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Cooperative Radiation Effects Simulation Program
Semiannual Progress Report for the Period
1 April to 31 August 1976

L. A. BEACH, COORDINATOR
Radiation Technology Division

and

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Engineering Materials Division

February 1977

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)		
<p>The Cooperative Radiation Effects Simulation Program (CORES) is a collaborative effort of the Engineering Materials and Radiation Technology Divisions of the NRL Materials and General Sciences Area. The goal of the research is to provide the theoretical and experimental bases for understanding the mechanisms of void nucleation, as well as a theoretical insight into energy deposition processes. In this the Van de Graaff and Cyclotron are used to simulate rapidly the radiation damage produced over long operating periods in reactor neutron environments.</p> <p style="text-align: right;">→ (Continues)</p>		

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Irradiation-induced creep	Radiation-induced displacements
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Neutron radiation damage	Temperature dependence of void formation
Nickel alloys	Transport theory
Nickel ion beams	Vacancies in metals
Precipitate stability	Van de Graaff bombardments
Primary knockons	Voids
Radiation damage	Void formation
Radiation damage simulation	Void nucleation
Radiation-induced defects	Void growth

20. Abstract (Continued)

Progress for the period 1 April - 31 August 1976 includes the continuation of studies on the stability of Ni₃Al precipitates in nickel under ⁶⁸Ni⁺ ion bombardment. Specimens examined by transmission electron microscopy after irradiation at different dose levels and dose rates showed a modification of the precipitate structure. The precipitate size distribution in the high level, high dose rate specimens decreased in size but increased in density. In the low level, low dose rate specimens, the precipitates developed contrast features but retained their size distribution. In another study a computer program was developed to convert the size of an arbitrary projection of a polyhedral void to a characteristic edge length for the void. These edge lengths can be used to calculate void volumes from the correct formula for each void shape. The microstructure of cold worked, high purity nickel has been investigated following ion-simulated irradiation-induced creep with 22-MeV alpha particles. A model based on the climb-controlled glide of dislocations over dispersed obstacles was found to be consistent with microstructure results and the experimental creep rate.

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COOPERATIVE RADIATION EFFECTS SIMULATION PROGRAM

Semiannual Progress Report
for the period:

1 April 1976 - 31 August 1976

PROGRAM DESCRIPTION

The Cooperative Radiation Effects Simulation Program (CORES) was initiated voluntarily by five Branches from the Engineering Materials and Radiation Technology Divisions of NRL on the basis of their common interests in the problems of simulating radiation damage in metals. The program promotes the exchange of information, discussion of problems, and the pursuit of collaborative research efforts. Semiannually a written report is prepared containing those portions of the work of the participating Branches which are judged to be of interest to the damage simulation problem. Since research findings which apply to the objectives of one sponsor may also be of interest to others the overall progress related to damage simulation is included in the written report. Several of the participating Branches have independent programs on other aspects of the radiation damage problem; when results obtained in these programs are judged to be of interest to CORES participants they may also be included, informally, in the CORES program reviews.

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COOPERATIVE RADIATION EFFECTS SIMULATION PROGRAM

Semiannual Progress Report
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SUMMARY

I. HEAVY ION DAMAGE STUDIES

A. Dose Rate Effects in a Precipitation Hardened Nickel-Aluminum Alloy

A Ni-14 at. % Al alloy containing γ' (Ni_3Al) precipitates was irradiated at 725°C with 2.8-MeV $^{58}\text{Ni}^+$ ions to determine precipitate stability under irradiation. The doses examined were 0.81, 2.5, and 8.1 displacements per atom (dpa) at both of two dose rates: 4.4×10^{-2} dpa/sec and 4.4×10^{-4} dpa/sec. Specimens were examined by transmission electron microscopy. The precipitates in the lower-dose-rate samples were seen to develop complex contrast features after irradiation but to retain their original size. The original precipitates in the higher-dose-rate samples developed a ragged appearance around the edges as if pieces were being dissolved, and a second smaller class of precipitates appeared in the matrix, and by 8.1 dpa the entire original precipitate size distribution had been converted to the new precipitates. These differences due to dose rate are not explained by current models of the behavior of precipitates under irradiation.

B. Measurement of Void Size by Transmission Electron Microscopy

A computer program has been developed to convert the size of an arbitrary projection of a polyhedral void to a

characteristic edge length for the void. The use of the program requires little additional effort and can significantly improve the accuracy of void size and volume measurements made by transmission electron microscopy. To use this method on a distribution of truncated regular polyhedra, void sizes are first measured in terms of the diameters of inscribed or circumscribed circles on the void images. The foil orientation, obtained from the corresponding diffraction pattern, and an estimate of the degree of truncation are then used in the program to convert the circle diameters to characteristic edge lengths for the given void shape. These edge lengths can then be used to calculate void volumes from the correct formula for this void shape. In the cases of truncated octahedra and edge-truncated cubes, the inscribed circle was found to be the better diameter to measure, since its relation to the edge length was less sensitive to the degree of truncation.

II. LIGHT ION DAMAGE STUDIES

A. Microstructure and Mechanism of Ion-Simulated Irradiation Induced Creep of Nickel

The microstructure of cold worked, high purity nickel has been investigated following ion-simulated irradiation-induced creep with 22-MeV deuterons and 70-MeV alpha particles. The irradiations were conducted at 224°C, at stresses between 170 and 345 MPa, and at displacement rates between 13 and 30×10^{-8} displacements per atom per second. Transmission electron microscopy (TEM) procedures were used to prepare, observe, and photograph the microstructure of the ion irradiated uniaxial creep specimens and companion unirradiated specimens.

The microstructural results were evaluated in terms of the theoretical mechanisms proposed for irradiation-induced creep and the previously reported creep simulation results for nickel by Hendrick et al. A model based on the climb-controlled glide of dislocations over

dispersed obstacles was found to be consistent with the microstructural results and the experimental creep data.

COOPERATIVE RADIATION EFFECTS SIMULATION PROGRAM

Semiannual Progress Report
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1 April 1976 - 31 August 1976

INTRODUCTION

L. A. Beach, Coordinator, Materials Modification & Analysis Branch, Radiation Technology Division.

The interdivisional cooperative research activity represented by this and prior reports has been successful and productive. The primary goal was to use simulation techniques to advance the understanding of neutron damage of materials for advanced nuclear systems. Light and heavy ion bombardment techniques are used to represent neutron damage in order to accelerate research and to permit an evaluation of critical parameters which might not be feasible using nuclear reactors at test facilities. This simulation of neutron damage by ion bombardments is strengthened by advancements in the theory of Atomic Collisions. These fundamental studies conducted within the CORES program are complemented by parallel applied experiments using nuclear reactors. The productivity and the maturity of the cooperative program is illustrated by the technical productivity listed in the last section.

Note: Manuscript submitted February 3, 1977.

RESEARCH PROGRESS

I. HEAVY ION DAMAGE STUDIES

A. Dose Rate Effects in a Precipitation Hardened Nickel-Aluminum Alloy

(J. E. Westmoreland and P. R. Malmberg, Materials Modification & Analysis Branch, Radiation Technology Division, and J. A. Sprague, F. A. Smidt, Jr. and L. G. Kirchnert, Thermostructural Materials Branch, Engineering Materials Division)

Introduction

The precipitate microstructure influences the mechanical properties of materials; hence, for materials operating in a radiation environment the stability of precipitates under irradiation is important. Heavy-ion irradiations for the simulation of neutron damage requires an awareness of any dose-rate effect because of the compressed time frame of these irradiations, and previous high temperature, high-dose-rate irradiations¹ suggest that dose rate is likely to be an important variable. Nickel ion irradiation of γ' precipitates in the same alloy employed in this experiment was previously studied² by transmission electron microscopy as a function of irradiation temperature and as a function of dose. At the high dose rate (see Experimental Procedures) of this experiment the largest effect on the precipitate microstructure was seen at 725°C, the highest irradiation temperature employed where the original 400 Å size precipitates were replaced by 80 Å size precipitates. At lower temperatures the precipitate structures were less

[†] Ph.D. Candidate of the Department of Nuclear Engineering, University of Wisconsin, in cooperative program with NRL.

well defined and took on a ragged appearance with a wide spread in sizes and a smaller precipitate formed in the matrix between the original ones. These observations were in contrast to an earlier study³ in which the γ' precipitates were observed to decrease in size in aged material and grow to the same size in solution-treated material. A model was proposed³ which predicted that an equilibrium precipitate size which would be a function of dose rate would be achieved after a dose of a few dpa. This work was recently reviewed by Hudson.⁴ The present study was undertaken to investigate the influence of dose rate on the γ' precipitate size distribution at 725°C, and to examine more closely the early stages of the effects of heavy-ion irradiation on these precipitates.

Experimental Procedures

Preparation of the Ni-Al alloy used in this investigation has been described previously.² The nickel-ion irradiations were performed with a 2.8-MeV $^{58}\text{Ni}^+$ beam from the NRL 5-MV Van de Graaff accelerator. The specimens were irradiated at 725°C to doses equivalent to 0.81, 2.5, and 8.1 displacements per atom (dpa), and at peak displacement rates of 4.4×10^{-2} dpa/sec-high dose rate (HDR) and 4.4×10^{-4} dpa/sec-low dose rate (LDR). The deposition of initial damage energy by the nickel ions as a function of distance into the target foils was calculated with the E-DEP-1 computer code.⁵ For 2.8-MeV $^{58}\text{Ni}^+$ ions, this calculation yielded a peak energy deposition for elastic collisions of 1.16 MeV/ μm at a depth of 5400 Å. A Kinchin-Pease secondary displacement model with an efficiency of 0.8 and a displacement energy of 40 eV was used to obtain dpa values. After ion bombardment, 4000 ± 500 Å of the front surface of each sample was removed using a laser interferometric polisher.⁶ The front face was then masked off, and the sample was polished to perforation from the rear surface with one jet of a semiautomatic dual-jet electro-polisher. Specimens were examined with transmission electron microscopy in a JEM-200A electron microscope operated at 200 kV by using the {100} class of super-

lattice spots for imaging. This method of imaging was chosen because the small mismatch of γ' and the matrix does not yield sufficient contrast to produce well defined images. A particle size analyzer was used to characterize the precipitate size distribution.

