making it impossible to monitor the beam current using the Faraday cup. Therefore, an alternate system consisting of a parallel plate ionization chamber and current meter is used to monitor the beam current during the experiment. This chamber is initially calibrated against the Faraday cup readings obtained with the cooling block removed. A typical calibration curve is shown in Fig. 16. While

Fig. 16. A Calibration Curve for the Output of the Plasma Sensor vs Beam current to the Beam Stop ($I_{\text{beam stop}} \approx 2.5 I_{\text{specimen}}$).

this system is adequate to monitor the operation of the cyclotron, it cannot make an accurate measurement of the fluence through the specimen, since the specimen intercepts only a portion of the beam passing through the ionization chamber. A separate measurement of the fluence is made by determining the amount of power being deposited in the specimen. This is accomplished by means of a signal, proportional to the Ohmic power being delivered to the specimen, available from the specimen temperature controller; see the block diagram in Fig. 11 — note the BEAMP output. This signal, when nulled at beam-off conditions and fed to a voltage controlled oscillator, provides a pulse train whose frequency is proportional to the incident beam current and which can be counted to provide a measure of the integrated beam current. Additionally, the end-of-run fluence is determined independently by measuring the ^{56}Co produced in the specimen by the $^{56}\text{Fe}(\alpha, p3n)$ reaction; see Fig. 17. The average reaction cross section for 54–60 MeV alphas is taken²⁷ to be

Fig. 17. The relative γ -intensity measured for an Irradiated Type 316 Stainless Steel Specimen. The corrections arise from the extra intensity from those portions of the voltage leads which are irradiated.

$69 \pm 5 \times 10^{-31} \text{ m}^2$ and the intensity of the 1.237 MeV gamma ray (67.0% abundant), measured by a Ge(Li) spectrometer, is used to determine the amount of ^{56}Co present. Gamma-ray scanning of the specimen can also be used to check the uniformity of the irradiation.

The Data Acquisition System

The data acquisition system for the ICF consists of a diverse collection of instruments designed to monitor the conditions of the specimen and its environment. The analog information from each of the various transducers is digitized and made available to a microprocessor-based data logging system. A block diagram is shown in Fig. 18 and each of the various subsystems is described below.

Changes in the length of the specimen are measured by two linear variable capacitance transducers mounted symmetrically on either side of the specimen. A microprocessor-controlled or manually-controlled switch box allows the transducers to be read individually or, as an average of the two analogue signals. The experimentally achieved resolution is

Fig. 18. The Data Acquisition System.

approximately ± 20 nm, given the noisy electrical and mechanical environment and the long signal leads. Tests have shown that, within the experimental resolution, the transducers are not affected by the helium pressure in the chamber or by the intense radiation produced by the cyclotron beam. Early experiments, however, using an ASL 1055 bridge did show a sensitivity to ionization events within the helium-filled spaces inside the transducers.

Stress is applied to the specimen by means of a spring attached to the lower grip and driven by a Slosyn stepping motor. The micro-computer/controller is programmed to maintain a load within set bounds or to change it rapidly by pulsing the Slosyn motor. Changes in spring length are measured by an LVDT and the signal digitized in a $3\frac{1}{2}$ digit digital panel meter. The resolution of the system is approximately 0.05 N which corresponds to a stress resolution of 0.25 MPa for a $76 \mu\text{m} \times 2 \text{ mm}$ specimen cross section. Frictional forces (~ 0.2 N) in the drive system require that the desired load be approached in the same direction as that used for the calibration curve. The limit on the applied load is about 58 N, which corresponds to an applied stress of 380 MPa for the cross section noted above.

The temperature control system has two important $4\frac{1}{2}$ digit outputs which are regularly monitored. These are a voltage proportional to the heating current and a voltage proportional to the instantaneous error in specimen resistance from the setpoint. The temperature controller may be controlled by the microprocessor (or a larger computer) by having an error signal sent to the temperature controller and driving the specimen resistance to a new set point. Thus, controlled temperature fluctuations or ramps may be programmed rather easily.

The infrared pyrometer is mounted on a remotely positionable X-Y scanner, which is driven by two Slosyn stepping motors. The travel in each direction is monitored by potentiometers. The pyrometer may be moved manually or may be scanned through a series of stops by the microcomputer. The scanning routine allows 50 separate measurements to be made in less than 100 s.

