Body-Centered Cubic Ion Correlation Experiment

Compiled by
J. L. Brimhall

April 1977

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EXPERIMENT

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April 1977

BATTELLE
Pacific Northwest Laboratories
Richland, Washington 99352

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1.0 SUMMARY

This report includes the results of an interlaboratory correlation experiment involving eight laboratory sites studying the ion irradiation of molybdenum. The program was sponsored by the Materials and Radiation Effects Branch (M and RE) of the Division of Magnetic Fusion Energy (DMFE) of the Energy Research and Development Administration (ERDA). The program was titled Body-Centered Cubic Ion Correlation Program (BCC-ICE) and was comprised of three parts.

The first part compared the radiation damage produced in a reference specimen of molybdenum by ions of different masses and different energies. The results showed that the swelling rate and temperature dependence of swelling due to different heavy ions can be compared on a gross dpa (atomic displacements per atom) basis. The swelling rate due to protons was significantly higher than heavy ions and gross dpa is not a good basis for comparison. Other factors such as differences in dose rate and environmental vacuum may have some effect and these remain to be explored.

The second part of the program compared measurements on a common micrograph supplied to all participants in the program. The results from all the sites agreed very well. The value of standard deviation in the measurement of void volume fraction corresponded to 10% of the average measured value and the spread between highest and lowest reported value was 35%.

The third part of the program compared the microstructural characteristics of a transmission electron microscope (TEM) specimen that was passed from site to site ("round robin"). The results of this part of the experiment showed greater variations from the different participants than the results from the common micrograph. One of the principal sources of discrepancy was the measurement of the foil thickness which was necessary for computation of void number density. The standard deviation in the measurements of void volume fraction (swelling) corresponded to 20% of the measured average value. The spread between the highest and lowest value was 70%. This latter value gives an idea of the conceivable differences in results due to experimental techniques between two experiments.

2.0 INTRODUCTION

Ion bombardment is a rapid, simple, and inexpensive way to simulate damage in metals. A variety of ions, ranging from electrons to tantalum ions, are used.^(1,2) Although results are generally compared on a calculated dpa basis (dpa = atom displacements per atom), there is little experimental evidence to support this procedure. Correlation factors are needed to compare, for example, the damage due to nickel ions to that due to nitrogen ions.

There are correlation programs underway in the iron and nickel base alloy system, mostly in systems with fcc lattice structure. However, the refractory metals that show promise for controlled thermonuclear reactor (CTR) application have bcc lattice structure and respond differently to irradiation than do fcc metals.

DMFE/M and RE requested that an ion correlation program be instituted to explore these problems. Selected participants in the Body-Centered Cubic Ion Correlation Experiment (BCC-ICE) met in October 1974 to plan an experimental program. The following sites and personnel participated in this.

Argonne National Laboratory	A. Taylor
Atomics International	D. W. Keefer
Mass. Inst. of Technology	A. L. Bement
Naval Research Laboratory	F. A. Smidt
Oak Ridge National Laboratory	F. W. Wiffen/J. Bently
Pacific Northwest Laboratory	J. L. Brimhall/E. R. Bradley
University of Cincinnati	J. Moteff
University of Wisconsin	G. L. Kulcinski

The experimental program was comprised of three parts.

- 1) Interlaboratory comparison of the change in microstructure produced in identical reference material by different ions of different energies.
- 2) Interlaboratory comparison of measurements on a common micrograph.
- 3) Interlaboratory comparison of measurements on a single transmission electron microscope (TEM) specimen transferred from site to site.

This report summarized the results and conclusions of these experiments.

3.0 INTERLABORATORY COMPARATIVE EXPERIMENT USING DIFFERENT IONS

The correlation experiment as initially proposed is outlined below, followed by a discussion of the results. Because of experimental difficulties, not all conditions could be met by all participants. These differences will be discussed in the course of the report.

3.1 THE PROPOSED CORRELATION EXPERIMENT

The material proposed for the correlation experiment was pure molybdenum, 0.001 to 0.010 in. thick. The specimens were to be cut at Pacific Northwest Laboratory (PNL) to the size specified by the participant and given identical vacuum annealing. Some materials would be reserved for chemical analysis after the annealing. There was to be no helium preinjection for standard materials. All surface preparation was to be done by the participants.

