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

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The photomicrograph on the front cover is the microstructure of high purity (99.999%) Al after 1 MeV electron bombardment at 100°C. The damage rate was $\sim 8 \times 10^{-4}$ dpa per sec and the total damage was 3.6 dpa. The microstructure is composed of several types of loops; unfaulted, faulted, and double faulted. The total loop density in this sample is approximately 10^{14} cm^{-3} and the average loop size is $\sim 1200 \text{ \AA}$. No voids were found in this material.

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Preface

This document is not intended to be a typical quarterly report which has a wide external distribution and which only summarizes positive results. The main purpose of this report is for internal communication with the Nuclear Engineering, Materials Science and Physics Departments of the University of Wisconsin as well as to keep the sponsor of this work, namely the Metallurgy and Material Branch of the Division of Research, USAEC, informed of our progress. The reporting in this and subsequent reports may be quite detailed and may even be quite frank about some errors in our theoretical or experimental approach. However, it is felt that such a report is valuable from the standpoint of both the students and staff members because it will provide an accurate description of how we arrived at certain conclusions, provide historical perspective for new members of this study and lay the groundwork for more polished topical reports and open literature papers. It is therefore requested that the contents of this and other reports not be referenced without the consent of the authors.

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SUMMARY

The first 1 MeV electron microscopy studies of high purity Al have been conducted and the samples are currently being analyzed. The studies were performed at WADCO because the UW EM-7 microscope acceptance tests have been postponed to November 1972. Four samples were irradiated at $\sim 8 \times 10^{-4}$ dpa sec^{-1} to 8.1 dpa at temperatures of 100, 150, 175 and 200°C. No voids ($> 30\text{\AA}$) were observed under any of the above conditions. The microstructures consisted of a high density of faulted and unfaulted loops and the density of such loops varied from 1×10^{14} at 100°C to $< 10^{12}$ at 175°C and no damage was observed after 200°C bombardment.

A total of 49 Al samples have been doped with 0.1, 1, and 10 appm He for future Al⁺ and electron bombardment studies. These doping experiments have delayed the operation of the heavy ion source by two months.

The physicon heavy ion source has been bench tested and has produced 2 μA of Al ions at 15 kV. This is not the optimum amount of current because of different optics in the accelerator but such a beam is entirely adequate for the heavy ion work. The source has been mounted in the accelerator and beam tests will begin shortly.

Several minor modifications have been made to the 2 MeV electron accelerator and thus far 2 MeV beam fluxes of $500 \mu\text{A}/\text{cm}^2$ (3×10^{15} electrons/ $\text{cm}^2\text{-sec}$) have been observed on a 5 mm diameter sample. This beam results in a 3×10^{-7} dpa/sec rate.

Samples of high purity Al and Al-Si, Al-Cu and Al-Ni alloys have been received from ORNL.

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 a high energy beam 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, and Zn will help to understand the role of these impurities in nucleating voids. This study will concentrate on high purity V and sample of 99.999% Al already extensively analyzed by ORNL scientists after neutron irradiation.

A. Progress in Experimental Programs

1. High Voltage Electron Microscopy (HVEM)

a. First Irradiation Results

The first HVEM study on high purity aluminum (99.999%, see Section B-4) has been conducted with the 1 MeV electron microscope at Westinghouse Hanford Corporation in Richland, Washington.¹ We can only make general observations at this time as the photo micrographs are currently being analyzed.

The information on the irradiation conditions is given in Table 1. The displacement rates and total damage levels were calculated on the basis of a displacement cross section of 60.5 barns for 1 MeV electrons (see Section B-1 for details of the calculation). The displacement rates for this test were held constant at 8×10^{-4} dpa sec⁻¹ for all samples. Photo micrographs were taken in the irradiated area at dpa levels ranging from 0.25 to 8.1. The irradiation temperatures were nominally 100, 150, 175 and 200°C. The sample thicknesses ranged from 11,000 to 43,000 Å.

Some general observations of the damage can be made at this time.