The thermocouples are monitored in one of two ways. First, the ten most critical thermocouple signals (i.e., the specimen, grips, helium

temperature, standard resistor, cooling block, etc.) are input to a multiplexor (Keithley, Model 702) which is directed by the microprocessor to send the desired signal to a $4\frac{1}{2}$ digit nanovoltmeter (Keithley, Model 180). The settling time is rather long; the entire scan of these 10 thermocouples takes 20 s. The second scanning technique uses an ORNL-built multiplexor which accepts up to 64 thermocouple signals, each of which is read and digitally recorded at a rate of 0.5/s.

The parallel plate ionization chamber output is measured by a $3\frac{1}{2}$ digit panel meter to provide an estimate of the beam current. The beam power integrator, the other beam monitor, is able to count and output to 10^7 counts. These two beam signals are continuously recorded.

Each of the instruments discussed above, barring the 64-channel multiplexor for thermocouples, provides a binary coded decimal (BCD) output which must be recorded at fixed intervals. The system which was developed for reading and recording these data is built around the NDT-COMP 8 microcomputer²⁸ developed at ORNL. A sixteen channel by 24-bit multiplexor was designed to interface the various devices to the microcomputer. Address information and data transfers are made via 24-bit parallel I/O ports. Operator control and data output are via an ASR 33 teletype/paper tape punch. A 1 s^{-1} signal, generated from 60 s^{-1} line frequency, is connected to the microcomputer interrupt line, providing an internal realtime clock. One additional parallel port is used to output visual and audible alarms to indicate that one or more data points are out of an acceptable range.

The microcomputer memory consists of 4 K bytes of electrically programmable read-only memory (EPROM) plus 4 K bytes of random access memory. The operating program is stored on 3 K bytes of EPROM and is easily entered by means of a manual interrupt mounted on the front panel. On entry, the program will respond to any of seven single character commands entered from the keyboard. The commands are summarized in Table 2. During normal operation, commands "I", "L", and "C" are used to set up the desired data recording operations and the "S" command used to initiate the data recording operation. Upon receiving an "S" or "R" command, the system will read all requested channels,

Table 2. Keyboard Commands Recognized by the ICF Data Acquisition System

K	Echoes subsequent keyboard entries, for printing of operator messages on data printout. Exit by typing \$.
I	Initializes system by entering format and control information. System will prompt with channel number and wait for two-digit control word. The first character supplies information about the sign convention and whether or not this channel is to be tested against preset limits. The second digit gives the number of digits in each data word.
L	Limits desired by the user are entered and stored in RAM. System will prompt with channel numbers entered under command "I" and wait for upper limit (UL) or lower limit (LL) to be entered.
C	Changes scan parameters. System will prompt for number of channels (NCH), scan period in seconds (DEL), number of thermocouples (NTC), thermocouple channel number (CHTC).
R	Resumes scanning following some interruption in the scanning process. Preserves scan number and running seconds counter.
S	Starts scanning of all requested channels. This will clear the scan number and running seconds counter.
T	Displays time of day.

storing the data in RAM until the last channel has been successfully read, at which time the data are outputted to the teletype. The data inputs to the 8-channel digital multiplexor are available essentially continuously and can be read very rapidly (<1 msec), but the thermocouple scanner-voltmeter channel requires a delay of about 2 sec between each reading. This is not considered to be a serious drawback since the thermo-couple readings are generally slowly varying. Since no "handshake" signals are employed, each channel is read repeatedly until two subsequent readings are found to be identical. This reduces the probability that a device will be read during a data output/update cycle. Following a data record-output cycle, the system continuously monitors those channels which have been selected by the operator, compares their readings against their present limits, displays any out-of-limit channel numbers, and sounds an alarm if any selected channel is outside the present limits. This cycle is repeated after the scan period set by the operator has elapsed.

As mentioned earlier the microcomputer is also used to control the three Slosyn motors attached to the loading drive and attached to the X-Y scanning drives of the infrared pyrometer. This is accomplished by controlling the number of pulses to these motors. The feedback is the load cell readout for the loading drive and a counter total for the pyrometer positioner.

Since the available memory cannot support precision mathematical operations other than addition and subtraction, all data analyses are presently done offline, using punched paper tape from the teletype as the data transfer medium. It is currently necessary to process and plot the information using routines stored in the PDP-10 computer. Clearly, on-line processing capabilities will be required for future experiments; therefore, it is planned that an LSI-11 based system with storage and interactive capabilities be added to the data acquisition system in the near future.