Results

Definite dose-rate effects on the precipitate microstructure were observed in this experiment. The unirradiated precipitate microstructure will be described, followed by that of the HDR samples for each dose, and then the LDR samples for each dose.

The Ni₃Al precipitates of this investigation prior to irradiation are cuboidal with {100} faces and a mean cube edge of about 400 Å. The standard deviation of the size distribution is about 100 Å. Figure 1 shows these precipitates prior to irradiation.

Figure 2 shows micrographs of the HDR 2.5 dpa sample. The precipitate microstructure is typical of the appearance of that in both the 0.81 and 2.5 dpa samples. The precipitates in the HDR cases developed somewhat irregular shapes and contrast features giving them a 'fractured' or 'ragged' appearance as if they were 'dissolved' around the edges. However, within the experimental error no size change was seen in the larger size precipitates in the 0.81 and 2.5 dpa HDR samples. In the HDR-irradiated samples a fine precipitate structure appeared between the larger precipitates even at the lowest fluence (0.81 dpa). With increasing HDR fluence these small precipitates became more evident until at 8.1 dpa the original 400 Å precipitates had been completely replaced by a smaller and higher density precipitate structure, as shown in Fig. 3, where the precipitate mean size is about 50 Å. One may recall the results of Kirchner *et al.*² at 20 dpa where a result similar to Fig. 3 was obtained but with a precipitate mean size of about 80 Å.

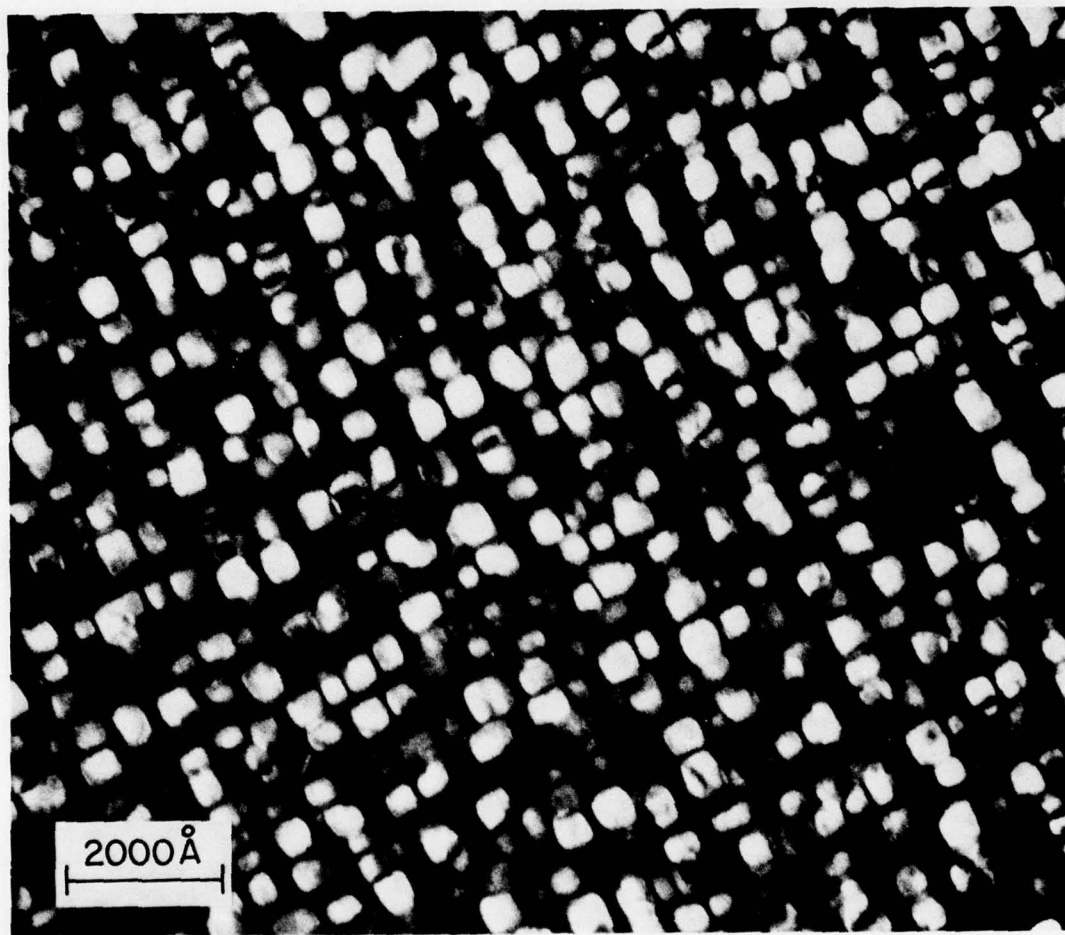


Fig. 1 — Ni_3Al precipitate microstructure in Ni-Al alloy before irradiation. $g = \{001\}$ class.

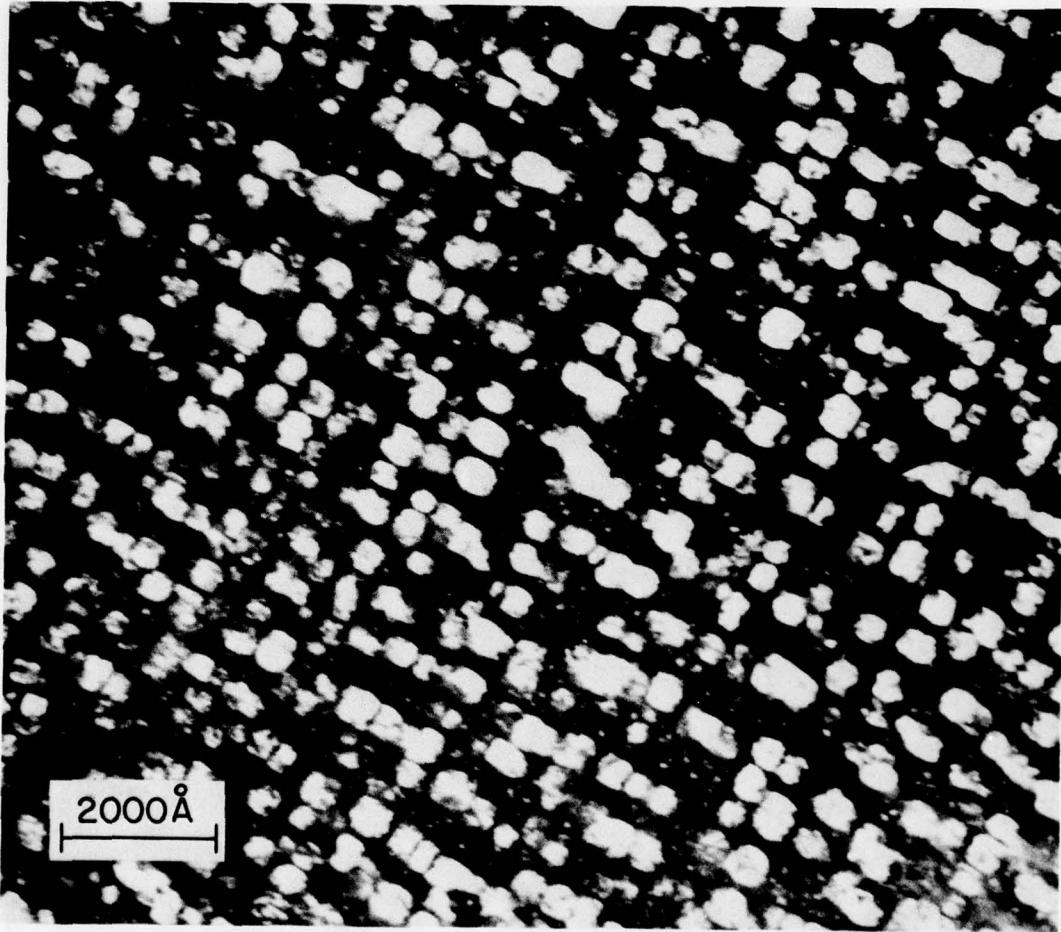


Fig. 2 — Ni₃Al precipitate microstructure of Fig. 1 after irradiation with 2.8-MeV ⁵⁸Ni⁺ ions to a dose of 2.5 dpa at a dose rate of 4.4×10^{-2} dpa/sec. $g = \{001\}$ class.

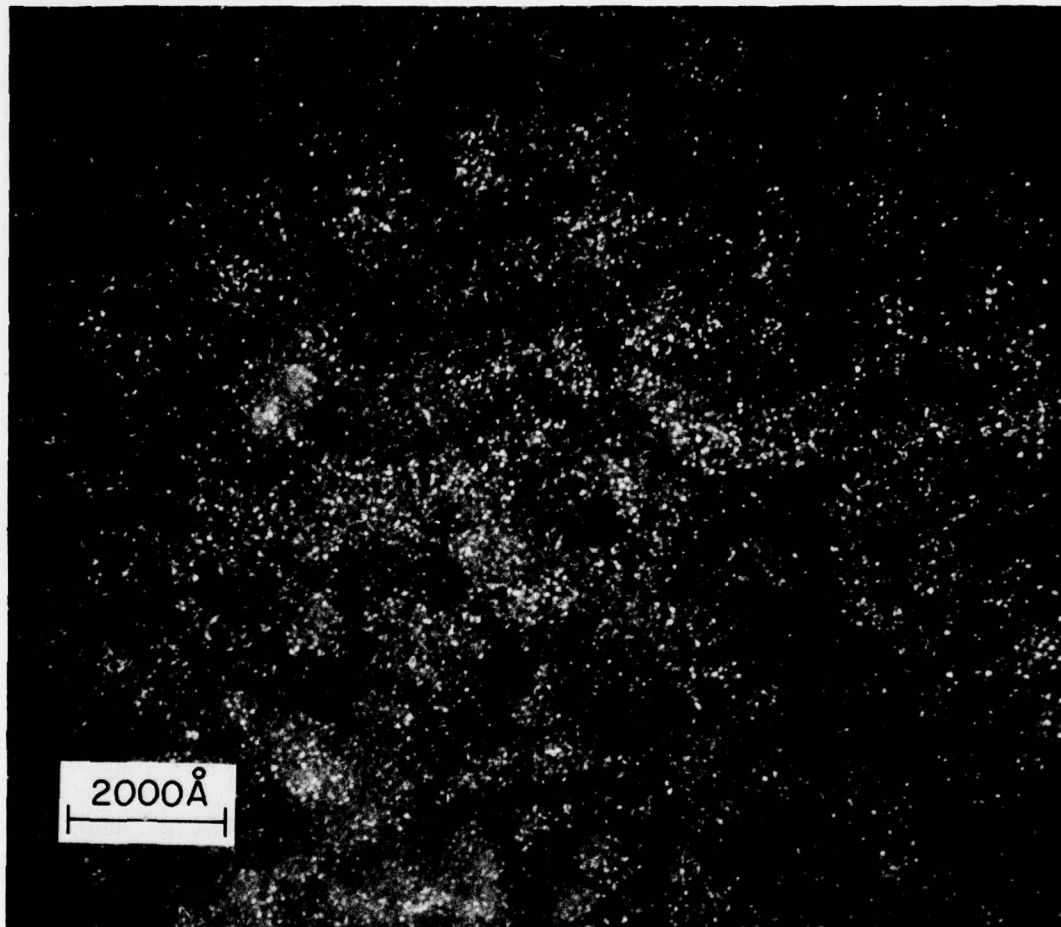


Fig. 3 — Ni₃Al precipitate microstructure of Fig. 1 after irradiation with 2.8-MeV ⁵⁸Ni⁺ ions to a dose of 8.1 dpa at a dose rate of 4.4×10^{-2} dpa/sec. $g = \{001\}$ class.

