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Void Nucleation and Growth in Heavy Ion
and Electron Bombarded Pure Metals

Progress Report
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Summary

Irradiation of high purity aluminum with 1.2 to 1.4 MeV aluminum ions at temperatures from 30-120°C revealed that helium has very strong influence on void formation. No voids could be formed in Al without helium even up to 2.7 dpa. Samples preinjected with 0.1, 1, and 10 appm helium and then irradiated at 100 and 120°C produced voids at damage levels of ~0.5 dpa. The void size and density varied in the customary fashion with increasing temperature and the voids were determined to be <111> octahedra.

A high temperature (1100°C) and high vacuum ($<10^{-8}$ torr at temperature) sample irradiation chamber has been constructed and operated. A residual gas analyzer has been used to determine the partial pressures of the gases in the chamber during high temperature irradiation of vanadium. It was found that partial pressures of hydrocarbons, and oxygen atoms were $<10^{-10}$ torr during the irradiation runs. This has greatly reduced the impurity pick up of samples and drastically reduced the precipitation reactions in the high purity vanadium.

Voids have been produced in high purity vanadium during bombardment at 700°C with 14 MeV copper ions. The average void size is $\sim 1000\text{\AA}$ which is much larger than previously reported for vanadium. It is thought that the smaller average void size in other experiments is due to the higher level of contamination in the sample region.

The University of Wisconsin EM-7 1 MeV TEM has been placed into full operation for irradiation studies. Well annealed Al without preinjected helium, has been irradiated in this microscope at 37, 50, 75, 100, 125, and 150°C to approximately 0.9 dpa. A highly developed loop structure was observed, consistent with previous work on the HEDL microscope, but again no voids ($> 10^{11} \text{ cm}^{-3}$) were observed. The previous work had been done on cold worked Al samples.

Analysis of the prismatic dislocation loops revealed that they were 60% interstitial and 40% vacancy.

Two papers were written during this period. "Role of Helium and Temperatures in Void Formation in Aluminum Irradiated with Aluminum Ions," M. L. Sundquist and J. M. Donhowe, to be submitted to J. Nucl. Materials, "A Sputter PIG Source (SPIGS) for Negative Ions," H. Vernon Smith, Jr., and H. T. Richards, Symposium on Ion Sources and Formation of Ion Beams, Berkeley, Calif., Oct. 22-25, 1974. One paper was published during this period, "A Review of Ion Simulation of High Temperature Neutron Damage and Void Formation," G. L. Kulcinski, p. 613 in Applications of Ion Beams to Metals, ed. by S. T. Picraux, E. P. EerNisse, and F. L. Vook, Plenum Pub. Corp., New York, 1974.

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Objective

The objective of this study is to clarify the important parameters for nucleation and growth of voids in pure aluminum and vanadium. The factors governing the production of voids during neutron irradiation of these metals are difficult to separate because both transmutation reactions and displacement spike cascades are superimposed on the pure displacement damage during irradiation. This study will use high energy beams of Al^+ or V^+ ions to produce the necessary damage while eliminating the transmutation effects. A high energy beam of electrons will be used to eliminate both the transmutations and displacement spike effects. Selected doping of the samples with helium, hydrogen, Si, Cu, Zn, or Ti will help us to understand the role of these impurities in nucleating voids. This study will concentrate on high purity V and samples of 99.999% Al already extensively analyzed by ORNL scientists after neutron irradiation.

Progress in Experimental Programs

A. Aluminum Ion Bombardment of Aluminum

1. Introduction

The growth of voids in pure, unstressed, well annealed metals has been reasonably well described by several authors. ⁽¹⁻⁴⁾ However, the understanding of the void nucleation mechanisms, either in pure materials or those which contain impurity atoms, has advanced only recently. ⁽⁵⁻⁷⁾ The key to swelling resistant metals and alloys now appears to lie with the retardation of nucleation because once voids are formed, they seem to grow regardless of the metallurgical state or the method of defect production (i.e. electrons, protons, heavy ions, or neutrons).

A vital question which still needs to be answered is what is the role of the insoluble gas, helium, in this nucleation stage. This is of practical importance because all metals irradiated in fission, and especially fusion, reactors will undergo a large number of (n,α) reactions during a component's lifetime. It has been shown that helium can significantly increase the void nucleation rate in nickel and steel ⁽⁸⁾ but it has also been shown that helium is not absolutely necessary for void nucleation in heavy ion bombarded steel, ⁽⁹⁻¹²⁾ nickel ^(8,13), Mo ⁽¹³⁻¹⁵⁾, Nb ^(13,16-17), V ⁽¹⁸⁻²¹⁾ and Ta ⁽²²⁾. On the other hand, helium does seem to be necessary for void nucleation in proton bombarded Ni and stainless steel. ^(23,24)

The object of this work is to determine experimentally the role of He in the nucleation of voids in high purity aluminum bombarded with Al ions. While there has been a substantial number of studies on voids in neutron irradiated Al ⁽²⁵⁻²⁷⁾, there is only one other known study of high temperature heavy ion bombardment of Al. ⁽²⁸⁾ Various workers ⁽²⁹⁻³²⁾ have also examined the use of electrons

on Al in attempts to produce voids, but to date none have been successful.

2. Experimental

2.1 Material Characterization and Preparation for Irradiation

The aluminum foils used in this experiment were furnished by Dr. J. O. Stiegler of Oak Ridge National Laboratory. They were Cominco Grade 69, high-purity, zone-refined aluminum. Analysis done at ORNL revealed 7 appm solid impurities.⁽²⁹⁾ Similar aluminum has been used in several neutron-irradiated studies at ORNL⁽²⁵⁻²⁷⁾, so comparison between neutron-irradiated and ion-irradiated material can eventually be made.

The uninjected samples were annealed at 500°C for about 2 hours in a vacuum of typically 5×10^{-6} torr, then irradiated. There was no intermediate polishing or cleaning process which could have introduced contamination. The samples to be implanted were similarly prepared, then irradiated with helium at energies from 400 keV to 2 MeV in steps calculated to produce uniform concentration between 2 and 7 microns depth.⁽²⁹⁾ After helium implantation, the samples were chemically thinned in a solution of 64% H_3PO_4 , 18% H_2SO_4 , and 18% HNO_3 at 70°C to remove the surface layer not containing helium. Samples were prepared both without helium and with 0.1, 1, and 10 appm helium.

2.2 Irradiation

Irradiation was carried out in a high-vacuum target chamber on the 2 MV Wisconsin Model 51 Electrostatic Accelerator. Foils were held between a stainless steel mask with 3mm diameter openings for the beam and a copper backing plate. A thermoelectric module between the backing plate and a heat sink was used to heat or cool the samples between 30°C and 120°C measured by a thermocouple on the sample.⁽²⁹⁾ Overall temperature control was within $\pm 5^\circ\text{C}$ and the vacuum during irradiation was typically 10^{-7} torr.