- No voids larger than 30 Å were observed in any of the samples. This is somewhat surprising in view of ORNL results for similar high purity Al samples irradiated at much lower damage levels and temperatures of 50-100°C.²

Table I

Summary of Irradiation Parameters for First HVEM Study

Material - 99.999% Al

Thickness of Samples	H-0-00-1	1.3 - 2.1 microns ^a
	H-0-00-2	1.1 - 4.3 microns ^b
	H-0-00-3	1.4 - 2.0 microns ^b
	H-0-00-4	- 2.5 microns ^b

Beam Flux - 1.36×10^{19} electrons/cm²/secDisplacement Rate - 8.2×10^{-4} dpa/secPhoto Micrographs

Sample # Temperature ^c		H-0-00-3 100°C	H-0-00-1 150°C	H-0-00-4 175°C	H-0-00-2 200°C
<u>Time (min)</u>	<u>dpa</u>				
0	0	X	X	X	X
5	0.25	X	X	X	X
11	0.54	X	X	X	X
17	0.84	X	X	X	X
33	1.6	X	X	X	X
60	3.0	X	X	X	X
83	4.1	X	X	X	X
115	5.7	X	X	X	X
165	8.1	X	X	X	X

^aactual irradiated zone^birradiated zone falls within this range^cto be adjusted when calibration data is available

- The temperature of irradiation is very critical to the density of dislocation loops in Al. It appears that the loop density drops off substantially between 100 to 150°C, drastically between 150 and 175°C, and we could observe essentially no damage in the sample during a 200°C irradiation. These results are in conflict with those of Shiraishi et. al.³ who observed no damage above 70°C. However, our foil thicknesses were much larger than theirs and it is possible that surface denuding prohibited them from seeing damage at temperatures above 70°C.
- A very large fraction of dislocation loops in the samples are faulted. There are also many doubly faulted loops in which the inner loop is either triangular or hexagonal in shape.

More quantitative observations will be made during the next quarter.

Some examples of the microstructures are shown in Figures 1-4.

Figure 1 shows the extent of a typical irradiated area. Note that the beam diameter is ~ 13.9 microns. The electron density is not uniform across the irradiated zone and we have assumed a Gaussian distribution to calculate the damage profile.

Figure 2 shows the development of the microstructure at 150°C after damages of 0.25, 1.6, 4.1 and 8.1 dpa. It

appears that the loop microstructure developed early in the irradiation and only the size seems to be changed by the higher fluences.

Figure 3 shows the effect of increasing irradiation temperature at 3 dpa. This figure emphasizes the fact that the loop density is drastically decreased between 150 and 175°C. The lack of any loop damage at 200°C ($\sim 0.5 T_m$) is expected from previous studies of irradiated metals. The dislocations in Figures 3c and 3d result mainly from the original dislocation structure which, surprisingly, was not annealed out after 4 hours at 200°C.

The variety of dislocation loops is amply demonstrated in Figure 4. In this photomicrograph we can see unfaulted loops (A), faulted loops (B), and doubly faulted loops (C) and (D). Note that there are many loops which are observed edge on because of the $\{110\}$ orientation of the foil.

TYPICAL DAMAGE ZONE IN Al AFTER IRRADIATION IN H.V. MICROSCOPE TO 0.25 dpa(Max.) at 100°C

FIGURE 1

EFFECT OF INCREASING FLUENCE ON
MICROSTRUCTURE OF Al AT 150°C.

a.) 0.25 dpa

b.) 1.6 dpa

c.) 4.1 dpa

d.) 8.1 dpa

FIGURE 2

EFFECT OF IRRADIATION TEMPERATURE ON THE
MICROSTRUCTURE OF Al AT 3dpa

a.) 100°C

b.) 150°C

c.) 175°C

d.) 200°C

FIGURE 3

VARIETY OF DISLOCATION LOOPS IN Al
IRRADIATED TO 8.1 dpa AT 150°C

FIGURE 4

b. Status of EM-7

Unanticipated technical problems (high voltage instability) developed during the past quarter which have delayed the acceptance of the EM-7 microscope until November 1972. This delay in schedule has prompted us to conduct some of the preliminary work at WADCO in collaboration with Dr. J. J. Laidler. Hopefully, the UW microscope will be available in the next quarter.