Figure 4 shows micrographs of the LDR 2.5 dpa sample. The precipitate microstructure is typical in appearance of those in both the 0.81 and 2.5 dpa samples. The LDR-irradiated samples developed contrast features during the irradiation, but retained their original size. No fine precipitation was observed in the matrix of the LDR-irradiated samples, even at 8.1 dpa; the appearance of the precipitate microstructure of the 8.1 dpa LDR specimen was similar to Fig. 4.

Measurements of the Ni_3Al precipitate microstructural parameters before and after irradiation are summarized in Table 1. The scatter in the mean size is believed to be due both to the relatively small number of precipitates counted and to the difficulty in sizing the precipitates.

Discussion

There is little direct information in the literature on the effect of dose rate on precipitate stability under irradiation. That information which might perhaps be useful will be summarized briefly and contrasted with the quite different behavior of the precipitate microstructure observed here under the factor of one hundred difference in heavy-ion dose rate employed. Some possible implications of these results will then be considered.

The relationship of the HDR-type irradiations employed in this experiment to other work has been discussed in more detail elsewhere.² The first reported study³ of the response of Ni_3Al precipitates to irradiation showed that in an alloy similar in composition to the one used in the present study the precipitates (γ' - Ni_3Al) were observed to break up during heavy-ion irradiation at all temperatures above 325°C to form a population of smaller precipitates. Solution-treated material was observed following the same heavy-ion irradiation to have formed precipitates of the same size. Based on these data, a model was proposed³ in which an equilibrium size precipitate was formed as a consequence of a kinetic equilibrium between the dissolution

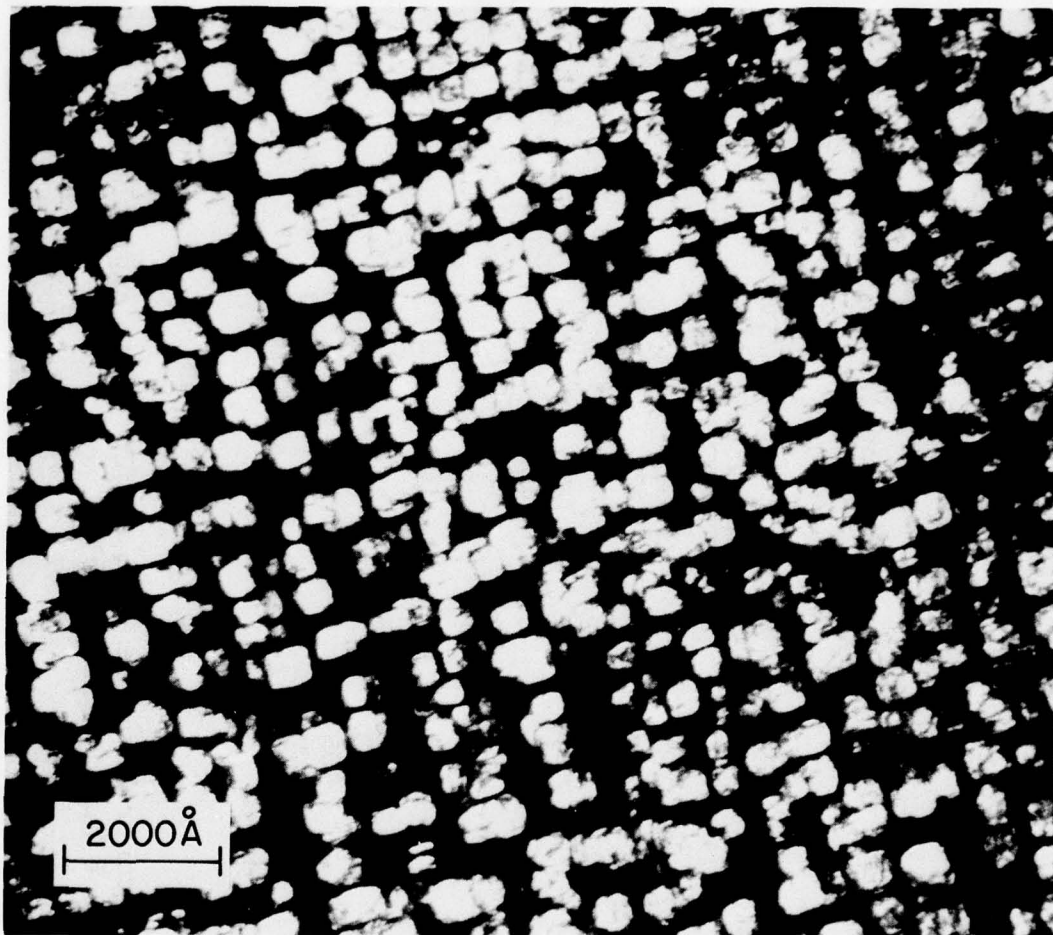


Fig. 4 — Ni₃Al precipitate microstructure of Fig. 1 after irradiation with 2.8-MeV ⁵⁸Ni⁺ to a dose of 2.5 dpa at a dose rate of 4.4×10^{-4} dpa/sec. $g = \{001\}$ class.

Table 1
 Characteristics of Precipitate Size Distributions in an Irradiated Ni-Al Alloy

Dose (dpa)	Dose Rate	Mean Size (Å)	Standard Dev. of Size Distribution (Å)	Number of Precipitates Counted
0 ^a	Thermal Control	396	109	573
0	Thermal Control	402	103	628
0.81	HDR	415	98	590
0.81	LDR	411	118	495
2.5	HDR	436	95	642
2.5	LDR	420	107	744
8.1	HDR	50	20	642
8.1	LDR	405	100	525

^aTwo micrographs of one control sample held at 725°C for approximately seven hours, the time to irradiate the 8.1 dpa LDR sample.

of volumes of precipitate near the precipitate surface when disordered by a displacement cascade or replacement collision sequence and the growth of the precipitates as a consequence of radiation enhanced diffusion. The occurrence of precipitation during the irradiation and the effect of precipitate density on the final equilibrium size was acknowledged³ but not incorporated into a predictive model.

The results of the present experiment show that in this case the behavior of precipitates under irradiation is more complex than the proposed model³ as the following effects due to dose rate alone are observed:

1. In the HDR samples examined new precipitates nucleate and grow in the matrix between the original precipitates.

2. The HDR irradiation causes areas at the "edge" of the image of the original precipitate to go completely out of contrast, that is, to appear to be dissolved. That this is the case is confirmed by the complete conversion of the precipitate size distribution by the 8.1 dpa HDR dose.

3. In the LDR samples examined complex contrast features associated with the precipitates are observed.

Whether one or more dissolution processes are operating is not yet clear. A model which will explain the present experimental results very likely must include one or more radiation enhanced nucleation mechanisms, homogeneous or inhomogeneous, as well as various dissolution processes.

Conclusions

A study has been performed of dose-rate effects observed in the irradiation at 725°C of a Ni-14 at. % Al alloy containing Ni₃Al (γ') precipitates. A factor of

100 difference in the rate of 2.8-MeV $^{58}\text{Ni}^+$ ions was employed to give dose rates of 4.4×10^{-2} dpa/sec (HDR) and 4.4×10^{-4} dpa/sec (LDR).

1. The precipitate size distribution in samples HDR-irradiated decreased from an average value of about 400 Å before irradiation to a value of about 50 Å after a dose of 8.1 dpa.

2. The precipitates in HDR-irradiated samples developed with increasing dose an increasingly ragged appearance. Fine scale precipitation occurred in the matrix between the larger precipitates. The precipitates in LDR-irradiated samples developed contrast features which were distinctly different in appearance from the HDR-irradiated precipitate microstructures. No fine scale precipitation was observed in the LDR-irradiated matrix.

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B. Measurement of Void Size by Transmission Electron Microscopy

(J. A. Sprague, Thermostructural Materials Branch,
Engineering Materials Division)

Introduction

As noted in many studies of voids in metals, these cavities often form as crystallographic polyhedra, in response to the variation of surface energy with surface orientation. The more commonly observed shapes include truncated octahedra,¹ cubes,² truncated cubes,^{3,4} right hexagonal prisms,⁵ and rhombic dodecahedra.⁶ The quantitative characterization of a distribution of voids should ideally include measurement of a characteristic dimension for the void shape in question and the correct translation of that dimension to the void volume. The most straightforward approach to this problem is to take all micrographs near a low-order orientation for which the characteristic length (e.g., the edge length of a cube) may be directly measured. In studies that involve measuring a large number of voids in many different specimens, however, this approach may not be convenient. This is especially true for large-grained specimens with limited thin areas, in which the foil normal may not be close to the desired orientation. As pointed out in the last CORES Report,⁷ variation in foil orientation can cause significant errors in measured void size, if one does not properly account for the variation.