Magnetically analyzed aluminum ions from a physicon Model 910 Ion Source were normally incident on the target foils. Ion currents ranged from $0.12 \mu\text{A}/\text{cm}^2$ ($1.5 \times 10^{12} \text{ Al}^+/\text{cm}^2\text{-sec}$) corresponding to displacement density rate of $1.5 \times 10^{-4} \text{ dpa/sec}$ to $0.56 \mu\text{A}/\text{cm}^2$ ($7 \times 10^{-4} \text{ dpa/sec}$) at about 75% of the distance from the surface to the peak of the nuclear energy loss curve.

The "effective" irradiation temperatures are compared to neutron irradiation by the expression,

$$T_n = \frac{T_i}{1 + \frac{kT_i}{Q_m^v} \ln\left(\frac{K_i}{K_n}\right)}$$

where T_n = equivalent neutron temperature

T_i = actual ion bombardment temperature

Q_m^v = migration energy of vacancies in Al

K_i = dpa rate during ion bombardment

K_n = dpa rate during neutron irradiation

Using 0.63 for Q_m^v in Al⁽³³⁾ and $10^{-6} \text{ dpa sec}^{-1}$ for K_n and an average of $4 \times 10^{-4} \text{ dpa sec}^{-1}$ for ion bombardment, the following temperature relations were calculated

T_i °C	T_n °C
30	-37
50	-18
75	- 2
100	13
120	24

2.3 Preparation for TEM

The displacement density in ion bombarded Al varies with depth of penetration as shown in Figure 1.⁽³⁴⁾ It is therefore necessary to thin irradiated samples in such a manner that the distance from the bombarded surface is precisely known. In the method used here, the foil strips, each containing two or more irradiated spots 3mm in diameter were chemically thinned to remove some specified fraction of the damaged layer. Uniform removal was assumed, and weight loss was used to measure depth exposed.

After the proper damage zone was exposed, the irradiated spots were coated with laquer, cut from the foil strips, dimpled opposite the center of each irradiated spot, and finally electropolished in a solution of 40% acetic acid, 30% phosphoric acid, 20% nitric acid, and 10% distilled water. Samples were examined in an AEI Model EM802 Electron Microscope at 100 keV and a JEOL Model 100C electron microscope at 100 keV.

3. Results

A summary of the initial helium content, irradiation temperature, dpa value at the point of observation, void density, void size and calculated swelling is given in Table I. The first point to note is that irradiation of 9 samples of pure, undoped Al at temperatures of 50-120°C and to damage levels of 0.26 to 2.8 dpa produced no voids, only dislocation loops. The addition of 0.1 appm helium atoms produced voids at 50, 75, 100 and 120°C for dpa values ranging from 0.45 to 0.68 dpa. Similarly, the addition of 1 appm He produced voids at 100°C and 0.48 dpa. Micrographs of the microstructures at 100°C for varying helium contents are shown in Figure 2. The effect of temperature on the void size and density is shown in Figure 3 for 0.1 appm

DISPLACEMENT DAMAGE IN Al BOMBARDED BY Al IONS

Figure 1

Table I

Microstructural Data of Aluminum Irradiated with
an Average of 5.3×10^{15} Al^+/cm^2 of Energy 1.2 to 1.4 MeV

Helium Content	Temperature °C	dpa value at point of Observation	Void Density cm^{-3}	Void Diam. Å	$\frac{\Delta V}{V_0}$ %
0	50	0.46	None	None	None
	50	0.46	Observed	Observed	Observed
	75	0.26	"	"	"
	75	0.26	"	"	"
	100	0.28	"	"	"
	100	0.36	"	"	"
	100	2.80	"	"	"
	120	0.27	"	"	"
	120	0.27	"	"	"
0.1 appm	50	0.6	4×10^{15}	140	0.58
	50	0.65	1×10^{15}	150	0.19
	75	0.6	2×10^{15}	180	0.63
	100	0.45	3×10^{14}	300	0.59
	100	0.45	$(9 \times 10^{13})^*$	310	0.17
	120	0.68	$(1.2 \times 10^{14})^*$	460	0.60
	120	0.68	1.5×10^{14}	490	0.61
	28	0.65	None	None	None
	30	0.65	Observed	Observed	Observed
1	50	0.56	"	"	"
	50	0.56	"	"	"
	50	0.65	"	"	"
	75	0.58	"	"	"
	75	0.56	"	"	"
	100	0.50	$(1 \times 10^{14})^*$	250	0.11
	120	0.58	$(6 \times 10^{13})^*$	410	0.22
	75	0.58	None observed	None observed	None observed
	100	0.48	3×10^{14}	310	0.55

* estimated with a foil thickness of 1500 Å

EFFECT OF HELIUM CONTENT ON VOIDS IN ALUMINUM

100° C - 1.2 MeV Al⁺ 0.58 DPA

Figure 2

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EFFECT OF TEMPERATURE ON DAMAGE IN ALUMINUM

0.1 appm He - 1.2 MeV Al

0.58 DPA

Figure 3

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doped specimens. The void size gets larger and the density reduces with increasing temperature consistent with many previous studies on voids in metals. Figure 4 summarizes the data graphically.

Finally, it was noted that when the helium content was raised to 1 appm we were unable to form voids at 50 and 75°C (Figure 5 and Table I). A similar result was obtained for 75°C at 10 appm.

4. Discussion

There are 3 basic results of this experiment that must be discussed;

- a) The lack of void nucleation in the undoped Al samples.
- b) The lack of void nucleation in the 1 and 10 appm helium doped samples at temperatures below 75°C.
- c) The variation of void size and number density with temperature.

The extreme sensitivity of Al to helium doping is the first such case discovered for heavy ion bombardment studies. It is quite possible that this effect shows up in Al because it can be obtained in such high purity that essentially all of the heterogeneous nucleation defects are eliminated. This would also explain why no one has yet been able to observe voids in electron irradiated high purity Al while several investigators have easily produced voids in steel and nickel based alloys.

The effect of helium on void nucleation can be described in the following qualitative fashion. In the absence of helium and other impurity atoms, the nucleation of voids in a sea of excess vacancies and interstitials is determined by the rate at which the void embryos exceed some critical cluster size. These clusters can be nucleated homogeneously or in the disordered region of displacement spikes. Those clusters exceeding the critical nucleus size have a greater probability of adding a vacancy than having an interstitial

Figure 4 - Effect of Irradiation Temperature and Helium Content on the Void Size and Density in Al Bombarded with 1.2-1.4 MeV Al Ions to an Average dpa Value of ~0.6

EFFECT OF TEMPERATURE ON DAMAGE IN ALUMINUM

1 appm Helium - 1.2 MeV Al

0.58 DPA

Figure 5

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atom to add to the cluster or emitting a vacancy from the cluster. Therefore, they tend to grow. Clusters below the critical size have a greater probability of shrinking via vacancy emission or interstitial atom addition than adding a vacancy.