2. MeV Electron Studies

Several additional modifications to the 2 MeV electrostatic generator have been made during this quarter.

- Increase in the range of beam steering capability.
- Design and purchase (on UW funds) of a quick closing valve to protect the accelerator from vacuum accidents in the sample chamber.
- Higher field focusing magnet on the beam tube (see Figure 7 in Reference 4).
- Additional temperature control equipment for the sample chamber.

These modifications have allowed us to focus 100 microamperes of a 2 MeV beam of electrons onto a 5 mm diameter spot. The beam current on the sample through a 5 mm aperture is a function of accelerator voltage with a maximum near 1.9-2.0 MeV. It is felt that the presence of the 0.0025 cm thick titanium window is responsible for some beam scattering

and the higher beam energies reduce this effect. The above results were obtained with air in the sample chamber to increase the cooling of the Ti window. This must be done to keep the Ti from melting and breaking the vacuum in the column. The installation of the quick-closing valve on the accelerator should allow us to remove the Ti window, thus reducing the amount of beam scattering.

The presently attainable beam current of $\sim 3 \times 10^{15}$ electrons/cm²/sec corresponds to a damage level of $\sim 3 \times 10^{-7}$ dpa sec⁻¹ for 2 MeV electrons. This damage rate is roughly comparable to that in the core of a fast reactor ($\sim 10^{-6}$ dpa sec⁻¹).

A preliminary sample temperature versus heat input curve has been developed and is shown in Figure 5. The curve corresponds to the first sample chamber designed⁴ and it is only valid for a coolant flow rate of 2 liters per minute. The major point of the figure is that at a heat input of 200 watts (or ~ 1000 watts/cm²) we can control the sample temperature to 100°C or more by controlling the coolant flow rate. Modifications are under way to increase the flow rate as well as lowering the coolant temperature to allow irradiation temperature of less than 100°C.

A sample of high purity Al is being prepared for irradiation of 0.025 dpa (a factor of 10 below the first HVEM samples). The object here will be to cover the lower fluence range and eventually overlap the lower dpa range for the HVEM studies.

Figure 5

3. Aluminum Ion Bombardment

a. Ion Source Bench Tests

During the second quarter the testing of the heavy ion source on the low voltage test bench was concluded, and the installation of the source in the accelerator was started but not completely finished. The low voltage test bench contained a 12° mass spectrometer which allowed the determination of the ion source products. However, the resolution was marginal and therefore the precise determination several products await installation in the electrostatic generator.

During proper operation with CCl_4 and aluminum powder, the discharge required anode voltages below 50 volts. Similar tests with a nitrogen discharge required anode voltages on the order of 100 volts to be stable. Variations in the anode voltage for a given discharge produced only slow variations in beam intensity. Any discharge was extremely sensitive to the magnet current, as the magnetic field strongly interacts with the electric field of the extractor. The discharge was generally insensitive to filament current, provided it was maintained above a threshold of about 40 amperes.

Products of the discharge with CCl_4 and air at high voltage included H, He, C, N, O, Al and Cl (Figure 6). When the CCl_4 discharge was properly adjusted, it produced only two peaks, Al and Cl, since the anode voltage was too low to cause ionization of the other elements present in the source chamber (Figure 7).

TYPICAL SPECTRUM OF HEAVY ION SOURCE AT HIGH ANODE VOLTAGE

Figure 6

TYPICAL SPECTRUM OF HEAVY ION SOURCE AT LOW ANODE VOLTAGE

Figure 7

b. Ion Source Installation

The RF source and its power supply have been removed from the accelerator and the installation of the Physicon heavy ion source, lens assembly and power supplies, 2 kVA alternator, new focus and extractor supplies, new telemetry, and an additional control rod is presently under way.

The decision to dope the Al samples with helium (see Section B.3) caused us to move back our source installation date by two months and we now hope to have the source installed and operating by 1 November.