The calculations presented in this report were undertaken to provide an easily applied method for relating the size of a void projection in some arbitrary orientation to a characteristic dimension of the void itself. Two shapes were chosen for the initial calculation, octahedra with $\{111\}$ faces, truncated by $\{100\}$ planes; and cubes with $\{100\}$ faces, truncated along the edges by $\{110\}$ planes. The technique, however, is fairly general, and the computer program developed for these calculations can be easily applied to most semi-regular polyhedra.

Method of Calculation

Although it would be possible to derive analytical expressions for the sizes of specific planar projections of a given polyhedral shape, the aim of this work was to develop a method for easily determining the sizes of a variety of polyhedra. The calculations, therefore, were written as a BASIC - language computer program to run on the small desktop computer used for other void analyses. The main features of the calculations are schematically shown in Fig. 1. The coordinates of the corners of the polyhedron are first determined in a coordinate system with x , y , and z parallel to the principal crystallographic axes. The polyhedron is then rotated so that the desired $[h k l]$ axis is parallel to the z -axis. The x - y coordinates of the corners then determine the $[h k l]$ projection of the solid shape. The diameters of inscribed and circumscribed circles on this projection are finally calculated. These two measurements of size were chosen to correspond to the use of a particle size analyzer with a variable-diameter circular light spot for sizing objects. The ratio of one of these diameters to a characteristic length of the polyhedron provides the required conversion factor for the $[h k l]$ orientation for calculating the diameter/edge length ratios.

The program, along with limited documentation, is available on request from the author. It was written specifically for a Hewlett-Packard Model 9830 with 3808 words of memory. All but a few of the statements, however, are fairly standard BASIC, and the program should run with only minor modification on most BASIC - language systems.

Results and Discussion

Two polyhedra were used as test calculations: an octahedron with $\{111\}$ faces, regularly truncated on the points by $\{100\}$ planes; and a cube with $\{100\}$ faces, regularly truncated on its edges by $\{110\}$ planes. The definitions of degree of truncation for these figures are illustrated in Fig. 2. For the octahedron, the truncation was defined as the fraction of the diagonal removed from

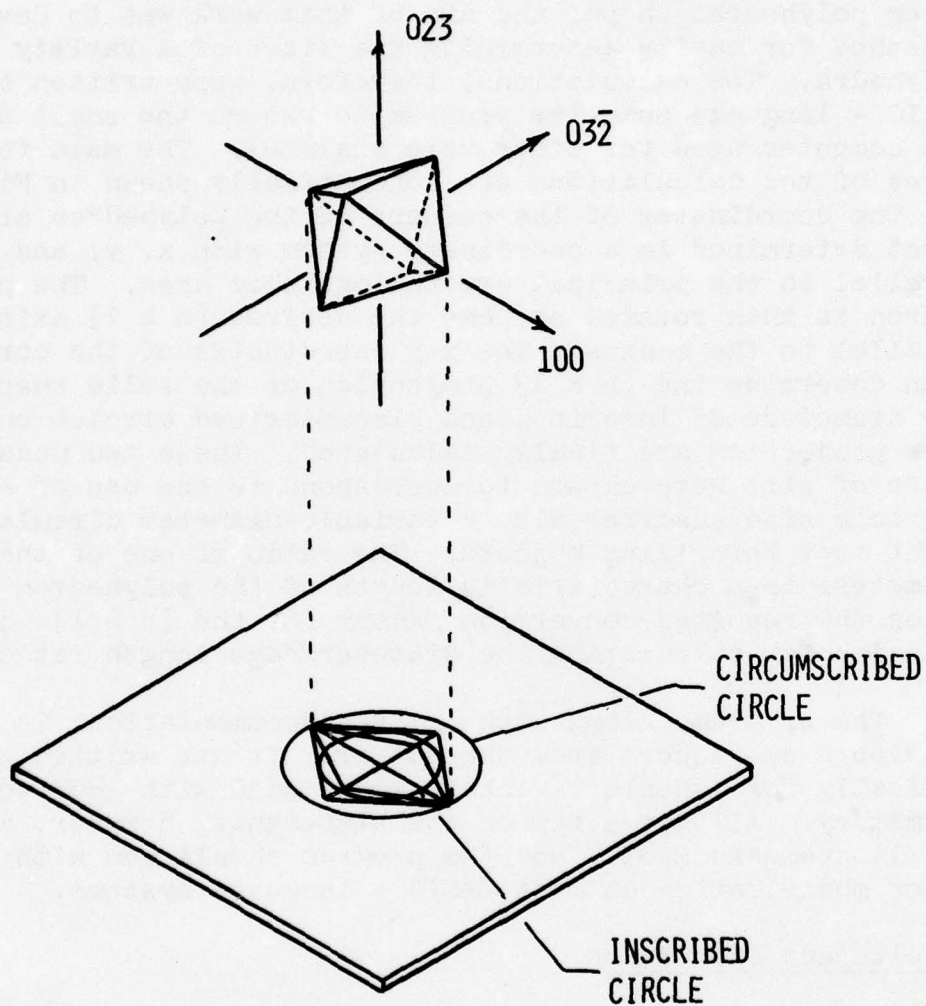


Fig. 1 - Schematic of the calculation of void size images, illustrated by the [023] projection of a simple octahedron. Void is rotated so that the desired axis is parallel to the Z direction, so the x-y projection represents the void image. The diameters of the inscribed (tangent) and circumscribed circles are then calculated.

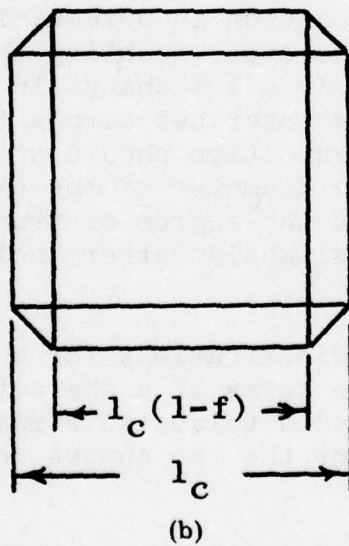
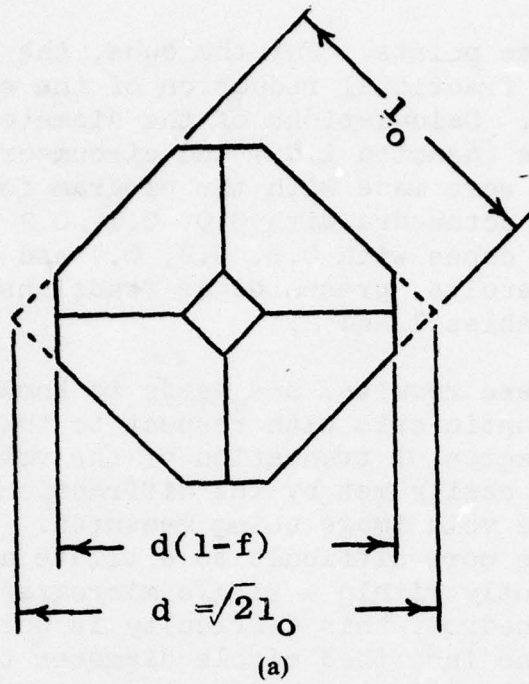


Fig. 2 — Definitions of the degree of truncation (f) for: (a) an octahedron of edge length l_0 ; (b) a cube of edge length l_c

the two opposite points. For the cube, the truncation was defined as the fractional reduction of the edge length of each cube face. Calculations of the diameters of inscribed circles (denoted i.d.) and circumscribed circles (denoted c.d.) were made with the program for 15 selected projections of octahedra with 0.0, 0.1, 0.2 and 0.3 truncation and for cubes with 0.0, 0.2, 0.4 and 0.6 truncation. The results, presented as fractions of t_o and t_c , are given in Tables 1 and 2.

To use these results, one needs to know the orientation of the optic axis with respect to the crystal structure and the degree of truncation of the voids. The first requirement is easily met by the diffraction pattern corresponding to the void image being measured. The truncation, however, may be more difficult to estimate and may indeed vary significantly within a single micrograph. For the truncated octahedron, this difficulty is not serious, since the ratio of the inscribed circle diameter to the edge length is hardly affected by the truncation. For the cube, the inscribed circle diameter is a somewhat stronger function of the truncation in orientations near $\{111\}$. Even in this case, however, doubling the truncation from 0.2 to 0.4 causes only a 10% change in the diameter/edge length ratio for the inscribed circle implying that errors in estimating the truncation should not be too serious. For both shapes, the diameter of the circumscribed circle is more sensitive to the degree of truncation, making the inscribed circle a slightly better choice of void measurement.

Once the size distributions for a set of polyhedral voids is obtained in terms of a characteristic edge length, the calculation of void volume is straightforward. The relevant formulas for the two shapes considered here are given below.