The addition of mobile helium atoms in the metal can aid the nucleation step in two different ways. First, the addition of gas atoms to a small void embryo will reduce the probability of vacancy emission. This results from the fact that the shrinking void must also do PV work in compressing the gas atoms in the void. Wiedersich et al.⁽⁵⁾ have shown that the rate constant for vacancy emission is

$$k_{\text{Vac. emiss.}} = J_v A (i-1)^{2/3} \exp(-\Delta F/kT)$$

where J_v is the vacancy flux per unit area

A is a geometrical constant

i is the number of vacancies in the cluster

$\Delta F = kT \ln S + \Delta(\text{surface energy}) + \Delta(\text{work on gas})$

S = supersaturation of vacancies

$\Delta(\text{surface energy})$ is the difference in surface energies of a cluster of $i-1$ and i vacancies

$\Delta(\text{work on gas})$ is the work required to compress the gas atoms

Expressions for the surface energy and PV work are⁽³⁵⁾

$$\Delta(\text{surface energy}) = A\sigma[(i-1)^{2/3} - i^{2/3}]$$

σ = macroscopic surface energy per unit area

$$\Delta(\text{work on gas}) = -jkT \ln \left\{ \left(\frac{i-1}{i} \right) \left(\frac{1 - [v_g/(i-1)v]^{1/3}}{1 - (v_g/iv)^{1/3}} \right)^3 \right\}$$

j is the number of gas atoms in the bubble

v_g is the volume of a single gas atom

v is the volume per atom

Since the logarithm of the gas term is <1 , positive work is required to compress the gas which means that ΔF is increased and $k_{\text{Vac. emiss.}}$ is reduced.

The second way in which gas atoms can aid in nucleation is to prohibit the subcritical embryos from completely collapsing. Qualitatively, this is obvious in that a cluster of i vacancies cannot get smaller than the space occupied by j helium atoms in the cluster. Hence, the number of potential nuclei will continually increase during a steady state irradiation.

Obviously, the helium cannot have these beneficial effects unless it can actually encounter the voids and be incorporated into them. This means that the flux of helium atoms must be comparable to the flux of vacancies. Wiedersich et al.⁽⁵⁾ have shown that for well annealed Ni (dislocation density $\sim 5 \times 10^9 \text{ cm}^{-2}$) irradiated at 0.4 the melting point at a $10^{-3} \text{ dpa sec}^{-1}$ damage rate, the flux of helium atoms to voids exceeds the net flux of vacancies when the bulk helium concentration exceeds the very small concentration level of $\sim 10^{-3}$ appm. For helium concentrations much below that value, void nucleation in extremely pure metals (like the Al used in this study) is governed by homogeneous nucleation and that rate is obviously not high enough in the temperature-damage range of these studies to produce visible voids. It is interesting to note that Paken⁽²⁵⁾ found a threshold for void formation at $1.5 \times 10^{19} \text{ n cm}^{-2} \text{ sec}^{-1}$ which corresponds to 6×10^{-3} appm helium. The dpa rate in that study was $\sim 10^{-6} \text{ dpa sec}^{-1}$.

Future work at higher damage levels will be performed to see if enough nucleation sites can be eventually built up to produce a visible void structure

in Al bombarded Al. The only other such work on Al, by Mazey et al.⁽²⁸⁾, circumvented the nucleation problem by doping this sample with 100 appm helium prior to irradiation.

The absence of voids in the 50-75°C at ~0.5-0.6 dpa range for samples containing 1 and 10 ppm helium concentrations is felt to be a fluence effect as opposed to any change in damage mechanism. Mazey et al.⁽²⁸⁾ observed voids in samples which contained 100 appm and were bombarded with 400 keV Al⁺ ions at 50 and 75°C to dpa values of ~15-30 (on the UW dpa scale.) The lack of observed voids in this study is probably due to the increased number of helium assisted nucleation sites and the effect that such a large density of traps has on lowering the effective supersaturation level of vacancies. Future studies at higher fluences must be conducted before more definite conclusions can be drawn.

Finally, the average void size and density vary in a manner consistent with current theories on void formation. The average diameter of ~150 Å in the 0.1 appm He containing sample is somewhat larger than those in Mazey's study, but this can be attributed to the higher helium content of the later study (even though it was terminated at higher total damage levels). This difference is also shown in the 75°C specimen (180 Å in this work versus 140 Å in Mazey's work).

The void size (and density) does not display a strong dependence on the initial helium content. This is somewhat surprising in view of Mazey's results which showed at least a factor of 10 larger void density in 100 appm He containing material. The difference may be due to the total damage accumulated and irradiation at higher fluences at Wisconsin may indeed reveal the effect of helium on size and number density. We expect that we are close to the

fluence threshold for void formation on the basis of work done by Packer.⁽²⁵⁾ He found that no voids could be formed below $1.5 \times 10^{19} \text{ n/cm}^2$ which is roughly 0.2 dpa using reported displacement cross section for Al. Since the majority of the present work was conducted at dpa levels of 0.26 to 0.68 dpa, the fluence argument appears to most plausible at the present time.

Finally, it was reported in the last progress report⁽³¹⁾ that a few, rather unorthodox voids had been formed in pure Al after Al ion bombardment at 75°C. However, those voids, which appeared to be elongated in the $\langle 011 \rangle$ directions, were quite inhomogeneously distributed and of rather low density. We were unable to repeat those observations under the present conditions and therefore we tend to dismiss those observations as not typical of the current system. Certainly the results of 27 samples carry considerably more weight than one observation.

5. Conclusion

Helium has a very dramatic effect on void formation in pure, well annealed Al. This effect is consistent with existing theories of void nucleation in the presence of mobile helium atoms, and shows that helium contents of less than 0.1 appm are required for void nucleation in aluminum.

B. EN Tandem Studies

1.) Ion Source Progress

The source of metal ions SPIGS (Sputter ION Source)⁽³⁶⁾ that is used for the radiation damage studies on the EN tandem was discussed in the last report.⁽³¹⁾ Below is an update of the information contained in that report.

The beams that SPIGS has produced to date are shown in Table 2. The V^- and Ta^- beams are new. Since the acceptance of the tandem is $25\text{mm mrad MeV}^{1/2}$ for $V_{\text{DOSE}} = 5\text{ MV}$ and SPIGS beams have emittance values $\lesssim 6\text{mm mrad MeV}^{1/2}$ (90% of beam), beams from SPIGS are acceptable for use on the accelerator. Doucas and McK Hyder,³⁷ report that the C^- beam from UNIS (the surface ionization sputter source of Middleton and Adams⁽³⁸⁾) has an emittance of $\approx 30\text{mm mrad MeV}^{1/2}$ (90% of beam). Beams with emittance values that large (1) probably would cause damage to the tandem accelerator beam tubes and (2) probably would have low transmission through the accelerator. Therefore our present efforts are on improving SPIGS and not acquiring a UNIS source.