SPECIMEN PREPARATION

The starting sheet material from which specimens are prepared is normally rolled, using carbide rolls, with intermediate anneals in a vacuum better than 6×10^{-4} Pa (5×10^{-6} torr) in a Ta foil wrap. Care must be taken to minimize grain growth following recrystallization. The ribbon specimens necessary for the experiments are sheared to approximate width, then ground to 2.00 ± 0.025 mm along the 20 mm gage length. The specimens are then scribed with labels designating the material, specimen number, nominal thickness, and batch.

Two pairs of voltage leads of 0.12 mm diameter stainless steel wires are attached by spot welding. One pair of leads is a backup set in the event that a wire comes loose. It is estimated that less than 2% of the specimen gage section is disturbed by this lead attachment. The lead spacing is currently set at 17 mm to avoid the difficulty of having a significantly different peak specimen temperature during beam off versus beam on conditions. This discrepancy, caused by different temperature profiles near the grips under the two conditions, is aggravated by wider

lead spacing. Further discussion on the effect of lead spacing is given later. Thermocouple leads, 50 μm Pt and Pt/10 Rh wires, are also spot-welded to the specimen; see Fig. 6.

The room temperature specimen resistance is measured to better than 100 ppm using a potentiometric system. A check on the resistance values is obtained by taking resistance measurements $R_{AC} - R_{AD} - R_{BC} + R_{BD}$, where A, B, C, and D are the four voltage leads. The value of this quantity is a good indicator of the validity of the measurements. A master resistance versus temperature curve for each material studied is generated by the Physical Properties Group at ORNL. Exploratory experiments have been performed to determine the stability of each material's resistivity as a function of thermal treatment. In some cases it is clear that a pre-anneal is necessary to stabilize the R vs T curve; in others this is not necessary.

The emissivity of each material to be studied must be at least partially investigated. A small (30 mm diameter) furnace has been adapted to the irradiation creep facility to allow specimens to be held at a known temperature while the infrared pyrometer output is followed with time. This gives a first approximation to the original emissivity as well as its time dependence. In some cases the emissivity may be estimated rather closely on the basis of tarnish darkness and color. It is convenient for testing certain materials to preoxidize in air.

Metallography must be performed on each material to be tested. It is necessary to know the number of grains across the specimen thickness to allow an estimate of the degree of unconstrained grain boundary sliding. For the case where a grain boundary completely traverses the specimen thickness grain, boundary sliding can occur without the normal degree of plastic accommodation. This would lead to a different overall creep rate of the thin specimen than is observed in bulk material; e.g., see Matlock and Nix.²⁹ Figure 19 shows a metallographic cross section of 20% cold-worked type 316 stainless steel specimen having a grain size of about 10 μm .

The normal procedure before an irradiation creep experiment is to creep the specimen without beam at the desired load and temperature to provide a baseline thermal creep rate against which to compare the irradiation creep. This also allows the intrinsic resistivity values and the

Fig. 19. The Metallographic Cross Section of a 20% Cold-Worked Type 316 Stainless Steel Creep Specimen — Grain Size $\sim 10 \mu\text{m}$.

emissivity to stabilize. After the experiment gamma spectrometry, chemical analysis, metallography, and electron microscopy analyses are normally performed along with other more discretionary measurements such as density, hardness, post-test thermal creep rate, tensile strength, etc.

RADIATION EFFECTS OF 60 MeV ALPHAS

There are several stages in which the damage processes of light ions must be examined to allow comparison with damage produced by fast neutrons. These are (i) the initial defect production in terms of the localized displacement events, (ii) the local recombination or cascade annealing, and (iii) the subsequent condensation of the point defects on existing microstructural features or in the evolution of new features. Each of these subjects contains unknowns and uncertain models which make a rigorous comparison of light ion versus fast neutron damage difficult. However, we are able to evaluate qualitatively the differences in behavior in several areas.

The number of displacements per atom, the primary damage unit, is calculated in two parts. The first part arises from elastic scattering processes. The elastic scattering contribution, which is strongly peaked at small scattering angles and, hence, small energy transfers is shown in Fig. 20 as the differential scattering cross section versus recoil energy for 60 MeV alphas on Fe. The curve shown is calculated using an optical model code for scattering angles greater than 5° ($E_{\text{RECOIL}} = 28 \text{ keV}$) and extrapolated to a recoil energy of 45 eV using Rutherford scattering [$d\sigma/d\Omega \propto \csc^4(\theta/2)$].