1. Truncated Octahedron - as illustrated in Fig. 3, the truncation of an octahedron of edge length t_o by the fractional amount f is equivalent to removing three

TABLE 1
 Ratios of Diameters of Inscribed (I. D.) and Circumscribed (O. D.) Circles
 to Octahedron Edge Length (lo) for Selected Orientation and Degrees
 of Truncation - See Text for details

Truncation Axis	0.0		0.1		0.2		0.3	
	I. D.	O. D.	I. D.	O. D.	I. D.	O. D.	I. D.	O. D.
100	1.000	1.414	1.000	1.281	1.000	1.166	0.990	1.077
110	0.816	1.414	0.816	1.277	0.816	1.149	0.816	1.034
111	1.000	1.155	1.000	1.102	1.000	1.058	0.981	1.026
210	0.943	1.414	0.943	1.279	0.943	1.159	0.943	1.075
211	0.816	1.291	0.816	1.215	0.816	1.143	0.816	1.075
221	0.943	1.333	0.943	1.237	0.943	1.151	0.926	1.076
310	0.973	1.414	0.973	1.280	0.973	1.163	0.973	1.073
311	0.905	1.348	0.905	1.255	0.905	1.163	0.905	1.074
320	0.904	1.414	0.904	1.278	0.904	1.156	0.904	1.059
321	0.816	1.363	0.816	1.260	0.816	1.164	0.816	1.076
331	0.905	1.376	0.905	1.266	0.905	1.164	0.897	1.075
510	0.990	1.414	0.990	1.280	0.990	1.165	0.990	1.074
511	0.962	1.388	0.962	1.276	0.962	1.165	0.962	1.072
710	0.995	1.414	0.995	1.280	0.995	1.166	0.990	1.075
711	0.980	1.400	0.980	1.280	0.980	1.160	0.980	1.074

TABLE 2

Ratios of Diameters of Inscribed (I.D.) and Circumscribed (O.D.) Circles to
 Cube Edge Length (l_c) for Selected Orientations and Degrees of Truncation
 See text for details

Truncation Axis	0.0		0.2		0.4		0.6	
	I.D.	O.D.	I.D.	O.D.	I.D.	O.D.	I.D.	O.D.
100	1.000	1.414	1.000	1.281	1.000	1.166	1.000	1.077
110	1.000	1.732	1.000	1.510	1.000	1.311	0.990	1.149
111	1.414	1.633	1.273	1.470	1.131	1.306	0.990	1.143
210	1.000	1.673	1.000	1.486	1.000	1.308	1.000	1.145
211	1.342	1.732	1.252	1.508	1.131	1.301	0.990	1.146
221	1.342	1.700	1.252	1.504	1.131	1.310	0.990	1.131
310	1.000	1.613	1.000	1.444	1.000	1.287	1.000	1.147
311	1.265	1.706	1.202	1.499	1.131	1.310	0.990	1.147
320	1.000	1.710	1.000	1.506	1.000	1.310	1.000	1.144
321	1.265	1.732	1.202	1.509	1.138	1.307	1.039	1.144
331	1.265	1.717	1.202	1.509	1.131	1.304	0.990	1.126
510	1.000	1.544	1.000	1.391	1.000	1.251	1.000	1.132
511	1.177	1.633	1.137	1.449	1.098	1.284	0.990	1.143
710	1.000	1.510	1.000	1.364	1.000	1.231	1.000	1.120
711	1.131	1.584	1.103	1.363	1.075	1.231	0.990	1.132

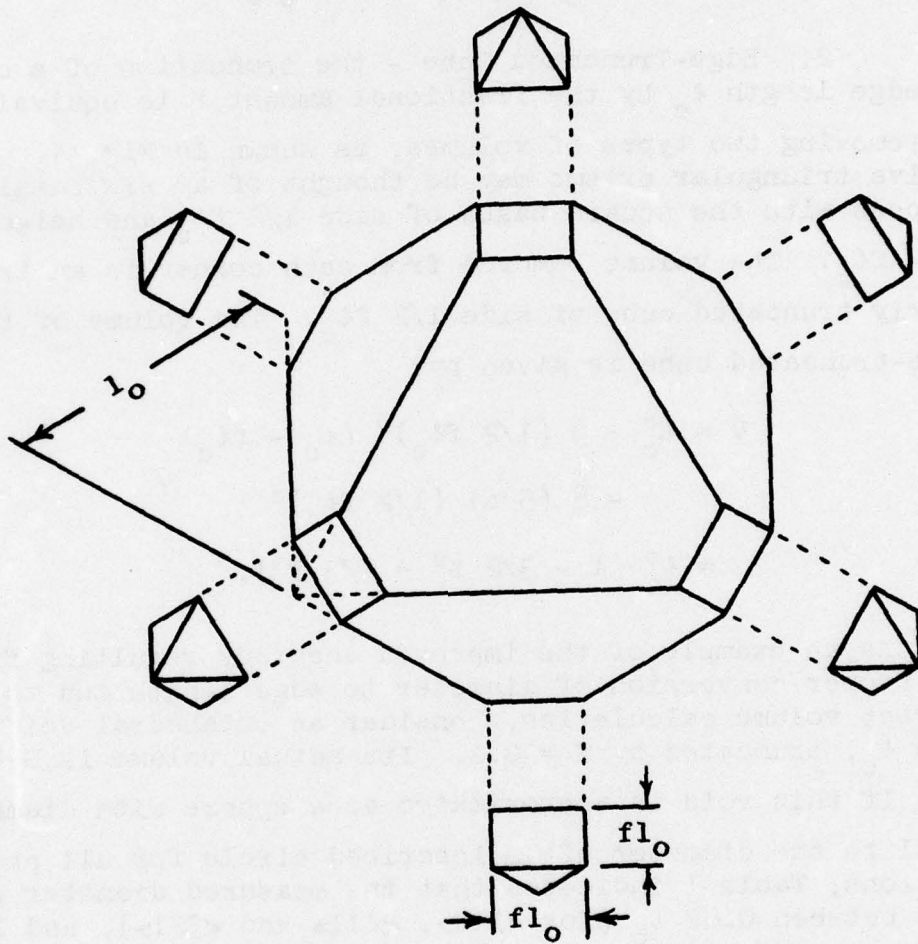


Fig. 3 — Illustration of the volume removed by truncating an octahedron of edge length l_0 by the fraction f . The six square pyramids removed are equivalent to three octahedron of edge length fl_0 .

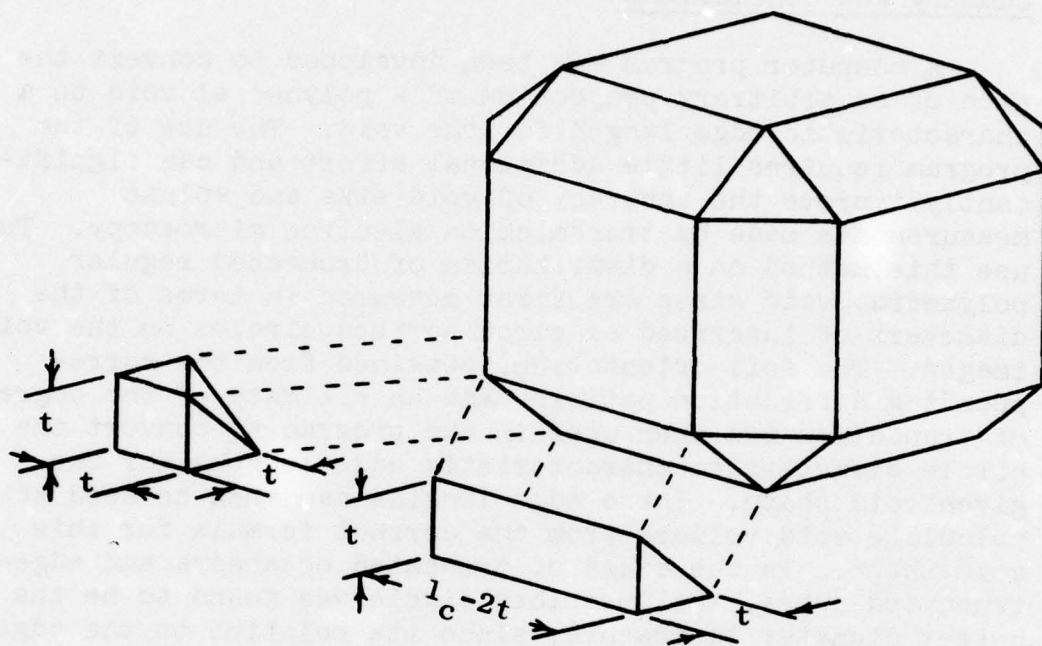
octahedra of edge length fl_0 . The volume of the remaining solid is thus

$$V = \frac{\sqrt{2}}{3} l_0^3 (1 - 3f^3)$$

2. Edge-Truncated Cube - the truncation of a cube of edge length l_c by the fractional amount f is equivalent to removing two types of volumes, as shown in Fig. 4. The twelve triangular prisms may be thought of as six parallel-pipedes with the square bases of side $1/2 fl_c$ and height of $l_c - fl_c$. The volume removed from each corner is an irregularly truncated cube of side $1/2 fl_c$. The volume of the edge-truncated cube is given by:

$$\begin{aligned} V &= l_c^3 - 6 (1/2 fl_c)^2 (l_c - fl_c) \\ &\quad - 8 (5/6) (1/2 fl_c)^3 \\ &= l_c^3 (1 - 3/2 f^2 + 2/3 f^3). \end{aligned}$$

As an example of the improved accuracy resulting from the proper conversion of diameter to edge length and the correct volume calculation, consider an octahedral void of edge l_0 , truncated by $f = 0.2$. Its actual volume is $0.46 l_0^3$. If this void is approximated as a sphere with diameter equal to the diameter of an inscribed circle for all projections, Table 1 indicates that the measured diameter will vary between $0.82 l_0$ (for $\langle 110 \rangle$, $\langle 211 \rangle$ and $\langle 321 \rangle$), and 1 (for $\langle 100 \rangle$ and $\langle 111 \rangle$). The calculated volume will vary from $0.28 l_0^3$ to $0.52 l_0^3$, depending on the orientation in which the void is imaged. For a cube, referring to Table 2, the variation of size with orientation is even stronger, and with no truncation, moving from $\langle 100 \rangle$ to $\langle 111 \rangle$ would change the measured diameter by 40%, representing a factor of 2.7 change in the calculated volume. Obviously, the



$$t = \frac{f l_c}{2}$$

Fig. 4 — Illustration of the volume removed by the edge truncation of a cube of length l_c . The truncation by the amount $t = 1/2 f l_c$ is equivalent to removing twelve right triangular prisms and eight irregularly truncated cubes, as shown.

proper conversion of the size of a void projection to the size of its three-dimensional shape can result in significantly improved accuracy of size and volume determinations.

Summary and Conclusions

A computer program has been developed to convert the size of an arbitrary projection of a polyhedral void to a characteristic edge length for the void. The use of the program requires little additional effort and can significantly improve the accuracy of void size and volume measurements made by transmission electron microscopy. To use this method on a distribution of truncated regular polyhedra, void sizes are first measured in terms of the diameters of inscribed or circumscribed circles on the void images. The foil orientation, obtained from the corresponding diffraction pattern, and an estimate of the degree of truncation are then used in the program to convert the circle diameters to characteristic edge lengths for the given void shape. These edge lengths can then be used to calculate void volumes from the correct formula for this void shape. In the cases of truncated octahedra and edge-truncated cubes, the inscribed circle was found to be the better diameter to measure, since its relation to the edge length was less sensitive to the degree of truncation.