We have discovered that it is not necessary to separately heat the Cs oven on SPIGS. The excess power from the Penning discharge is sufficient for this purpose. The resulting lower power input to the source means less Cs consumption and less Cs accumulation inside the source and therefore longer operating lifetime (up to 100 hours for Cu^-). Typical operating parameters for Cu^- are shown in Table 3.

Table 2
Negative ion Beams from SPIGS

Beam	Cathode Material	Discharge Gas	Analyzed Beam Current (μA)	Emittance (mm mrad $\text{MeV}^{1/2}$)
C^-	Graphite	Ar+Cs	3	4.5
O^-	V	O_2	12	4.5
V^-	V	Ar+Cs	0.1	---
Ni^-	Ni	Ar+Cs	4	---
Cu^-	Cu	Ar+Cs	8	6.1
Nb^-	Nb	Ar+Cs	0.2	---
Ta^-	Ta	Ar+Cs	0.2	---

Table 3
Typical operating parameters of SPIGS for Cu^-

Parameter	Arc Gas	
	Ar only	Ar + Cs
Anode voltage	2250V	2250V
Anode current	17 mA	26 mA
Magnetic field	650-800 G	650-800 G
Source pressure	7×10^{-2} mm Hg ^a	7×10^{-2} mm Hg ^a
Extractor voltage	6.5 kV	6.5 kV
Cs vapor pressure	$\sim 10^{-7}$ mm Hg ^b	$\sim 10^{-5}$ mm Hg ^c
Cu^- current (analyzed)	0.05 μA	5.0 μA

- a) Estimated from known pumping speeds, pressures, and conductances
 b) Vapor pressure of Cs at room temperature
 c) Vapor pressure of Cs at 40°C

2.) Chamber

a.) General Description of the Beam Line and Sample Chamber

A schematic of the U.W. tandem accelerator facility showing the location of the sample chamber was given in Fig. 20 of the last report.⁽³¹⁾ The new beam line and sample chamber discussed on pp. 33-35 and shown in Fig. 25 of that report, is completed and in operation. Preliminary data for Cu irradiation of V has been obtained (see section 4).

An updated schematic drawing of the new beam line and sample chamber is given in Figure 6 and a photograph shown in Figure 7. In order to isolate the sample chamber vacuum from the tandem accelerator vacuum, the beam line is divided into three separate pumping stages. A 200 l/sec diffusion pump with a liquid nitrogen trap provides a vacuum in the low 10^{-6} mm Hg range for the high pressure stage, a 300 l/sec orbitron pump provides 10^{-7} mm Hg for the intermediate pressure stage, and a 1000 l/sec orbitron pump provides 10^{-9} mm Hg for the sample chamber stage. A 9.5 mm Ta aperture and LN cold trap (anti-creep design) separates the intermediate pressure stage from the high pressure stage and a series of fifteen 6.4 mm dia. Ta apertures isolates the sample chamber state from the intermediate stage.

The devices which the beam encounters (see Figure 6) and a brief description of their function follows. The 90° analyzing magnet is used to deflect the beam $1/4^\circ$ for crude magnetic analysis (elimination of neutrals and low Z ions from the accelerator). A set of vertical and horizontal slits are used to locate the beam for transport purposes. The beam then encounters the 9.5 mm Ta aperture which protects the LN anti-creep cold trap. The anti-creep trap prevents hydrocarbons from the accelerator from entering the high vacuum stage and a series of fifteen 6.4 mm dia. Ta apertures isolates the sample chamber

Figure 6 - Ultra High Vacuum Chamber for Heavy Ion Bombardment Studies

Figure 7 - Photograph of Ultra High Vacuum Chamber Shown in Figure 6

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from the intermediate stage.

A combined Faraday cup-mask arrangement on one adjustable arm allows either (1) final collimation of the beam with the mask to 3 mm diameter (with a 3mm dia. Ta aperture) before sample irradiation or (2) measurement of the total beam current with the Faraday Cup. The beam then enters the sample chamber which contains a radiation heater for heating the samples, an adjustable sample holder which allows irradiation of multiple samples, a Bayard-Alpert gauge for pressure measurement, a residual gas analyzer for measuring the partial pressure composition of the vacuum environment, and chromel-alumel thermocouples for temperature measurement. The sample heater and sample holder are discussed in Section b. below. If desired, the beam can be transported through the sample chamber to the charge state measuring device (discussed in Section 3 below) for measurement of the composition of the beam. It can also pass through the charge state device to allow visual observation on a quartz viewer. An optical telescope mounted behind the quartz viewer allows alignment of the various beam defining apertures, the sample holder, and the charge state device. In the near future, we will add a beam scanner (on order) between the sample chamber and charge state device, and a Faraday cup (under construction) between the charge state device and quartz viewer. The beam scanner will determine beam uniformity and the Faraday cup, beam intensity.

b.) Sample holder and sample heater

The new sample holder and sample heater are shown schematically in Figure 8. The beam is collimated to 3 mm diameter by the Ta mask before it encounters the heat shield - heater - supressor screen arrangement. The beam then passes through a 9.5 mm hole in the heat shield (the heat shield has 15 close-wound layers of 1 mil Ta⁽³⁹⁾), a 9.5 mm hole in the heater (the sample heater is a single layer of 1 mil Ta^(40,41)), and a 9.5 mm hole in the supressor (the supressor is a single

layer of Ta wire screen which is 72% transparent) before striking the sample.

The samples (Fig. 9) are held by the sample holder as shown in Fig. 10. All construction materials are Ta with the exception of the sample, the chromel-alumel thermocouples and the insulators. In order to allow the beam to pass through the sample chamber to either the charge state device or the quartz viewer, there is one 5 mm hole between each of the six sample positions. The vertical position of the sample holder is adjustable to allow selection of one of the six sample positions or one of the five holes. The exit holes of the suppressor screen, heater, and heat shield are also 9.5 mm dia.

After the beam exits the sample chamber, its composition can be measured with the charge state device (see section 3 below) or its shape determined with the quartz viewer. In the future its shape and intensity can be measured by the new scanner and Faraday cup respectively.

c.) Vacuum Conditions

The vacuum environment in which the samples are irradiated has been considerably improved. With the old sample chamber (stainless steel sample holder) the irradiations were performed at 400-600°C and pressures $\sim 10^{-6}$ mm Hg. The new sample chamber (Ta sample holder) allows irradiation at 700°C and pressures $\sim 5 \times 10^{-9}$ mm Hg. The sample chamber environment is so clean that we anneal our specimens in situ. The vanadium samples are annealed for 1 hour at $\sim 1050^\circ\text{C}$ and pressures $\lesssim 5 \times 10^{-8}$ mm Hg.

A residual gas analyzer (RGA) (Monitorr 702 manufactured by Aero Vac Division of High Voltage Engineering Corporation) is attached to the sample chamber. The RGA is used to determine the vacuum conditions during the sample annealing and irradiations (the RGA is also used as a He leak chaser).