The desired conditions of temperature and dose are shown in Table 1.

TABLE 1. Experimental Test Matrix

<u>Temperature (C°)</u>	<u>Dose (dpa)</u>	
800	20	2
900	20	2
1000	20	2

All participants would bombard the material to the standard condition of 20 dpa at 900°C. The other dose-temperature conditions should be tested as resources permit. The dose should be evaluated at the region of subsequent observation in the microscope.

The dose rate was to be $\sim 2 \times 10^{-3}$ dpa/sec, within a factor of two, for the standard conditions (900°C, 20 dpa). This dose rate would apply to the region of observation. Dose rate for protons would be the maximum feasible.

Displacement damage curves for heavy ions were to be calculated using the EDEP-1 code of Manning and Mueller and the recommended procedures of HEDL-TME-73-76 for dpa calculation.⁽³⁾ This assumes a β factor of 0.8 and

an effective displacement energy of 62 eV for molybdenum. If requested, PNL would obtain the curves for the various ions to be used. The displacement dose for protons would be determined by procedures in HEDL-TME-73-76.

The observed region should correspond to a section in which the concentration of deposited ions at 20 dpa is no greater than 0.02%, averaged over a 1000 Å section. The 2-dpa samples should represent the same distance from the surface as the 20-dpa samples; i.e., variation in dpa should be made only by time of irradiation.

The different laboratories would do as many samples as they desire. At least one sample that has been bombarded under the standard condition (900°C, 20 dpa) would be interchanged with one other laboratory. PNL would run samples for possible high-voltage electron microscopy (HVEM) analysis. Other laboratories could run samples for this analysis, as their resources permitted. If feasible, dummy samples would be put in a bombardment furnace for subsequent chemical analysis.

A time history of the temperature, vacuum and ion dose would be recorded for all irradiations. Gas analysis of the vacuum would be done if possible. All information relating to microstructural analysis would be recorded, including, but not limited to, void size, shape, density, dislocation density, void lattice and precipitate structure. Microstructure of unirradiated material would be analyzed by TEM after surface treatment but prior to bombardment. Analysis would be done in the region corresponding to same depth as the ion bombardment region.

In addition to these experimental conditions which were agreed upon, the recommended practice for Neutron Radiation Damage Simulation by Charged Particle Irradiation prepared by ASTM Subcommittee E10.88 would be consulted and followed as close as practicable.

3.2 EXPERIMENTAL RESULTS - PART 1

The following sites completed Part 1 of the experiments using the ions and energies listed below:

<u>Sites</u>	<u>Ions</u>	<u>Energies (MeV)</u>
Pacific Northwest Laboratory	Ni ²⁺	5
Atomics International	H ⁺	0.5
University of Wisconsin	Cu ⁴⁺	17-18.5
Naval Research Laboratory	Mo ⁺	2.7

These values of ion masses and energies provide a wide range of ion irradiation conditions. The specified dose rate could not be obtained in some facilities, but the above sites did attain the specified dose of 20 dpa at 900°C. The vacuum in the ion irradiation chamber varied considerably among the experiments. The details of the irradiation conditions for this part of the experiment are given in Appendix A.

The vendors analysis of the original molybdenum ingot is given in Table 2. Prior to distribution to the sites, the molybdenum was annealed 2 hr at 1600 °C in a vacuum of 1×10^{-7} torr. An analysis of the interstitial elements after this anneal is shown in Table 3. The microstructure of the molybdenum is typical of a fully annealed state. An optical micrograph is shown in Figure 1. The grain size is approximately 60 μm . A transmission electron micrograph is shown in Figure 2. Some large precipitates are found widely dispersed and mostly at grain boundaries. The dislocation density was very low.

The microstructural parameters reported for the different irradiations are tabulated according to irradiation temperature and presented in Tables 4-7. The original intent of the experiment was to hold all experimental conditions constant other than ion type and energy. Because of limitation of facilities, the dose rate was also not constant for all experiments. In addition, the vacuum during irradiation varied from 2×10^{-6} to 1×10^{-8} torr.