B. Related Information

1. Electron Damage Cross Section

It is very important to have a common basis for comparing electron and heavy ion induced damage in aluminum to that produced by neutrons. The current method of correlation relies on theoretical calculations of the number of times each atom is displaced during irradiation. The model does not take into account the annihilation of various point defects resulting from thermal migrations, spontaneous recombination due to interaction of defect strain fields, or that recombination present in the displacement spike. Despite the shortcomings of this approach it does provide us with a first order correlation and we will therefore use it in this study. In this section we calculate the displacement cross section due to electrons.

The cross section for energy transfers between energetic relativistic electrons is quite well known. Mott⁵ has expressed the scattering cross section for electrons with two conditional convergent infinite series. These series have been expanded by McKinley and Feshbach⁶ in power series of α and α/β where

$$\alpha = - \frac{Z\epsilon^2}{\hbar c} = \frac{Z}{137} \quad \text{for electrons}$$

and

$$\beta = \frac{v}{c}$$

where ϵ is the electronic charge,

Z is the nuclear charge of the struck atom,

v is the velocity of the electron,

c is the velocity of light, and

$\hbar$ is Planck's constant/ 2π .

These expansions give results accurate to 1% for nuclei up to $Z = 40$ and are certainly applicable to the case of aluminum ($Z = 13$). The McKinley-Feshbach (MF) differential scattering cross section for electrons of energy E_e can then be expressed as

$$d\sigma(E_e, T) = \frac{4\pi a_o^2 Z^2 E_R^2}{m_o^2 c^4} \left(\frac{1 - \beta^2}{\beta^4} \right) \left[1 - \beta^2 \frac{T}{T_m} + \pi\alpha\beta \left\{ \left(\frac{T}{T_m} \right)^{\frac{1}{2}} - \frac{T}{T_m} \right\} \right] \frac{T_m dT}{T^2}$$

(1)

Where a_0 is the Bohr radius ($0.53 \text{ \AA}$),
 E_R is the Rydberg energy (13.56 eV),
 m_0 is the rest mass of the electron,
 T is the energy of the struck nucleus, and
 T_m is the maximum energy that can be transferred to
the heavy atom and is equal to

$$T_M = \frac{2E_e(E_e + 2m_0c^2)}{m_2c^2} \quad (2)$$

where m_2 is the mass of the struck atom.

We wish to calculate the total displacement cross section for electrons over the range of interest here (i.e. 0-2 MeV). We must first define the model which we will use to calculate not only the total number of atoms displaced by the electrons, but also those displaced by the energetic primary knock-on atom (PKA). This will be done with the conventional Kinchin and Pease⁷ model which assumes that $V(T)$, the total number of atoms displaced when the struck nucleus is given energy T , is given by

$$\begin{aligned} V(T) &= 1 & T_d \leq T \leq 2T_d \\ V(T) &= \frac{T}{2T_d} & T > 2T_d \end{aligned}$$

where T_d is the displacement energy.

There is some confusion in the literature about the

value of T_d . Luccasson and Walker⁸ made the first measurement of 32 eV but later studies^{9,10} have indicated values of 16-19 eV. For the purposes of this work we will choose 16 eV and apply it to all the displacement calculations.

Hence our total displacement cross section for electrons of energy E_e is

$$\sigma_T(E_e) = \int_{T_d}^{2T_d} V(T) d\sigma(E_e, T) + \int_{2T_d}^{T_m} V(T) d\sigma(E_e, T) dt \quad (3)$$

We can make the following substitutions

$$A = \frac{4\pi a_o^2 Z^2 E_R^2}{m_o c^4} \left(\frac{\beta^2}{1 - \beta^4} \right) = A' \left(\frac{\beta^2}{1 - \beta^4} \right)$$

$$A' = 42.25 \text{ barns for Al}$$

$$x = \frac{T}{T_m}$$

$$dx = \frac{dT}{T_m}$$

when

$$T = T_d$$

$$x_1 = \frac{T_d}{T_m}$$

$$T = 2T_d$$

$$x_1 = \frac{2T_d}{T_m}$$

$$T = T_m$$

$$x = 1$$

Then Equation (3) becomes

$$\begin{aligned} \frac{\sigma_T}{A} = & \int_{x_1}^{x_2} \frac{1}{x^2} \left[1 - \beta^2 x + \pi \alpha \beta (x^{1/2} - x) \right] dx \\ & + \int_{x_2}^1 \frac{1}{x^2} \left(\frac{1}{x^2} \right) \left[1 - \beta^2 x + \pi \alpha \beta (x^{1/2} - x) \right] dx \end{aligned} \quad (4)$$