Figure 9 - Geometry of Sample Used for Heavy Ion Bombardment Studies. All Dimensions in Centimeters.

Front View

Side View

Figure 10 - Schematic of Sample Assembly for Heavy Ion Bombardment. All Dimensions in Centimeters.

Typical partial pressure composition of the residual gas during an anneal, at the temperature of a typical irradiation, and at room temperature are shown in Figure 11. In all cases the bulk of the residual gas is hydrogen, presumably from the stainless steel components of the sample chamber. (42)

3.) Charge State Measurement

A gas stripper canal (N_2 gas) is located inside the terminal of the tandem accelerator. When the heavy ion beams (i.e. Cu^- & V^-) traverse this canal, two or more electrons may be removed from the heavy ions by atomic interactions with N_2 molecules. Positive ions of N_2 and N can also be produced by the heavy ion beam. The ~~positive heavy ions~~ and the N_2 and N ions are accelerated to the exit of the accelerator. The desired positive heavy ions are then transported to the sample chamber but unfortunately it is possible for undesired positive N_2 and N ions also to be transported to the sample chamber along with the heavy ions.

The only beam transport elements located between the tandem exit and the sample are magnetic devices. Thus, all beams having the selected magnetic rigidity will be transported to the sample.

For example, $^{63}Cu^{3+}$ and $^{14}N_2^{1+}$ ions have identical magnetic rigidity (see Table 4). In the absence of steering effects by the HE quadrupole lens, the $^{63}Cu^{3+}$ and $^{14}N_2^{1+}$ beams will be transported simultaneously to the sample. Fortunately, if either beam is centered off the magnetic axis of the lens, the HE quadrupole can introduce additional spatial separation of the beams (43) (such spatial separation is actually observed with the HE beam scanner). The HE quadrupole lens can also cause spatial separation of the various Cu charge states provided they are located off the magnetic axis of the lens. (43) Despite the helpful steering effects, care must be

PARTIAL PRESSURE COMPOSITION OF SAMPLE
CHAMBER RESIDUAL GAS

exercised in selecting the component of the beam used for sample bombardment.

Table 4. Magnetic rigidity, $\frac{mE}{q} \left[\frac{\text{amu MeV}}{2 e} \right]$ of Cu^+ , N_2^+ , and N^+ ions for $V_{\text{Dome}} = 3.5 \text{ MV}$.

Ion	$\frac{mE}{q}$	Ion	$\frac{mE}{q}$	Ion	$\frac{mE}{q}$
$^{63}\text{Cu}^{1+}$	441	$^{14}\text{N}_2^{1+}$	98	$^{14}\text{N}^{1+}$	49
$^{63}\text{Cu}^{2+}$	165				
$^{63}\text{Cu}^{3+}$	98	$^{14}\text{N}_2^{2+}$	49	$^{14}\text{N}^{2+}$	24.5
$^{63}\text{Cu}^{4+}$	69				
$^{63}\text{Cu}^{5+}$	53	$^{14}\text{N}_2^{3+}$	32.7	$^{14}\text{N}^{3+}$	16.3
$^{63}\text{Cu}^{6+}$	43				

The beam from the tandem undergoes slight magnetic analysis ($\sim 1/4^\circ$) by the 90° analyzing magnet before entering the sample chamber beam line. This analysis (1) prevents neutrals from striking the samples and (2) allows separation of light and heavy beam components. A charge state device is used to determine the composition of the beam⁽⁴⁴⁾ The charge state device consists of a solid-state detector which records beam particles elastically scattered through 90° by a carbon backed gold foil.

Figure 12 shows a spectrum obtained with the charge state device during a preliminary run (the beam has passed through the 3 mm dia. mask). Visible are the Cu^{2+} , Cu^{3+} , Cu^{4+} and N_2^+ components of the beam. The portion of the beam selected with the steering devices to irradiate the samples is shown in Figure 13. After correcting for the $\frac{1}{E^2}$ dependence of the Rutherford cross section, the composition of the beam is found to be $\sim 95\% \text{ Cu}^{3+}$ and $\sim 5\% \text{ Cu}^{1+}$ (particle current). After an

Figure 12 - Beam Composition before charge state selection with steering devices.

Figure 13 - Beam Composition Selected for irradiation. Start of run.

COUNTS (x)

Cu^{1+}

Cu^{3+}

0 50 100 150 200 250 300 350

CHANNEL

irradiation run (9 hours later), the beam composition was remeasured. The result is shown in Figure 14. Not only had the Cu charge state distribution changed, but we found a N_2^{1+} beam striking the sample. The new beam composition was ~3% N_2^+ , 51% Cu^{3+} and ~46% Cu^{4+} (particle current). Thus, even though it is possible to select one component of the heavy ion beam (Fig. 13), our experience shows that the composition of the beam needs to be carefully monitored to insure (1) that no N_2^+ (or N^+) ions strike the samples (resulting in contamination) and (2) that we know the charge state composition of the beam (so that the dpa value may be determined).

Figure 14 - Beam Composition at
end of 9 hour run.

COUNTS ($\times 10$)

CHANNEL

4. Microstructure

The major problem in damage studies of vanadium and other b.c.c. metals lies in their high affinity for carbon, nitrogen and oxygen. This problem is exacerbated by high temperature. The major achievement of this year's work has been the building of a specimen chamber for irradiation capable of maintaining vacua better than 10^{-8} torr at specimen temperatures as high as 1100°C . An associated problem is to ensure that the irradiating beam itself does not introduce impurities. The results presented here indicate -

- a) that we can now anneal specimens of vanadium at 1100°C for 1 hour without introducing contamination as indicated by precipitate formation.
- b) that irradiation can be completed at 700°C for 15 hours with doses as high as ~ 50 dpa without introducing contaminating precipitates.

A further series of problems in obtaining high quality thin foils of damaged regions in vanadium has also been overcome during the year's work.

Fig. 15 is a micrograph of the vanadium as received. The structure is typical of cold worked material with a high dislocation density in a cellular sub-structure. The structure is precipitate free so that we can be sure that any subsequent precipitation is the result of contamination during annealing or irradiation. Fig. 16 is a micrograph of the structure obtained after annealing at 1200°C . The dislocation density throughout the foil is now low, as required, although in the region shown it is higher than average. Once again, the important feature is the absence of precipitation. This is only obtained by careful control of the vacuum pumping to maintain a vacuum of $<10^{-7}$ torr in conjunction with slowly increasing temperature. This heating sequence can take as long as 96 hours although recent modifications to the specimen holder have reduced this to 24 hours. If the vacuum is allowed to

Figure 15. As received vanadium.

Figure 16. Vanadium annealed at 1100°C for 1 hour.

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deteriorate to $\geq 10^{-7}$ torr, the foils become contaminated with large numbers of fine "black dot" precipitates which did not give any identifiable diffraction reflections.