The second part of the damage cross section arises from inelastic processes. Given the large margin by which the 60 MeV alpha particles exceed the Coulomb barrier for the target atoms ($\sim 10 \text{ MeV}$), the inelastic events may not be neglected. The nonelastic differential cross section is also shown in Fig. 20. The shape of the curve is based on measurements of charged particles emitted in the bombardment of ^{54}Fe with 59 MeV alphas.^{30,31} This cross section is dominated by the $(\alpha, 2p)$ and $(\alpha, \alpha'x)$ reactions, where x represents unobserved light ions emitted in the decay of the highly excited compound nucleus. For the first reaction the protons were assumed to be emitted isotropically and with equal energy, and for the second reaction the emitted light ions were ignored. Given these approximations the detailed shape of the nonelastic cross section may be in error, but the general shape, in particular the

Fig. 20. The Differential Cross Section vs Recoil Energy for 60 MeV Alphas on ^{56}Fe .

peaking at 4 MeV, should be accurate. The nonelastic cross sections are normalized to the total reaction cross section, $1.65 \times 10^{-28} \text{ m}^2$, predicted by the optical model calculations.

Thus, from above we have the probability of a given recoil energy. At this point we must consider the energy losses through electronic excitation as the recoils move through the lattice. The accepted procedure^{32,33} is the use of the LSS theory which is used to predict the damage efficiency of recoil of given energy, E_{RECOIL} . For iron this damage efficiency is given by

$$\mu = 1 + 0.0713 E_{\text{RECOIL}}^{1/6} + 7.41 \times 10^{-6} E_{\text{RECOIL}}^{3/4} + 9.017 \times 10^{-7} E_{\text{RECOIL}}.$$

The differential cross sections were added and multiplied by this factor to give the damage energy spectrum shown in Fig. 21. In Fig. 22 is shown the damage energy integrated up to a given recoil energy.

An important factor to be noted from Fig. 22 is that most of the damage from 60 MeV alphas occurs from high-energy recoils ($E > 1 \text{ MeV}$).

Fig. 21. The Damage Energy Spectrum for 60 MeV Alphas on ^{56}Fe , Including Nonelastic Events. These data include reductions arising from electronic losses (see Refs. 32 and 33).

Fig. 22. The Calculated Damage Energy vs Upper Integration Limit for Primary Recoil Energy for 60 MeV alphas on ^{56}Fe .

Given the significant probability of subcascade formation for $E_{\text{RECOIL}} > 100 \text{ keV}$ ³⁴ which increases with energy, it is reasonable to expect a qualitative similarity in damage between these subcascades and the damage produced during fast reactor irradiation. The majority of fast-reactor irradiation damage is caused by primary recoils having $E_{\text{RECOIL}} > 100 \text{ keV}$. Furthermore, the damage predicted for materials subjected to 14 MeV neutrons in a fusion reactor should be matched even more closely by 60 MeV alpha damage, perhaps making the ICF a reasonable simulator for irradiation creep under fusion reactor conditions as well as fast-reactor conditions.

Once the cascades are produced, close-pair recombination and cascade annealing occur which decrease the number of point defects available. Estimates of the number of defects available after annealing²⁵ indicate that only about 20% of the original point defects survive cascade annealing in a 0.1 MeV cascade in nickel at temperatures where interstitials are very mobile. The point defects which escape the cascades are then available to contribute, for example, to irradiation creep processes.

A review of irradiation creep theories will not be given here, since recent reviews have been published elsewhere.^{7,35} However, at this time little direct evidence exists to support the two dominate theories: Stress Induced Preferred Absorption (SIPA),^{36,37} or I-creep.³⁸ The former is a climb mechanism in which dislocations of a given orientation climb by the condensation of free interstitials at a rate which is dependent on the orientation of the Burgers vector with respect to the applied stress. The I-creep mechanism is one in which the net climb of dislocations (occurring only in the presence of swelling) leads to unpinning of the dislocations and subsequent glide. These are independent, but by no means exhaustive mechanisms and as yet are unsubstantiated by controlled experiments. It is toward an understanding of these or alternative mechanisms and associated chemical or microstructural effects that the irradiation creep experiments in the ICF will be directed.

PERFORMANCE OF THE ICF

The ICF is one of the most, if not the most, precise length measuring device for creep measurements at elevated temperature. Figure 23 gives a portion of a thermal creep curve for type 316 stainless steel tested at 810 K and 170 MPa. The elongation rate is about 10^{-2} nm/s, corresponding to a strain rate of $4 \times 10^{-10} \text{ s}^{-1}$ (40% strain in 30 years). Thus, for thermal creep measurements the ICF is extremely accurate.