Integrating and rearranging Equation

$$\begin{aligned} \frac{\sigma_T}{A} = & \frac{T_m}{2T_d} \left[\ln \frac{T_m}{2T_d} + 1 \right] - \beta^2 \left[\ln 2 + \frac{T_m}{T_d} - 1 \right] + \pi \alpha \beta \left[\left(\frac{T_m}{T_d} \right)^{1/2} [2 - v_2] \right. \\ & \left. + \frac{T_m}{T_d} - \left(\frac{2T_m}{T_d} \right)^{1/2} - \ln 2 + 1 - \frac{T_m}{2T_d} \right] \end{aligned} \quad (5)$$

We can also solve for the primary displacement cross section by the following expression

$$\frac{\sigma_P}{A} = \left[\frac{T_m}{T_d} - 1 \right] - \beta^2 \ln \frac{T_m}{T_d} + \pi \alpha \beta \left\{ 2 \left[\left(\frac{T_m}{T_d} \right)^{1/2} - 1 \right] - \ln \frac{T_m}{T_d} \right\} \quad (6)$$

The mean energy of the displaced atom can be found by¹¹

$$\frac{\bar{T}_d}{T_m} = \frac{\ln \left(\frac{T_m}{T_d} \right) - \beta^2 \left(1 - \frac{T_d}{T_m} \right) + \pi \alpha \beta \left\{ 2 \left(1 - \sqrt{T_d/T_m} \right) - \left(1 - T_d/T_m \right) \right\}}{\left(\frac{T_m}{T_d} - 1 \right) - \beta^2 \ln \left(\frac{T_m}{T_d} \right) + \pi \alpha \beta \left\{ 2 \left(\sqrt{T_m/T_d} - 1 \right) - \ln \left(\frac{T_m}{T_d} \right) \right\}} \quad (7)$$

Calculated values of β , T_m , σ_p , σ_T and $\bar{T}_d$ for E_e from 100 keV to 2.2 MeV are given in Table II. The primary and total displacement cross sections are shown in Figure 8. It should be noted that σ_T/σ_p is 1.28 at 1 MeV and ~ 2 at 2 MeV so that neglect of the secondary atom production could underestimate the displacement rate (or total damage) by as much as 20-25% in the range of electrons of interest for this study.

Another point to note is that the total damage cross section calculated here agrees with that given by Oen¹² but not with that published by Makin.¹³ Appropriate corrections must therefore be made when comparing this work to that of other groups using Makin's cross section.

Figure 9 graphically displays how the maximum and average cross sections vary with incident electron energy. It is noted that the maximum PKA energy increases from 161 eV at 1 MeV to 482 eV at 2 MeV while the mean PKA energy only increases from 35 to 47 eV over the same energy range.

2. Range of Electrons in Aluminum

The range of electrons in Al has been very precisely measured and we wish to show in this section the exact numbers used for this study. We have chosen to use the data from Reference 14 and we have listed the ranges in Table III. The information is graphically presented in Figure 10. The density of Al has been assumed to be 2.7 gm cm^{-3} . It is interesting

Table II

Calculated Damage Parameters
for Electron Bombardment of Aluminum

Electron Energy E_e -MeV	$\beta = \frac{V}{C}$	T_m eV	$\bar{T}_d$ eV	σ_p barns	σ_T barns
0.2	0.695	19.5	17.6	11.7	11.7
0.3	0.776	31.6	21.4	27.7	27.7
0.4	0.827	45.4	24.1	34.2	35.1
0.5	0.862	60.7	26.4	38.1	40.7
0.6	0.887	77.6	28.4	40.9	45.5
0.75	0.914	106	31.0	43.8	51.7
0.9	0.932	138	33.4	46.0	57.2
1.0	0.941	161	34.9	47.1	60.5
1.1	0.948	186	36.3	48.1	63.6
1.25	0.957	227	38.3	49.3	58.1
1.40	0.963	270	40.2	50.2	72.1
1.5	0.967	302	41.4	50.8	74.7
1.6	0.970	335	42.6	51.3	77.2
1.75	0.974	387	44.3	51.9	80.7
1.8	0.975	405	44.8	52.0	81.8
2.0	0.979	482	46.9	52.7	86.1
2.1	0.981	523	47.9	52.9	88.1
2.3	0.983	609	49.8	53.4	92.0