A different form of precipitate has been obtained when irradiating beams containing nitrogen ions (from the stripping gas) have been used (Fig. 17). This micrograph shows such precipitates associated with voids forming in the damaged region. The plate-shaped precipitates appear on the {100} vanadium planes. No extra diffraction reflections occur (Fig. 18) which implies that the precipitates have a similar atomic arrangement (in the plane of the plates) to that of the matrix {100} planes.

Vanadium carbide (VC), vanadium nitride (VN) and the mixed carbo-nitride all have fcc structures with "a" ~ 4.13 Å. The misfit between the {100} planes of this structure and the {100} vanadium planes is $< 0.1\%$. The reflections from the precipitate structure then would lie so close to those of the matrix that they could not be distinguished (Fig. 18C). The misfit normal to the precipitate plates is 25% with this structural analysis, which indicates that considerable strain would be associated with growth of the plates in this dimension, which is why they remain platelike. In view of all this, we conclude that the precipitates are likely to be fcc vanadium nitride. All the precipitates observed after this irradiation were associated with voids which indicates that either the precipitates or the voids, or both have to overcome a substantial nucleation barrier. It is not possible at this stage to decide whether voids nucleate on pre-existing precipitates or vice versa. However, voids forming in the absence of precipitates do have difficulty nucleating since they appear to be very heterogeneously distributed (Figs. 19 & 20). From this, one might tentatively conclude that pre-existing precipitates

Figure 17. Voids and precipitates in vanadium irradiated with Cu and N ion beams. Note the black spot damage due to HVEM analysis with 1 MeV electrons. Damage level is approximately 5 dpa at 700°C.

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Fig. 18 a) Diffraction pattern from foil containing precipitates like those in Fig. 17
b) Indexing of above pattern on the bcc vanadium unit cell
c) Indexing of above pattern based on fcc vanadium nitride.

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Figure 19. Voids in 14 MeV Cu ion irradiated vanadium. Note the distance from the grain boundary on the right. Damage level is approximately 15 dpa at 700°C.

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Figure 20. A row of voids in 14 MeV Cu ion irradiated vanadium. The dark patches on the surface are oxide particles which formed during the post irradiation thinning process. Damage level is approximately 15 dpa at 700°C.

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33206-C-2

(a)

(b)

Figure 21. As Figure 20. Note the blurring of outlines of voids due to the formation of oxide film during post irradiation thinning process. Damage level is approximately 15 dpa at 700°C.

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probably nucleate voids, however, the experimental results illustrated by Figs. 19 and 20 are only the precursors of a series of controlled experiments on nitrogen injection which we hope to carry out to clarify the role of nitrogen during irradiation of this material. A definitive conclusion will await results from these studies.

Figs. 19-21 illustrate the structures obtained for pure copper beam damage at a dose of ~ 15 dpa. The voids are large and very scattered appearing in groups at distances of about $\sim 5000 \text{ \AA}$ from grain boundaries. Such void arrangements indicate that the threshold for void formation has only just been crossed and also constitute good evidence for a nucleation barrier. The association with grain boundaries at intermediate distances suggests that voids are nucleating in regions where the interstitial concentration has dropped below the vacancy concentration due to the greater effectiveness of the grain boundary as a sink for interstitials. The large void size ($\sim 700\text{--}1000 \text{ \AA}$) indicates that growth is easy as compared to nucleation. These results are in contrast with those obtained by Okamoto et al.⁽²⁰⁾ and Kulcinski et al.⁽¹⁸⁾ under poorer vacuum conditions, when a higher concentration of smaller voids was observed for similar doses. Under conditions of difficult void nucleation, it is to be expected that impurity levels will be extremely important since impurities can form nucleation sites. Our results are, therefore, consistent with the higher vacuum conditions in conjunction with difficult void nucleation.

During the year, polishing and thinning techniques have been refined to the point where we are confident of producing substantial transparent regions with good surface quality. The most important developments have been improved jetting conditions and the routine use of a 90% Methanol/10% Perchloric acid electropolishing solution. The extreme affinity of vanadium for oxygen presents

us with difficulties in foil storage. Fig. 21 in fact illustrates the effect of overnight storage in a vacuum of $\sim 10^{-5}$ torr. A thin layer of oxide causes a lack of definition in the void images. In addition, oxide particles form on the surface and also obscure detail. The practice of storing foils has now been abandoned and all work is being done on freshly prepared foils.

The new 100B JEOL electron microscope has become available during the year and has been used for much of this work. This machine includes a facility for scanning transmission electron microscope microscopy (STEM). Preliminary tests have been carried out in the STEM mode and we find that foils which are too thick to reveal any detail whatsoever in the conventional mode, can be penetrated to give satisfactory scanning images. Fig. 22 is an illustration of this showing a dislocation substructure in annealed vanadium, 5000 Å thick, which indicates a resolution of $\sim 20-30$ Å in this mode. This technique will be invaluable in current work on large voids which are too thick to be contained within the foil at thicknesses < 1000 Å. The 1 MV electron microscope on the UW campus has also been used to achieve greater penetration and in fact Fig. 17 was taken using this machine. We expect this machine to be valuable for work on thick foils which is now becoming necessary.

Figure 22. STEM picture of deformed vanadium showing dislocation substructure in a thick ($\sim 5000 \text{ \AA}$) foil.

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C. Electron Irradiation of Pure Aluminum

1. Experimental

A series of electron irradiation damage experiments on pure Al (99.999%) have been completed using a AEI EM-7 electron microscope. The microscope was operated at 1 Mev with a 750 μm condenser aperture opening. Electron beam intensity was measured by a Faraday cage beneath the viewer screen. The electron current measured on the screen with objective aperture withdrawn represents ~75% of the actual current intensity on the specimen. The total beam current measured for these irradiation studies was 8.0×10^{-7} A. The irradiated area was maintained at a size of 15 ± 1 μm diameter spot for all irradiated specimens, and it is assumed that the beam was roughly gaussian in shape. All the irradiation data were taken within 3 microns radius of the beam center area. The average beam flux in the irradiated area was calculated to be 4×10^{18} electrons/ cm^2/sec ; the average displacement rate was 2.4×10^{-4} dpa/sec. The total displacement cross section used for calculation is 60 barns at 1 Mev. All specimens irradiated were bombarded for 1 hour which gave a total average dpa value of 0.87 dpa. The specimen temperature was controlled by a small platinum resistance furnace in a single tilt stage. This stage was designed by Dr. P. Swann, Imperial College, London, and modified in the University of Wisconsin mechanician shop.