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II. LIGHT ION DAMAGE STUDIES

A. Microstructure and Mechanism of Ion-Simulated Irradiation Induced Creep of Nickel

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Background

The dimensional stability of structural materials is of major importance to the design of breeder and, in the future, controlled thermonuclear reactors. One of the critical parameters to be considered in the evaluation of dimensional stability is the irradiation-induced creep. However, the lack of prototypical irradiation test facilities for in-reactor creep experiments coupled with the necessity for careful control of all experimental variables has motivated the development of techniques for ion-simulation of irradiation-induced creep.

Previous NRL experiments have shown that ion-simulation can be successfully applied to produce irradiation-induced creep in materials whose thickness is characteristic of proposed breeder reactor fuel cladding material.¹⁻⁷ During the current reporting period, the microstructures of the creep specimens were characterized and the mechanism responsible for the observed creep behavior was evaluated.

Progress

Transmission electron microscopy (TEM) discs were prepared from the gage section of the creep specimens using

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electrical-discharge cutting techniques. Similar discs were obtained from unirradiated end tab areas of the creep specimens as well as from as-received specimen material. Since preliminary studies indicated that foils prepared directly from the 0.38-mm specimen thickness did not contain sufficient thin area for quantitative microscopy, careful low-speed, water-cooled grinding procedures were used to reduce the thickness of the TEM discs to approximately 0.19 mm. During preparation of the discs from the specimen gage section, the grinding was done uniformly from both the front and rear surfaces of the specimen.

The TEM discs were thinned using a twin-jet electropolishing technique⁸ and a 9:1 (by volume) acetic-perchloric electrolyte. The thin foils were examined in a JEM 200A electron microscope operated at 200 kV and equipped with a double tilt goniometer stage. During TEM examination, the irradiated discs were oriented such that the direction of applied stress was coincident with the goniometer x-tilt axis.

The dislocation density and cell diameters were determined by previously described line intercept methods.⁹ The dislocation density measured was the average density of the network dislocations as opposed to the localized density of the dislocation cell walls. Weak-beam, dark-field procedures were employed to observe the defect clusters (presumed to be unresolvable dislocation loops) and dislocation loops in the irradiated specimens whenever possible. However, the highly deformed nature of the specimens coupled with the small defect cluster/dislocation loop size prevented the use of the weak-beam, dark-field method in all cases. The defect cluster/loop sizes and distributions were evaluated from enlarged micrographs using a particle size analyzer. In addition, individual size measurements were made from the central region of highly enlarged micrographs to confirm the results from the particle size analyzer. Foil thickness was determined using dynamical stereo micrographs. At least three thickness determinations were used to compute the dislocation

and defect cluster/loop densities for each specimen. These values are believed to be accurate to only within $\pm 50\%$ due to the high density of defects and initial deformation structure.

Examination of TEM specimens prepared from unirradiated, as-received nickel stock material and from unirradiated portions of the creep specimens indicated a deformation microstructure characteristic of cold-worked material. Small dislocation cells, deformation bands and slip traces were observed in most cases. A typical example of the dislocation cell structure is shown in Fig. 1(a). The dislocation density of the cell walls was estimated to be $> 1 \times 10^{13}$ cm/cm³. Small dislocation loops were occasionally observed within the cell interiors in these specimens, Fig. 1(b).

The TEM evaluation of the irradiated microstructures revealed no substantial difference between the deuteron and alpha particle irradiated specimens. A heterogeneous distribution of small defect clusters, dislocation loops, individual dislocations, and larger dislocation cells with reduced cell wall dislocation densities ($\sim 10^{11}$ cm/sm³) was distinctly visible in all specimens. Typical examples of the defect cluster/dislocation loop structure and network dislocations are shown in Figs. 2(a) and 2(b). In both figures, the pinning of individual dislocation by the defect clusters/dislocation loops can be seen. In certain specimens, resolvable dislocation loops were observed as shown in Fig. 2(c) and Fig. 2(d). Repeated attempts to deduce the character of these loops by weak-beam, dark-field stereo methods¹⁰ and bright-field tilting procedures¹¹ were inconclusive due to the small loop size and high loop density. In certain specimens, limited evidence of loop orientation on {111} habit planes was observed. However, efforts to determine the extent, if any, of preferential loop alignment with respect to the applied stress direction were inconclusive.

The microstructural results from all specimens examined in this study are given in Table 1. It was noted

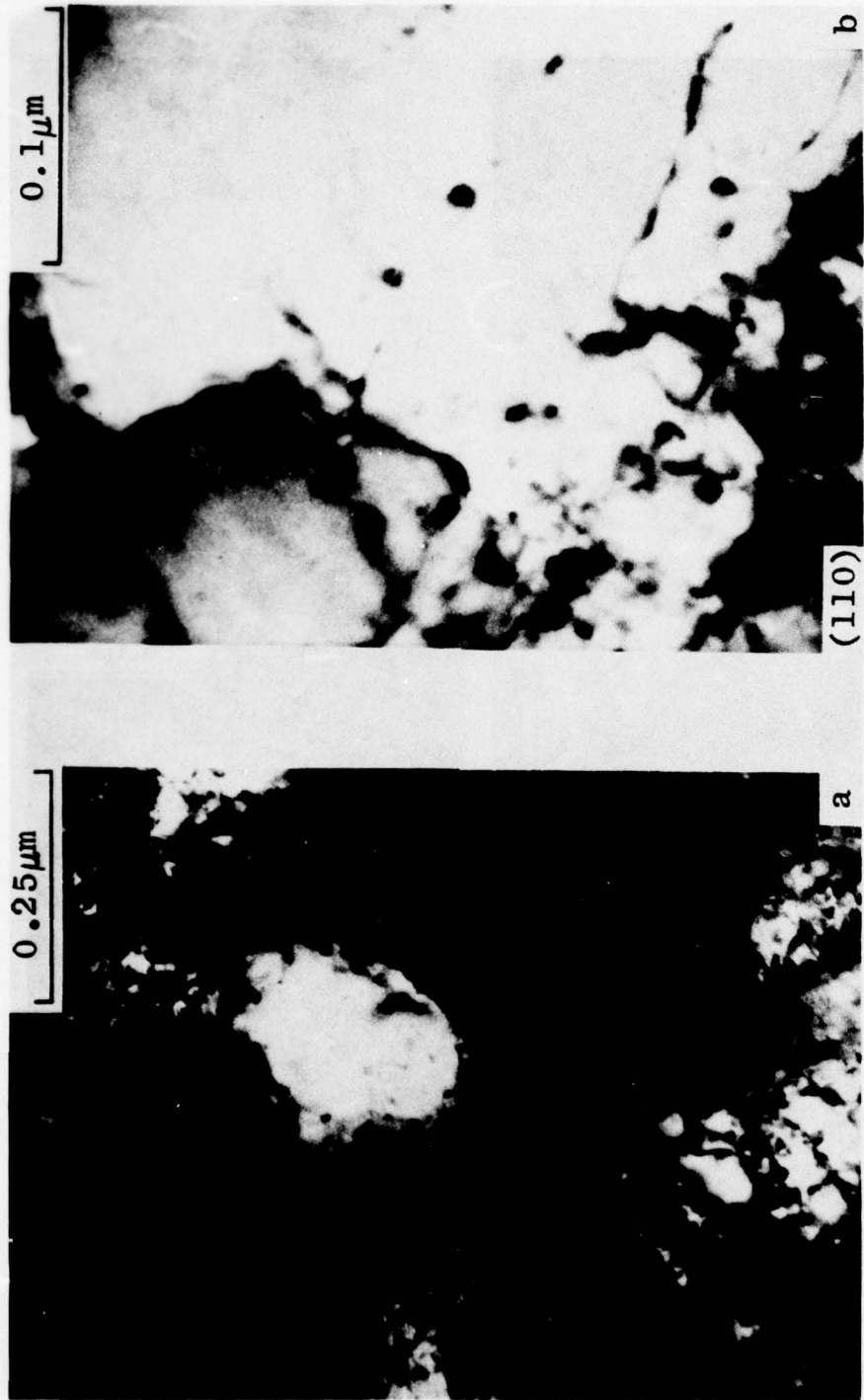


Fig. 1 — Microstructure of unirradiated nickel specimen material. (a) Dislocation cells characteristic of the cold work level of the material, and (b) small dislocation loops occasionally observed within the cell interiors.