Table III

Summary of Range-Energy Information for Aluminum

Electron Energy MeV	Mg/cm ²	Inches	cm
0.05	3.8	0.000556	0.00141
0.08	9.4	0.00136	0.00348
0.1	14	0.0026	0.00518
0.5	155	0.024	0.0574
1.0	401	0.06	0.148
1.5	760	0.096	0.242
2.0	950	0.14	0.352
2.5	1230	0.20	0.456
3.0	1440	0.24	0.550

Figure 8

Figure 9

Figure 10.

to note that the range of a 1 MeV electron is 1480 microns and a 2 MeV electron will penetrate to 3520 microns. Since our sample thickness is on the order of 75-100 microns, we can see that we will have a rather uniform defect generation rate.

3. Doping of Aluminum with Helium

A total of 49 high purity aluminum samples were injected with helium before the RF source was removed from the heavy ion accelerator. The doping levels were nominally 0.1, 1, and 10 atomic parts per million (appm) of helium.

The basic assumptions of the doping procedure are:

- The helium ions are essentially immobile after coming to rest in the aluminum.
- The straggling of the helium ions in aluminum at any given energy can be represented by a Gaussian distribution about the mean range.
- The half width, Γ_i , (full width at half maximum) of the Gaussian distribution, at any given energy is related to the mean range $\bar{R}_i$ by the expression

$$\Gamma_i = 0.04 \bar{R}_i$$

The last assumption is based on an estimate of the straggling parameter for helium ions in aluminum and on the relation between the half width of a Gaussian and the straggling parameter.

The mean range energy curve in Figure 10 was plotted using data from Northcliffe and Schilling.¹⁵ It shows that the maximum depth of penetration attainable with the 2 MV accelerator is about 7 microns. The minimum energy of the accelerator is about 450 kV and therefore a 2 micron layer from the surface cannot be doped. We will remove this denuded zone by vibropolishing, leaving a uniformly, helium doped layer approximately 5 microns thick.

The uniformity of the doping was maintained by changing the energy and the dose in small enough steps such that the Gaussian distributions of helium overlapped one another at their half maximum values. The general procedure is illustrated in Figure 12 for the first four energy steps of a 10 appm doping run. The dose needed at each energy step to attain the desired doping level was determined by assigning a volume to each energy step equal to the cross section of the beam times the half-width of the corresponding Gaussian. It took a total of 32 energy steps to uniformly dope a sample from a depth of 2 microns to 7 microns. Figure 13 shows a schematic of the integration over the 32 Gaussian distributions in a typical run.

These doped samples will be utilized in future heavy ion, HVEM and high energy electron bombardment studies.

Figure 11

SCHEMATIC OF CONCENTRATION PROFILES OF HELIUM INJECTED
INTO ALUMINUM AT VARIOUS ENERGIES

Figure 12

Figure 13

4. Chemical Analysis of Starting Materials

Foil specimens of high purity Al and 6 different Al alloys were received from ORNL¹⁶ this quarter. The alloys were prepared by J.C. Ogle, Metals and Ceramics Division and fabricated into foil form by R.E. Reed, Solid State Physics Division.

The pure aluminum was obtained from Cominco American, Inc., Spokane, Washington. The resistivity ratio was 13,600 and the material came from lot #RS 175 C-37. The chemical analysis is shown in Table IV.

Six alloys with the following nominal compositions were supplied along with the pure aluminum.