Ten specimens were irradiated at nominal temperatures of 27°C, 40°C, 65°C, 90°C, 115°C, 140°C, 165°C, and 190°C. The temperature rise due to beam heating at the fluence used is estimated within the range of 5-10°C. The irradiation temperatures after correction then would be 37°C, 50°C,

100°C, 125°C, 150°C, 175°C and 200°C. The foil thickness of the irradiated area was in the range of 8,100 Å to 20,000 Å. The technique used for foil thickness determination was that described by M. von Heimendahl.⁽⁴⁵⁾ The specimen was dipped in a diluted latex milk of known ball size (0.5µ dia. latex balls were used). The foil thickness can be calculated by knowing the tilting angle and the distance between balls on different sides of the specimen. Two samples were irradiated in the thinner area (~3,000 Å) at 75°C and 100°C for loop analysis. The analysis was done on a JEOL 100B microscope operated at 100 KV.

Table 5 lists the information on the irradiation conditions.

Table 5

Irradiation Conditions for 1 MeV HVEM Studies

Material: 99.99% Al(ORNL)

Foil Thickness: 8,100 Å - 20,000 Å

Beam Flux: 4×10^{18} electrons/cm²/sec

Displacement Rate: 2.4×10^{-4} dpa/sec

Photo Micrographs

		H-0-00							
Sample		-23	19	24	20	25	21	22	18
Temp. °C		37	50	75	100	125	150	175	200
Irrad. Time (min)	dpa*								
0	0	X	X	X	X	X	X	X	X
1	.015		X	X	X	X	X	X	X
2	.029	X							
3	.044		X	X	X	X	X	X	
5	.072	X	X	X	X	X	X	X	
10	.14	X	X	X	X	X	X	X	X
20	.29	X	X	X	X	X	X		
30	.43	X	X	X	X	X	X		
40	.58	X	X	X	X	X	X		
50	.72								
60	.87	X	X	X	X	X	X		

*Average value in irradiated zone ≈ 0.75 (dpa)_{max}

2. Results

a. General Results

Figure 23 shows irradiated areas after 1 hour of irradiation. Fig. 23-A refers to irradiation at 37°C, and Fig. 23-B to 125°C. The irradiated areas show a consistent size of $15 \mu\text{m} \pm 1 \mu\text{m}$, and the damage is also seen to be quite uniform across the irradiated zone except at the very outer rim.

Dislocation loops are the general microstructure seen in the irradiated area. There are some, although very few, void-like structures in the damage areas throughout the entire irradiation temperature range. However, these have not been verified as voids in the present study. The number of these void-like defects is about one to two on a 6.5 X 9 cm film at 20,000X. There is no damage produced when the irradiation temperatures exceed 175°C. Fig. 24 shows the loop development during the irradiation process. The density of loops decreases both with increasing temperature and damage. Above 100°C, the density of loops drops drastically. Fig. 25 and Table 6 show that the density of loops changes with both temperature and dose. The loop density reaches a maximum after 10 minutes of irradiation at temperatures of 27°C and 50°C. However, the density of loops reaches a maximum within the very first minute of irradiation for temperatures between 75°C and 125°C. At 150°C, there are hardly any dislocation loops developed in the first minute; the loop density reaches a maximum after three minutes of irradiation.

The dislocation loops induced by electron bombardment have two different forms, namely prismatic loops and Frank sessile faulted loops. In the case of faulted loops, some are double faulted.

The number of these double-faulted loops becomes larger at the higher irradiation temperatures. However, faulted loops with a wide variety of configurations form at all temperatures. A small fraction of prismatic loops

Figure 23A. Damage in aluminum irradiated with 1 MeV electrons at 37°C. Total damage 0.87 dpa.

Figure 23B. Damage in aluminum irradiated with 1 MeV electrons at 135°C. Total damage 0.87 dpa.

37°C

50°C

75°C

100°C

125°C

150°C

Figure 24A. Microstructures of aluminum irradiated at different temperatures to 0.45 dpa with 1 MeV electrons.

.072 dpa

.14 dpa

.29 dpa

.43 dpa

.87 dpa

Figure 24B. Microstructures of aluminum irradiated with 1 MeV electrons at 50°C to different dpa values.

.072 dpa

.14 dpa

.29 dpa

.43 dpa

.87 dpa

Figure 24C. Microstructures of aluminum irradiated with 1 MeV electrons at 75°C to different dpa values.

.072 dpa

.14 dpa

.29 dpa

.43 dpa

.87 dpa

0.87 dpa

Figure 24D. Damage in aluminum irradiated with 1 MeV electrons at 125°C to different dpa values.

Figure 25 - Effect of Damage Level and Temperature on the Loop Density in Well Annealed Al Bombarded with 1 MeV Electrons

Table 6

Density of Loops (# of loops/cm³)

Irradiation Temperature °C	37	50	75	100	125	150
<u>DPA</u>						
.015						3.2×10^{11}
.029	1.74×10^{14}					
.044		1.87×10^{14}	1.46×10^{14}	1.64×10^{14}	7.6×10^{13}	7.8×10^{12}
0.072	1.78×10^{14}	2.07×10^{14}	1.17×10^{14}		7.3×10^{13}	6.65×10^{12}
0.14	1.78×10^{14}	1.62×10^{14}	1.03×10^{14}	1.03×10^{14}	5.2×10^{13}	6.01×10^{12}
0.29	1.62×10^{14}	1.46×10^{14}	6.3×10^{13}	8.9×10^{13}	5.1×10^{13}	4.11×10^{12}
0.43	1.27×10^{14}	1.28×10^{14}	7.3×10^{13}	5.2×10^{13}	3.8×10^{13}	1.24×10^{12}
0.58	1.28×10^{14}	1.17×10^{14}	6.4×10^{13}	5.1×10^{13}	3.2×10^{13}	9.41×10^{11}
0.72						6.47×10^{11}
0.87	6.92×10^{14}	1.09×10^{14}	5.9×10^{13}	3.2×10^{13}	3.2×10^{13}	6.57×10^{11}

grow in size as the irradiation time and irradiation temperature increase. The majority of the prismatic loops have a size of $\sim 500\text{\AA}$, and reach this size within 20 minutes of irradiation time at all temperatures (Fig. 26). The size of the faulted loops, however, grows with irradiation time and temperature. For instance, at 60 minutes of irradiation time, the size of faulted loops changes from $\sim 500\text{\AA}$ to $2,000\text{\AA}$ for an irradiation temperature change of 37°C to 125°C .

Fig. 27 shows the change in defect structure when irradiation is at 150°C . The beam size was maintained at 15μ diameter, but the defects developed only within the central $\sim 8\mu$ diameter area. A more concentrated beam, $\sim 3\mu$ dia., was used to irradiate both thinner ($\sim 4,000\text{\AA}$) and extremely thick ($> 2.5\mu$) areas. Dislocation loops produced by irradiation filled the entire irradiated area (Fig. 28). Of course, the displacement rate here is about 25 X greater than that corresponding to the 15μ dia. beam. Fig 27 also shows that most of the dislocation loops were annealed out after 30 minutes of irradiation. There were only a few faulted loops left to continuously grow. A few prismatic loops were generated occasionally here and there and then annealed out.

b. Faulted Loops

No determination of the nature of the faulted loops was made in the present study. Only the general features of the faulted loops will be described.