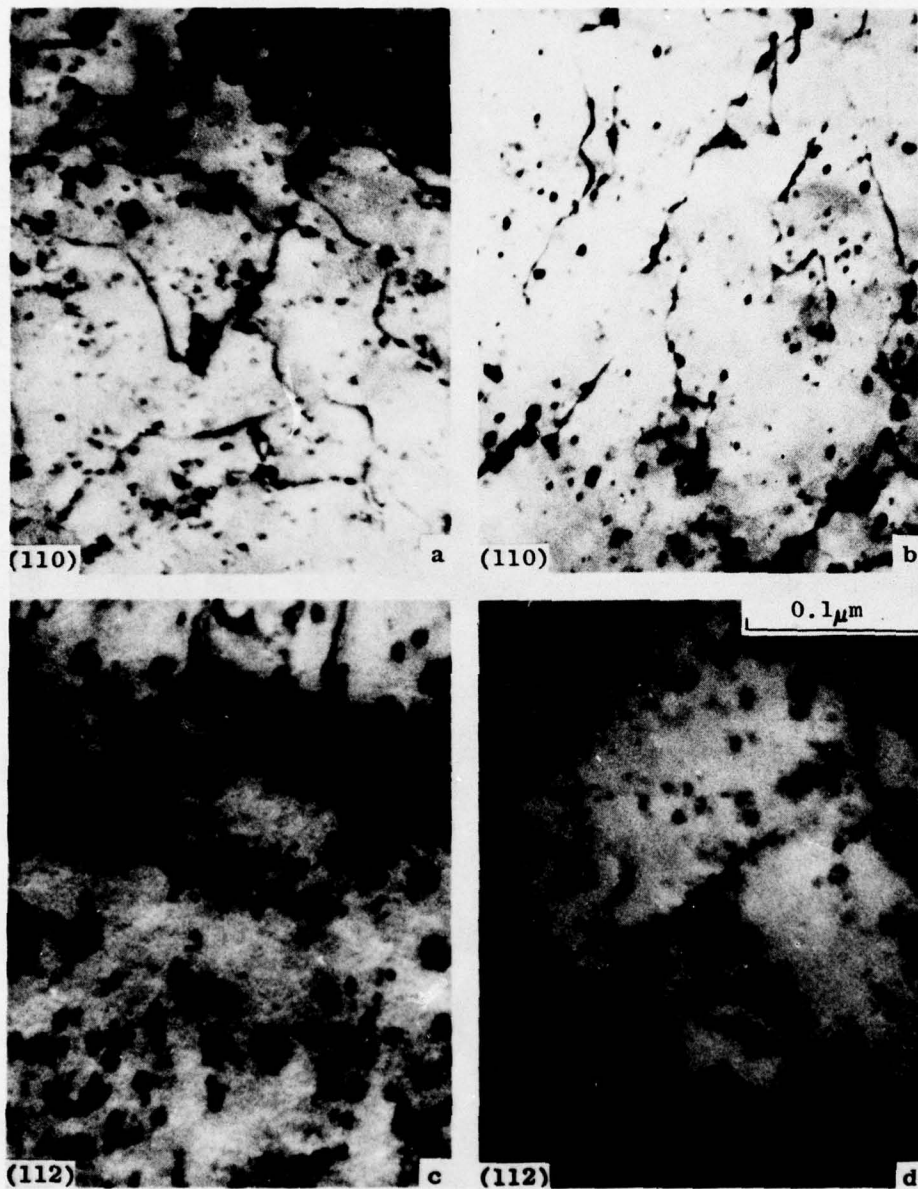


Fig. 2— Microstructure of ion-simulated irradiation-induced creep specimens. The direction of applied stress in all micrographs was left to right within the plane of the paper. (a) and (b) — Defect cluster/dislocation loop structure and network dislocations. Note the pinning of individual dislocations by the defect clusters/dislocation loops. (c) and (d) — Distinct dislocation loops observed in specimen 4D-2-5.

TABLE 1--Summary of ion-simulated irradiation-induced creep test and microstructure results

Material No.- Specimen No.- Test No. ^a	Irradiation Test Temperature ^c deg C	Stress ^a MPa	Displacement Rate ^a x 10 ⁶ dpa/sec	Fluence ^b x 10 ²⁰ dpa	Steady-State Creep Rate ^a x 10 ⁶ hr ⁻¹	Normalized Steady-State Creep Rate ^a x 10 ³ dpa ⁻¹	Defect Cluster/ Loop Number Density #/cm ³	Mean Defect Cluster/Loop Diameter Å	Dislocation Density cm/cm ²	Mean Dislocation Cell Dia µm
4D - 2-5	224	345	30.40	3.6	4.3	4.9	1.6 x 10 ¹⁶	46	3.6 x 10 ¹⁰	0.82
4D - 3-2	224	170	30.40	1.6	1.2	1.4	2.5 x 10 ¹⁶	58	3.4 x 10 ¹⁰	1.20
4D - 4-2	224	345	27.17	2.3	4.8	4.9	1.4 x 10 ¹⁶	43	3.2 x 10 ¹⁰	0.85
4D - 5-2	224	345	13.53	0.9	2.7	5.5	2.0 x 10 ¹⁶	42	2.8 x 10 ¹⁰	1.00
4E - 1-2	224	170	27.17	1.9	1.8	1.8	2.2 x 10 ¹⁶	52	3.6 x 10 ¹⁰	1.20
4E - 2-2	224	207	27.17	2.1	1.4	1.4	3.0 x 10 ¹⁶	67	3.5 x 10 ¹⁰	1.15
4E - 4-2	224	247	27.17	1.7	2.6	2.7	1.8 x 10 ¹⁶	50	2.9 x 10 ¹⁰	1.10
4D							<10 ¹³	~30	> 2 x 10 ¹¹	~0.28

Unirradiated, unstressed material

^a Data from Reference 12.

^b Includes particle factor (Reference 12).

that the defect cluster/dislocation loop diameters exhibited a very narrow distribution about the mean values given in the table. The normalized steady-state creep rates were computed from the experimental results in the manner described by Hendrick *et al.*⁷ The microstructural results indicate that the combined effects of both ion irradiation and applied stress produced an increase in dislocation cell size with an accompanying net decrease in dislocation density. The defect cluster/loop size and density were found to vary inversely with the applied stress as shown in Fig. 3.

Discussion

The previously published ion-simulated irradiation-induced creep experimental results for the specimens examined in this work have shown that the stress dependence of the steady-state creep rate* was approximately quadratic ($\dot{\epsilon} \propto \sigma^2$) and the flux dependence was approximately linear.^{7, 12} By comparison, it was shown that these results were in overall agreement, within experimental error, with previous ion-simulated irradiation-induced creep results for nickel obtained by Hendrick *et al.*^{13, 14} using 3.0- and 5.25-MeV protons. Other simulation results reported by Harkness *et al.*¹⁵ for an austenitic stainless steel show a stress dependence of the creep rate between one and two at stresses and homologous temperatures comparable to those used for the nickel experiments. Although no microstructural data were obtained in the previous studies for comparison with the present results, the experimental data in all cases suggest that irradiation-induced, climb-controlled glide could be responsible for the observed creep behavior. In contrast

* The steady-state creep rate, as used in this study, may be temporal in nature (i.e., specimens irradiated to higher fluence levels may exhibit lower values of steady-state creep rate due to hardening effects).

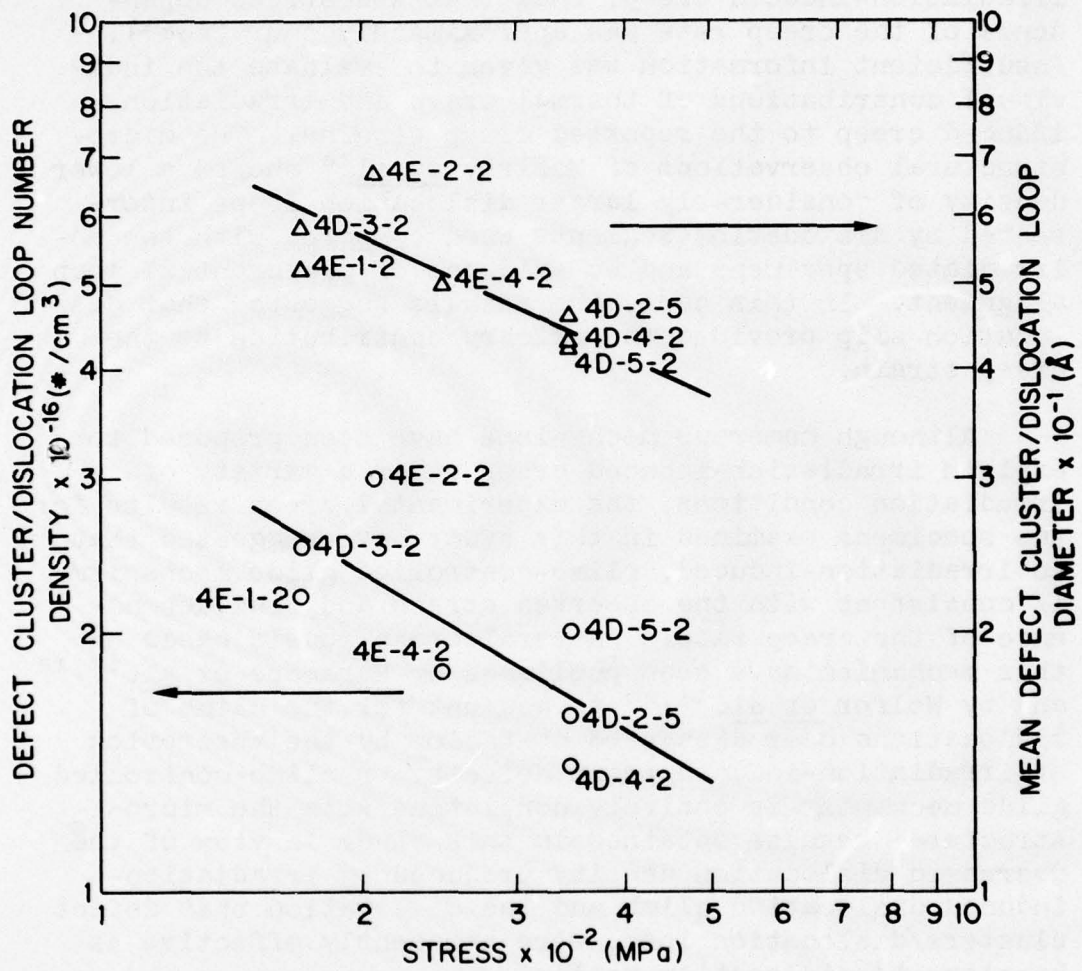


Fig. 3 - Stress dependence of defect cluster/dislocation loop number density and mean diameter

to these observations, recent simulation results for nickel by McElroy *et al.*¹⁶, obtained at temperatures where thermal creep occurred simultaneously with the irradiation-induced creep, show that the stress dependence of the creep rate was approximately four ($\dot{\epsilon} \propto \sigma^4$). Insufficient information was given to evaluate the individual contributions of thermal creep and irradiation-induced creep to the reported creep strains. The microstructural observations of McElroy *et al.*¹⁶ showed a lower density of considerably larger dislocation loops intersected by dislocation segments when compared with the unirradiated specimens and no evidence of preferential loop alignment. In this case, the results suggested that dislocation slip provided the primary contribution to the creep strain.

Although numerous mechanisms have been proposed to explain irradiation-induced creep under a variety of irradiation conditions, the experimental creep results for the specimens examined in this study have suggested that an irradiation-induced, climb-controlled glide mechanism is consistent with the observed stress and flux dependence of the creep rate.⁷ Several creep models based on this mechanism have been published by Harkness *et al.*^{17, 18} and by Wolfer *et al.*^{19, 20} to account for the climb of dislocations over dispersed obstacles by the absorption of irradiation-induced point defects. A climb-controlled glide mechanism is entirely consistent with the microstructural results obtained in this study in view of the decreased dislocation density produced by irradiation-induced dislocation climb and the observation that defect clusters/dislocation loops were apparently effective as barriers to dislocation motion.