TABLE 2. Vendors Chemical Analysis of Molybdenum Ingot

<u>Element</u>	<u>Wt.%</u>
C	0.005
O	0.0005
N	0.002
H	<0.0001
Fe	0.004
Ni	<0.001
Si	0.001

TABLE 3. Chemical Analysis After Vacuum Anneal

<u>Element</u>	<u>Wt.%</u>
D	<0.001
O	0.001
N	0.001
H	0.0003

Since void volume fraction or swelling is of prime technological interest, the swelling vs. dose at 900°C is plotted in Figure 3. Although there are only two points in most cases, the data show trends for the different ion species. PNL data for 5 MeV Ni²⁺ at 1000 and 800°C are not plotted, but the swelling rate is similar to that found at 900°C for 5 MeV Ni²⁺ ions.

The temperature dependence of the void swelling at 2 and 20 dpa is shown in Figure 4. Again there is only limited data but there is a clear separation between the low and high dose. The data also indicate that the temperatures which were used were below the maximum swelling temperature.

The data on the void size and density generally fall into two groups. The Mo⁺ and Ni²⁺ ion data agree reasonably well and the H⁺ and Cu⁴⁺ are somewhat similar. The void size is smaller and void density larger in the Mo²⁺ and Ni²⁺ ion experiments than in the H⁺ or Cu⁴⁺ ion experiments. The lower dose rate in the latter case may account for much of the differences. The

FIGURE 1. Micrograph Showing Grain Structure of Molybdenum After 1600°C Anneal (130 X)

FIGURE 2. Transmission Electron Micrograph of Molybdenum After 1600°C Anneal (32,000 X)

TABLE 4. Microstructural Data for 700°C

Site	Ion Mass and Energy	Dose (dpa)	Dose Rate (dpa/sec)	Void Dia (nm)	Void Density (n/m ³)	Void Vol. Fraction (%)	Void Lattice (nm)	Loop Dia (nm)	Loop Density (n/m ³)	Total Line Density (n/m ²)
UWi	18meV Cu ⁴⁺	1.0	4 x 10 ⁻⁴	4.8	1 x 10 ²²	0.08	(a)	7.6	2 x 10 ²¹	9 x 10 ¹³
(a) Not observed.										

TABLE 5. Microstructural Data for 800°C

Site	Ion Mass and Energy	Dose (dpa)	Dose Rate (dpa/sec)	Void Dia (nm)	Void Density (n/m ³)	Void Vol. Fraction (%)	Void Lattice (nm)	Loop Dia (nm)	Loop Density (n/m ³)	Total Line Density (n/m ²)
PNL	5meV Ni ²⁺	20	3 x 10 ⁻³	4.4	5 x 10 ²²	0.65	(a)	10.0	1.4 x 10 ²²	6.4 x 10 ¹⁴
PNL	5meV Ni ²⁺	2	1 x 10 ⁻²	≤2.0	low	(b)	(a)	15.0	6.7 x 10 ²⁰	8.7 x 10 ¹³
PNL	5meV Ni ²⁺	20	1 x 10 ⁻²	≤2.0	low	(b)	(a)	30.0	3-1 x 10 ²¹	1-2 x 10 ¹⁴
NRL	2.7meV Mo ⁺	2	2.5 x 10 ⁻³	3.6	6.1 x 10 ²²	0.2	(a)	10.5	5.6 x 10 ²¹	5.6 x 10 ¹⁴
NRL	2.7meV Mo ⁺	20	2.5 x 10 ⁻³	5.6	8.1 x 10 ²²	1.01	31.0	(a)	(a)	4.1 x 10 ¹⁴
UWi	17meV Cu ⁴⁺	2.2	4-8 x 10 ⁻⁴	4.4	1.7 x 10 ²²	0.11	34.0	16.0	2 x 10 ¹⁹	9 x 10 ¹²
AI	0.5meV H ⁺	8	2 x 10 ⁻⁴	3.6	4.8 x 10 ²³	1.4	--	--	--	--
AI	0.5meV H ⁺	12	2 x 10 ⁻⁴	3.5	5.9 x 10 ²³	1.4	--	--	--	--