Simple Faulted Loops (Fig. 29)

The simple faulted loops usually appear as dark spots or light spots depending on the diffraction condition. In some diffraction conditions, the faulted loops appear to have light-dark shadows or thick fringes inside the loop. Simple faulted loops appear more often at lower irradiation temperatures and in lower dose specimens.

Figure 26A. Faulted and double-faulted loops in aluminum irradiated with 1 MeV electrons at 37°C. Total damage 0.90 dpa.

Figure 26B. Faulted and double-faulted loops in aluminum irradiated with 1 MeV electrons at 125°C. Total damage 0.90 dpa.

.015 dpa

.044 dpa

.14 dpa

.29 dpa

.58 dpa

.87 dpa

Figure 27. 1 MeV electron damage in aluminum irradiated at 150°C.

Figure 28. 1 MeV electron
damage in aluminum
irradiated at 150°C
with a $\sim 3\mu$ diam. beam
to 0.125 dpa.

- A. Foil thickness 4000Å
- B. Foil thickness $\sim 2.5\mu$

28A

28B

Figure 29. Faulted loops in 1 MeV electron irradiated aluminum at 100°C, 0.87 dpa.

Double Faulted Loops (Figs. 30, 31)

A good fraction of faulted loops formed by irradiation at higher temperatures are double faulted. They usually appear as triangles or hexagons superimposed on a circle or on an ellipse. Some of these triangles and hexagons have sharp corners. Others have rounded corners and appear as irregular polygons. Sometimes the triangular part of the faulted loop is off-centre. No triple-faulted loops were detected in the dose range of this study.

3. Nature of the Prismatic Loop Analysis.

Two samples were irradiated in a thinner area ($\sim 3,000 \text{ \AA}$) at 75°C and 100°C respectively. They were then analyzed in a JEOL 100B microscope operated at 100KV. Only the nature of the prismatic loops was determined. The method used to determine whether the loops are of interstitial or vacancy type is as follows:

A. Burgers vector is defined by following the FS/RH convention for the perfect crystal of Hirsch et al. ⁽⁴⁶⁾

B. The contrast from loops follows the general rules

a. $\bar{g} \cdot \bar{b} < 0$ for outside contrast ($s > 0$)

$\bar{g} \cdot \bar{b} > 0$ for inside contrast ($s > 0$)

b. $\bar{b} \cdot \bar{n} > 0$ for vacancy type

$\bar{b} \cdot \bar{n} < 0$ for interstitial type

Figure 30. Double faulted loops in aluminum after irradiation with 1 MeV electrons at 125°C, to 0.87 dpa.

Triangle superimposed on
a circle or ellipse

Polygon on a circle

Hexagon on a circle

Off-centre triangles on a circle

Figure 31. Different configurations of double faulted loops
in electron irradiated aluminum.

t = triangles on a circle
h = hexagon on a circle
p = polygon on a circle
o = off-centre triangle on a circle

C. $\bar{g}$ Vector is determined by the technique described by Wu et al. (47)
 Except for the faulted loops and the loops laying parallel to the foil plane, about 90% of the prismatic loops can be characterized. The result shows that 54% of the prismatic loops are interstitial in nature, 36% are of vacancy type and the remaining 10% are indeterminate. Examples are given in Fig. 32; this figure correlates with Table 7.

4. Discussion

The main difference between this work and that reported in the last progress report⁽³¹⁾ is that well annealed Al has now been irradiated versus cold worked Al in the previous study. However, the same general features of the damage have been observed;

a) no statistically meaningful void formation in the temperature range of 30-175°C

b) observation of a high density of faulted and unfaulted loops

c) a decrease in loop density after damage levels of 0.1 dpa have been reached

There were some minor differences noticed

a) the loop density in the well annealed Al at ~100°C range was $\sim 10^{13} \text{ cm}^{-3}$ versus 10^{14} cm^{-3} at 100°C in the cold worked Al

b) no triple faulted loops were noticed in the present study

c) the percentage of single faulted loops was lower in the well annealed Al than in the cold worked samples

None of the latter observations are felt to be of major significance because of the difficulty in working with cold worked samples. However, it is reasonable to assume that since the defect loops can climb or glide to surfaces with greater ease in the well annealed structure that the overall density of loops should be less .

The lack of triply faulted loops is probably due to the fact that the highest damage levels in the well annealed Al were a factor of 10 lower than the cold worked samples. Since triple faulted loops must grow from double faulted loops and there were only a few of those around toward the end of the

present irradiation, it is not surprising that we failed to observe these advanced loop stages.

It is not known why there were fewer Frank faulted loops observed and future work will concentrate on verifying this effect.

A major advance during this period was the identification of the nature of the loops formed. If only those identified are counted, we find that 60% are interstitial and 40% are vacancy. This correlates very well with Rao⁽⁴⁸⁾ who found that, of the loops he could identify after 650 keV electron bombardment, 58% were interstitial and 42% vacancy. Such an analysis still leaves open the possibility that there are small, unresolvable vacancy clusters (or loops) in the Al which are mainly vacancy in nature. However, it is obvious from this work, and others reported to date^(29-32,48) that the nucleation of void embryo in high purity Al with 1 MeV electrons is exceedingly difficult. An obvious answer is that there are not sufficient spikes available for void nucleation but our work with Al ion bombardment shows that this is not the case. The existence of spikes may be necessary but not sufficient for void nucleation. On the other hand, the addition of helium to the Al samples before electron bombardment⁽³¹⁾ did not result in void formation either. This is in direct conflict with the Al ion work reported here. Hence, one might be tempted to also state that the existence of helium (or some gas atom) is necessary but not sufficient. A logical statement might now be -

"Void nucleation in Al requires both the existence of helium
(or some gas atom) and spikes."

This question will be addressed in detail in the next reporting period.

Figure 32. Bright field micrographs of aluminum irradiated at 100°C with 1 MeV electrons.

Table 7

Details of the Loop Analysis

Loops	$\bar{g}$		$\bar{g} \cdot \bar{b}$	$\bar{b}$	$\bar{n}$	$\bar{b} \cdot \bar{n}$	
A	$\bar{2}20$	outside	<0	$a/2 [\bar{1} 0 \bar{1}]$	$[1 \bar{1} 1]$	<0	interstitial
	$\bar{2}20$	inside	>0	$a/2 [\bar{1} 0 \bar{1}]$			
B	$\bar{2}02$	outside	<0	$a/2 [0 \bar{1} \bar{1}]$	$[\bar{1} 1 1]$	<0	interstitial
	$20\bar{2}$	inside	>0	$a/2 [0 \bar{1} \bar{1}]$			
C	$0\bar{2}2$	outside	<0	$a/2 [1 1 0]$	$[1 1 \bar{1}]$	>0	vacancy
	$02\bar{2}$	inside	>0	$a/2 [1 1 0]$			

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