<u>Alloy</u>	<u>ORNL Designation</u>
Al + 3.8 wt% Cu	2B
Al + 0.025 wt% Cu	5A
Al + 3.8 wt% Cu + 0.5 wt% Si	6A
Al + 1 wt% Ni	11A
Al + 0.1 wt% Si	4A
Al + 0.5 wt% Si	4B
Pure Al	1A

The above alloys were obtained by melting and zone leveling under a vacuum of 10^{-6} torr or less. The vacuum system was made of quartz and heating was by an induction coil. The alloys were melted in graphite boats. The boats were made from high purity graphite by Ultra Carbon Corporation, Bay City, Michigan, lot - Carbon B-2574-1 (UF 4-S).

Table IV

Chemical Analysis of Aluminum Used for Present Study

<u>Element</u>	<u>Pure Aluminum</u>		<u>Al+3.8wt%Cu</u>	<u>Al+3.8wt%Cu+0.5wt%Si</u>
	<u>As Received</u>	<u>After Fabrication*</u>		
B	0.1	0.01	5	1
Ca		<0.1	1	0.5
Cr	<0.05	<0.05	0.1	0.2
Cu	<0.1	0.1	86200 (3.7wt%)	87400 (3.75wt%)
Fe	0.1	0.05	0.1	<0.1
K	<0.1	<0.1	0.1	<0.05
Mg	5	<0.03	1	1
Mn			0.1	0.1
Na	<0.03	<0.03	1	0.3
Ni			0.1	
P	<0.1	0.3	0.3	0.3
S	<1	<1	7	7
Si	<0.7	<0.7	<5	6500 (0.68wt%)
Ti			0.1	0.1
V			<0.1	<0.1
Zn	<0.2	<0.2	0.2	0.2
F	5	2		
Cl	5	5		
Br	<0.1	<0.1	<0.2	<0.5
O ₂	116	2	16	42
N ₂	6	2	2	2
H ₂	162	1175	108	108
C	39	18		

*ORNL Analysis

Each time the pure aluminum or alloys were handled, cut, or fabricated they were cleaned by etching for 10 minutes in the following solution at 60 to 80°C.

H_3PO_4	-	70%
H_2SO_4	-	25%
HNO_3	-	5%

After etching, materials were washed in distilled water and dried in lint-free nylon cloths.

A 10 minute etching time was used because X-ray fluorescence analysis shows that this amount of etching time assured that all contamination from cutting and handling was removed. Other etchants were tried but the one listed above apparently gave the brightest and cleanest surface. Sodium hydroxide etchants revealed a powdery film and a rougher surface that oxidized more readily.

The alloying materials were obtained from the following sources.

- Copper - Central Research Laboratories
American Smelting and Refining Company
South Plainfield, New Jersey
(99.999+%)
- Silicon - Dick Fox of the Instrument and Control Div.
ORNL
(99.9999+%)
- Nickel - INCO, #270
(99.99%)

The alloys were rolled from blocks about 1.25 cm on edge to foils 0.012 cm thick. The rolls were lubricated with a light coat of oil during rolling.

Chemical analyses of the alloys after preparation and fabrication are listed in Table IV. Samples 4A, 4B, 5A and 11A have not yet undergone final analysis.

5. Sample Labeling Procedure

A code has been developed to facilitate the identification of samples and the conditions to which they have been subjected. This code consists of four sections which are explained below.

A - 4 - 01 - 1			
			1,2,3 etc. Sample #
<u>Denotes Radiation Source</u>	<u>Alloy</u>	<u>Helium Content</u>	
A = Al ion	0 - high purity Al	00 = none	
E = electron accelerator	1 - Al+3.8 wt% Cu	X1 = 0.1 appm	
H = HVEM	2 - Al+0.025 wt% Cu	01 - 1 appm	
C = controls, non-irradiated or others	3 - Al+3.8 wt% Cu + 0.5 wt% Si	10 = 10 appm	
N = neutron	4 - Al+1 wt% Ni		
	5 - Al+0.1 wt% Si		
	6 - Al+0.5 wt% Si		
	7 - to be designated		
	⋮		

C. Future Work

During the next quarter we will continue with the analysis of the samples H-0-00-(1-4) and attempt to repeat that test with helium samples doped with 10 appm He. The aluminum ion source should be installed and we hope to have beam on target. Irradiations with the 2 MeV beam of electrons should begin if we can complete the temperature control equipment.

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