(a) Not observed
(b) Not determined

TABLE 6. Microstructural Data for 900°C

Site	Ion Mass and Energy	Dose (dpa)	Dose Rate (dpa/sec)	Void Dia (nm)	Void Density (n/m ³)	Void Vol. Fraction (%)	Void Lattice (nm)	Loop Dia (nm)	Loop Density (n/m ³)	Total Line Density (n/m ³)
PNL	5meV Ni ²⁺	2	3 x 10 ⁻³	3.4	9.3 x 10 ²²	0.47	(a)	34.0	4.3 x 10 ¹⁹	3.8 x 10 ¹³
PNL	5meV Ni ²⁺	20	3 x 10 ⁻³	5.4	8 x 10 ²²	2.0	35.0	35.0	1.8 x 10 ¹⁹	5.6 x 10 ¹³
PNL	5meV Ni ²⁺	2	1 x 10 ⁻²	3.7	4 x 10 ²²	0.3	(a)	16.5	3.8 x 10 ²⁰	8.7 x 10 ¹³
PNL	5meV Ni ²⁺	20	1 x 10 ⁻²	6.4	5 x 10 ²²	1.85	34.0	27.0	2.6 x 10 ¹⁹	6 x 10 ¹³
NRL	3.2meV Mo ⁺	2	2.5 x 10 ⁻³	4.0	8.2 x 10 ²²	0.34	(a)	5.9	1.8 x 10 ²²	6.8 x 10 ¹⁴
NRL	3.2meV Mo ⁺	20	2.5 x 10 ⁻³	8.0	5.8 x 10 ²²	2.07	35.0	(a)	(a)	5.4 x 10 ¹⁴
UWi	18.7meV Cu ⁴⁺	1.0	5 x 10 ⁻⁴	7.8	6 x 10 ²¹	0.17	(a)	(a)	(a)	(b)
UWi	17meV Cu ⁴⁺	1.9	4 x 10 ⁻⁴	9.8	6.6 x 10 ²¹	0.39	(a)	(a)	(a)	5 x 10 ¹³
UWi	17meV Cu ⁴⁺	19.0	5 x 10 ⁻⁴	7.5	3 x 10 ²²	0.87	34.0	42.0	7 x 10 ¹⁸	1 x 10 ¹³
AI	0.5meV H ⁺	3	2 x 10 ⁻⁴	14.0	6 x 10 ²¹	1.1	(a)			
AI	0.5meV H ⁺	6	2 x 10 ⁻⁴	14.0	1.2 x 10 ²²	2.9	(a)			
AI	0.5meV H ⁺	16	2 x 10 ⁻⁴	17.0	1.14 x 10 ²²	5.15	(a)			

(a) Not observed
(b) Not determined

TABLE 7. Microstructural Data for 1000°C

Site	Ion Mass and Energy	Dose (dpa)	Dose Rate (dpa/sec)	Void Dia (nm)	Void Density (n/m ³)	Void Vol. Fraction (%)	Void Lattice (nm)	Loop Dia (nm)	Loop Density (n/m ³)	Total Line Density (n/m ³)
PNL	5meV Ni ²⁺	21	3 x 10 ⁻³	5.3	2.1 x 10 ²²	0.45	38.0	(a)	(a)	1.7 x 10 ¹³
PNL	5meV Ni ²⁺	2	1 x 10 ⁻²	3.8	9 x 10 ²²	0.48-0.96	40.0	23.0	1-5 x 10 ¹⁹	2.6 x 10 ¹³
PNL	5meV Ni ²⁺	20	1 x 10 ⁻²	6.4	7.5 x 10 ²²	3.0	40.0	(a)	(a)	5 x 10 ¹³
UWi	18.7meV Cu ⁴⁺	1.3	5 x 10 ⁻⁴	25.2	5.8 x 10 ²⁰	0.76	(a)	(a)	(a)	1 x 10 ¹³

(a) Not observed

FIGURE 3. Swelling Rate in Molybdenum Bombarded by Different Ions at 900°C

FIGURE 4. Temperature Dependence of Swelling in Molybdenum Bombarded by Different Ions

void nucleation rate is proportional to the displacement rate so a higher dose rate should produce a greater void density if other conditions are equal.^(4,5) However, the data from dose rates higher than the standard condition do not show large differences.