For irradiation creep where the beam provides much of the heat input, the creep resolution is controlled largely by beam stability. The reason is related to the position of the voltage leads. Since the temperature controller is designed to keep the resistance constant between the voltage leads and since the length measurement is made over the entire length of the specimen, a change in temperature of the length adjacent to the grips, L_G , but outside the voltage leads, will lead to a change in measured length; see Fig. 24. In Fig. 24 is shown (a) the ideal temperature profile, (b) the condition with the voltage lead

Fig. 23. A Portion of a Thermal Creep Curve for Type 316 Stainless Steel at 810 K, 170 MPa, Obtained in the ICF.

Fig. 24. Schematic Showing the Temperature Profiles for (a) the Ideal Case, (b) the Case for L_G Large (or Voltage Lead Spacing Small), and (c) the Case for L_G Small (or Voltage Lead Spacing Large).

spacing small, and (c) with the lead spacing large. In (b) a change in length occurs as the beam current drops, since the electrical current heats the length L_G to a greater extent. In (c) a change in beam current results in a length change because of an overall change in temperature, as shown.

Since it is desirable to work with many materials of differing thermal conductivities and at different temperatures and beam currents, a solution which optimizes one set of conditions is not practical. The approach taken, which has worked satisfactorily, is to monitor the heating current (which is inversely proportional to the beam current) and find the short-term correlation coefficient between length and heating current. Then the raw length data may be adjusted for variations in beam current. This procedure has been applied to the data shown in Figs. 25 and 26 from an irradiation creep experiment. With this technique the irradiation creep rate resolution (in a period of 4 h) is $\sim 10^{-9} \text{ s}^{-1}$. For this experiment, the correction factor, λ , of

Fig. 25. Length vs Time Data for an Irradiation Creep Experiment on 20% Cold-Worked Type 316 Stainless Steel at 710 K, 810 K, and Several Stresses.

Fig. 26. The Strain Rates Measured During Irradiation Creep of 20% Cold-Worked Type 316 Stainless Steel Using the ICF.

about 2 μm per ampere heating current was found to reduce the short-term data scatter most effectively:

$$L_{\text{corrected}} = \lambda \cdot \Delta I_{\text{heating}} + L_{\text{measured}}$$

Another small correction was made to the length measurement to account for changes (~ 1 K) in grip temperature. Here the correction factor is 150 nm/K for both grips. It is likely that more experiments will improve the control of unwanted temperature variations and the creep rate resolution. For further analysis and discussion of these results, see Reiley, et al.²⁴

A typical beam current (electrical) measured on the specimens during an experiment is about 0.2 A/m² or a total of 8 μA . For the damage energy cross section given earlier ($1.38 \times 10^{-22} \text{ m}^2 \cdot \text{eV}$) this results in a damage rate of 2.2×10^{-6} dpa/s, *very similar to damage in a fast reactor* ($\sim 10^{-6}/\text{s}$). Slightly higher beam currents may be used; however, the foil separating the chamber from the beam line was broken during an experiment at 0.25–0.3 A/m². This seems to be rather close to the cooling rate limit for the present cooling block design, and as well, to the cyclotron limit for long-term beam production. Therefore, a practical limit in displacement rate is presently about 3×10^{-6} dpa/s.

One other experimental feature observed in our early experiments is the evolution of helium in the stainless steel tested. A specimen irradiated to a dose of 2.1×10^{22} α/m^2 was found to contain 0.5 ppm He, resulting in a calculated He/dpa of about 7. This value is in a range similar to that estimated for the first wall of current fusion reactor designs; ~ 20 for stainless steel, ~ 5 for refractory alloys.³⁹ The helium arises from transmutation reactions and/or from backscattering from the cooling block.

SUMMARY

From the standpoint of the original objective of this project, it appears that the ICF satisfies the criteria mentioned earlier. That is, using the ICF we may test bulk material (or material which exhibits the mechanical properties of bulk material) in a radiation environment which simulates the damage processes caused by fast neutrons. There is

insufficient data at present to make a reasonable comparison between our light ion results and neutron results; however, the apparatus can impose experimental conditions allowing such a comparison. The apparatus has demonstrated high accuracy and resolution during irradiation creep measurements, qualities which are likely to improve with operating experience and on-line data analysis capabilities. We are now in a position to perform experiments to understand mechanisms of irradiation creep and other effects of irradiation. Experiments are currently planned on pure metals and alloys in the preirradiated or unirradiated condition.

Another quality of the ICF is the very high resolution for creep processes without irradiation, especially for imposed transient conditions in stress or temperature. This is an area basic to the interpretation of thermal creep mechanisms and one which allows unique experiments to be performed using the ICF, at times when the particle beam is not available.

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