The agreement between the climb-controlled glide mechanism and both the previous experimental results and the present microstructural observations for the same nickel specimens was analyzed by computing the steady-state creep rate on the basis of the microstructural data. For the case where a metallic material is undergoing

irradiation, the steady-state defect concentrations may be calculated according to the models proposed by Brailsford and Bullough^{21,22} using the defect balance equations:

$$K - D_i C_i k_i^2 - \alpha C_i C_v = 0 \quad (1)$$

$$K' - D_v C_v k_v^2 - \alpha C_i C_v = 0 \quad (2)$$

where K is the atomic displacement rate, K' is the effective defect generation rate (i.e., the displacement rate augmented by vacancy emission from sinks), C_i and C_v are the concentrations of interstitials and vacancies, D_i and D_v are the diffusion coefficients for interstitials and vacancies, k_i^2 and k_v^2 are the total strengths of fixed sinks for interstitials and vacancies, and α is the recombination coefficient. The sink strengths were then expressed as:

$$k_i^2 = Z_i \rho_d \quad (3)$$

$$k_v^2 = Z_v \rho_d \quad (4)$$

where ρ_d is the dislocation density (network dislocations and loops), and Z_i and Z_v are the sink strengths for interstitials and vacancies. The dislocation cell walls were assumed to act as neutral sinks and their strength was estimated as $1/r_c^2$, where r_c is the cell radius. From the results in Table 1, their maximum strength is approximately $6 \times 10^8 \text{ cm}^{-2}$ which is considerably less than that of the network dislocations and loops. Therefore, the effect of these sinks on the calculated creep rates was negligible. The interstitial and vacancy diffusion coefficients were calculated by:

$$D_i = D_i^0 \exp(-E_i^m/kT) \quad (5)$$

$$D_v = D_v^0 \exp(-E_v^m/kT), \quad (6)$$

where D_i^0 and D_v^0 are the diffusion pre-exponentials, E_i^m and E_v^m are the activation energies for interstitials and vacancies, T is the absolute temperature and k is Boltzmann's constant.

The creep rate, $\dot{\epsilon}$, was evaluated according to the expression for climb-controlled glide:^{19,20}

$$\dot{\epsilon} = \rho_d^n b |v_c| (L/d), \quad (7)$$

where ρ_d^n is the network dislocation density, b is the Burger's vector, L is the obstacle spacing, and d is the obstacle height. The dislocation climb velocity, v_c , was evaluated from the expression for a stressed solid:²³

$$v_c = \frac{\sigma \Omega}{6(1-\nu) b k T} \left(C_v D_v Z_v^S - C_i D_i Z_i^S \right), \quad (8)$$

where σ is the applied stress, Ω is the atomic volume, ν is Poisson's ratio, and Z_i^S and Z_v^S are the dislocation bias for interstitial and vacancy capture under stress, respectively. The thermal vacancy concentration term was not included in Eq. (8) since preliminary calculations confirmed that it was negligible at the irradiation temperatures where the experimental data were obtained. For purposes of calculation, the material parameters for nickel given by Sprague *et al.*²⁴ were used. The bias terms used were $Z_i = 1.01$ and $Z_v = 1.00$. The bias terms under stress were taken as $Z_i^S = 1.2$ and $Z_v^S = 2.7$ following Heald and Speight.²³ The obstacle

spacing was determined from the loop density, N_l , as $L = \frac{1}{2}(N_l^{-1/3})$, and the obstacle height was taken as the defect cluster/loop diameter given in Table 1.

The creep rates computed from Eq. (7) are compared in Fig. 4 with the experimental creep rates for the same specimens⁷ as a function of stress. It can be seen that the creep rates calculated from the values in Table 1 are in reasonable agreement with the experimental results. The extremes of the vertical lines in Fig. 4 represent the creep rates calculated from the maximum and minimum observed defect cluster/dislocation loop diameters. The solid line through the experimental data points indicates a slope of two in accord with the results obtained by Hendrick *et al.*⁷ In addition, the creep rates expected on the basis of the dislocation climb model of Wolfer and Ashkin²⁵ and the loop orientation model of Brailsford and Bullough²¹ were computed using the same material and microstructural parameters. The creep rates from both of these models are several orders of magnitude below those calculated for the climb-controlled glide model and those observed experimentally. The calculations confirm that, of those models considered, only the climb-controlled glide model is in reasonable agreement with the steady-state ion-simulated irradiation-induced creep behavior observed in the present specimens. Evidence which supports this suggestion has been recently published by Wolfer²⁶ which shows that, for low fluence ($\leq 10^{22}$ n/cm²) and temperatures, the primary contribution to creep is provided by dislocation loops in agreement with the experimental data of Mosedale *et al.*²⁷

Previous calculations of creep rates using a climb-controlled glide model by Harkness *et al.*^{17, 18} did not distinguish between the transient and steady-state neutron irradiation-induced creep, but predicted a decrease in creep rate with dose rate reflecting the accumulation of defects with increasing fluence. Since the microstructural results obtained in the present study represent the defect parameters after steady-state creep was

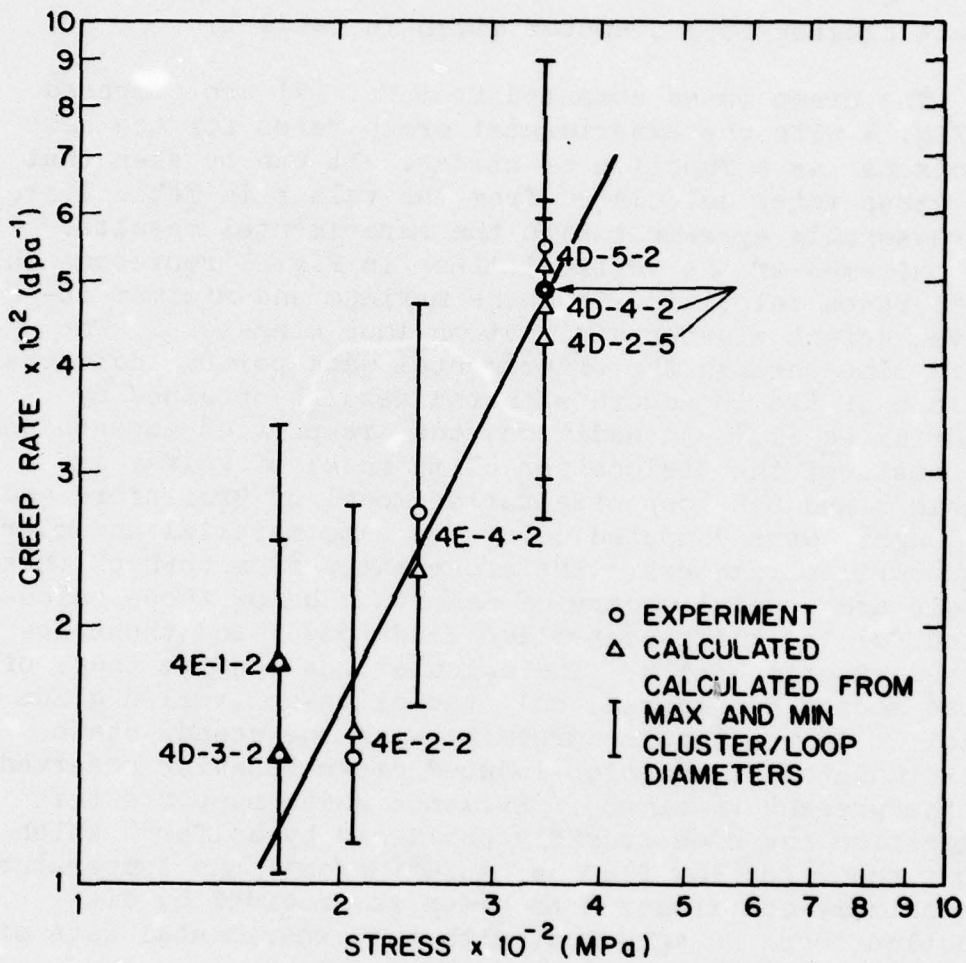


Fig. 4 - Stress dependence of calculated and measured ion-simulated irradiation-induced creep rate

achieved, no effect of fluence on the creep rate was calculated. However, the experimental data given by Hendrick *et al.*⁷ for these specimens show that transient creep is essentially saturated at fluences greater than approximately 0.01 dpa. This evidence indicates that the initial accumulation of irradiation defects occurs very rapidly, and may continue to fluence levels beyond those achieved experimentally to produce further growth of the defect clusters/dislocation loops. It is, therefore, reasonable to expect that climb-controlled glide will continue to operate at these higher fluences, but that the creep rate will diminish with the increase in defect cluster/dislocation loop size. When this occurs, other mechanisms may become dominant and provide the primary contribution to the creep rate.

Conclusions

1. The effect of both ion irradiation and applied stress was to increase cluster/dislocation loop number density and size and dislocation cell diameter and to increase network dislocation density. No evidence of preferential dislocation loop alignment with respect to the applied stress direction was observed.
2. The defect clusters/dislocation loops appeared to act as effective obstacles to network dislocation motion during steady-state irradiation-induced creep. This observation together with the decreased network dislocation density is consistent with a climb-controlled glide mechanism.
3. Calculations based on the microstructural results confirmed that a climb-controlled glide mechanism was in agreement with the squared stress dependence and linear flux dependence observed during ion-simulated irradiation-induced creep experiments.
4. Creep rates calculated from the microstructural results using dislocation climb and loop

orientation models were several orders of magnitude less than those calculated for the climb-controlled glide mechanism.

Summary

The microstructure of cold worked, high purity nickel has been investigated following ion-simulated irradiation-induced creep with 22-MeV deuterons and 70-MeV alpha particles. The irradiations were conducted at 224°C, at stresses between 170 and 345 MPa, and at displacement rates between 13 and 30×10^{-8} displacements per atom per second. Transmission electron microscopy (TEM) procedures were used to prepare, observe, and photograph the microstructure of the ion irradiated uniaxial creep specimens and companion unirradiated specimens.

The microstructural results were evaluated in terms of the theoretical mechanisms proposed for irradiation-induced creep and the previously reported creep simulation results for nickel by Hendrick *et al.* A model based on the climb-controlled glide of dislocations over dispersed obstacles was found to be consistent with the microstructural results and the experimental creep data.

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