Since changes in void size and density tend to compensate, a comparison of the void volume fraction is more meaningful. At the low doses, the absolute values of swelling are within a factor of two for the heavy ions and somewhat higher for protons. At 20 dpa, the protons give much greater swelling than do the heavy ions. This is reflected in the much higher swelling rate from protons than the heavy ions. The swelling rate from Ni^{2+} and Mo^{+} ions agree even for Ni^{2+} data using a higher dose rate. The swelling rate for Cu^{4+} ions is somewhat lower for no obvious reason. The dose rate in the Cu^{4+} experiment rate in the proton experiment is also low, but yet a high swelling rate is obtained.

Differences in void size and, hence, swelling can also be attributed to differences in interpretation of void shape. The assumed void shape ranged from pure cubes to spheres. Although this will result in differences in absolute values of void swelling, the swelling rate should not be affected.

It seems reasonable to conclude that dpa can be used as a basis for comparing swelling results obtained from different heavy ion bombardments. This does not hold true for protons and a correction factor should be applied. This conclusion agrees with work in iron-nickel alloys in which the swelling rate from protons is much higher than that from either heavy ions or neutrons. The difference is probably due to the different PKA spectra produced by proton bombardment compared to heavy ion bombardment.

The data are rather sparse in the plot of swelling versus temperature. However the agreement is reasonable among the different heavy ion experiments. The peak in the swelling occurs above 1000°C, if we assume the curve does not have a very sharp peak. At 20 dpa, there is only a slight difference between the high and low dose rates.

At 2 dpa, there is no consistent difference between the Mo^{+} and Cu^{4+} data, even though the Cu^{4+} data represent a lower dose rate. The difference

in dose rate in these experiments may not be sufficient to affect differences in the void swelling outside of the experimental scatter band.

A significantly reduced swelling value is found at 1000°C for the high-dose (20 dpa), low-dose rate specimen in the Ni^{++} ion experiments. This is believed to be related to the concurrent formation of precipitates in this specimen. The precipitates are further believed to be a result of the deposited nickel ions. The presence of a precipitate is also reported in the Cu^{4+} experiments which may explain their low swelling value at 19 dpa (see Appendix A). These results do point up the problems from implantation of the bombarding ion. The effects of the implanted ion is more severe with higher temperatures and high doses.

The above results further suggest that examination should be from a region as far from the maximum damage zone as possible, particularly when a foreign ion is used. In most cases, this will be a compromise between free surface proximity and the proximity of the maximum damage zone (deposited ion zone). In these experiments, the maximum damage zone was examined only in the Mo^{+} irradiations. Since this is a self-ion, little effect was expected. However, at high temperatures and low dose rates, diffusion can increase the zone of influence of the deposited ion beyond the peak damage zone, as was evident in some of the Ni^{++} irradiations.

One additional factor which may affect the results but has not been fully explored is the level of vacuum in the various experiments. The Cu^{4+} experiment had a much higher vacuum than the others. This higher vacuum may explain some of the differences observed in that data, but any further discussion would be speculation at this point.

4.0 INTERLABORATORY COMPARISON OF A COMMON MICROGRAPH

One micrograph which depicted typical void microstructures in good contrast was selected for measurements of void parameters (Figure 5). The Naval Research Laboratory copied this micrograph and distributed it to all sites participating in the program. Each site measured the void size, determined the void number density (foil thickness was given), and the void volume fraction. The details of the measurement techniques and complete data acquired as supplied by the individual sites are given in Appendix B. The important void parameters from this comparative experiment are given in Table 8.

Measurements of the average void size agreed. The standard deviation was only $2.2\text{\AA}$ (0.22 nm), which is as good as can be expected based on the degree of judgment in determining the boundaries of a void. The spread in the measurement of void density is somewhat surprising in that this measurement only involves counting voids. The variations reflect differences in interpretation as to what is a void and differences in the accounting for voids that intersect surfaces and edges. The greatest variation is in the void volume fraction. The standard deviation was 0.21 which corresponds to 10% of the average value of 1.99. If one looked at the differences between the maximum and minimum values, then the difference is ~35%. Therefore, it is conceivable that two experimenters under the most optimum conditions could reach considerably different values, but still have similar results.

FIGURE 5. "Common Micrograph" Measured by All Sites Participating in Part 2 of BCC-ICE

TABLE 8. Void Parameters from Measurements On a Common Micrograph

Site	Void Dia. (nm)	Number Meas.	Void Density (n/m ³)	Void Vol. Fraction (%)
AI	7.9	1840	5.96×10^{22}	1.96
ANL	8.2	1703	6.4×10^{22}	2.2
MIT	7.8	1280	5.1×10^{22}	1.63
NRL	8.0	1664	5.85×10^{22}	2.07
ORNL	7.8	700	6.04×10^{22}	2.04
PNL	8.2	600	5.91×10^{22}	2.2
U. Cinc.	7.7	1850	5.57×10^{22}	1.68
U. Wisc.	8.4	--	5.86×10^{22}	2.2
Average	8.0	--	5.78×10^{22}	1.99
Standard Deviation	0.22	--	0.3×10^{22}	0.212

5.0 INTERLABORATORY COMPARISON OF MEASUREMENTS ON A "ROUND ROBIN" TEM FOIL

In addition to variations due to measurements on a micrograph, site-to-site variation due to microscopy techniques would be expected. To evaluate these variations, the same TEM specimen, supplied by NRL, was sent to all the participating sites. Each site performed the microscopy and measurements of the void parameters. Experimental details are given in Appendix C. This one specimen suffered very little deterioration from all of the analyses at the sites and being mailed around the country. The results of the examination of the "round robin" specimen are given in Table 9.

TABLE 9. Microstructural Parameters from Measurements of "Round Robin" Foil

<u>Site</u>	<u>Void Dia. (nm)</u>	<u>Number Meas.</u>	<u>Void Density (n/m³)</u>	<u>Void Vol. Fraction (%)</u>	<u>Void Lattice (nm)</u>
AI	8.2	2123	4.55×10^{22}	1.74	--
ANL	6.4	--	12.0×10^{22}	2.1	--
MIT	7.44	875	10.0×10^{22}	2.96	--
NRL	8.0	--	5.8×10^{22}	2.07	35.0
ORNL	8.74	687	6.04×10^{22}	2.85	37.5
PNL	8.4	1167	6.35×10^{22}	2.62	36.5
U. Cinc.	8.6	2110	4.4×10^{22}	1.75	--
U. Wisc.	8.5	--	6.4×10^{22}	2.6	35.5
Average	8.04	--	6.94×10^{22}	2.34	36.1
Standard Deviation	0.77	--	2.5×10^{22}	0.43	--

The agreement among the sites is not as good as that found on the "common micrograph", as would be expected. The standard deviation in the void size measurements is $7\text{\AA}$ (0.7 nm). For the void density, the standard deviation is 2.5×10^{22} which corresponds to more than 30% of the average measured value. Both these measurements reflect differences in magnification, and the density measurement has the additional problem of determining foil thickness. In the computation of void volume fraction, errors in the magnification cancel out as they appear both in the numerator and denominator. The standard deviation of the measurements of void volume fraction is about twice that found for measurements on the common micrograph. This must be due to differences in microscopy conditions, particularly to variations in the measurement of foil thickness.

The spread in the results in this part of the experiment represents the best agreement attainable among different experimenters since all sites were looking at exactly the same specimen. The spread between the maximum and minimum value is approximately 70%. Therefore, it is conceivable but unlikely that differences of this magnitude are insignificant. There is a high probability, however, that differences in the void volume fraction of less than 35% between two experimenters are not meaningful.

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APPENDIX A

EXPERIMENTAL DETAILS OF PART 1 OF
BODY-CENTERED CUBIC ION CORRELATION PROGRAM

APPENDIX A

EXPERIMENTAL DETAILS OF PART 1 OF BODY-CENTERED CUBIC ION CORRELATION PROGRAM

This appendix contains detailed descriptions of the experiments performed in Part 1 of the BCC-ICE program. The material is presented as supplied by the participants. There has been no attempt to organize it into a uniform format, although some editing has been done for purposes of condensation.

A.1 ATOMICS INTERNATIONAL BCC ION CORRELATION EXPERIMENT DATA SHEET FOR EXPERIMENTAL PARAMETERS

I. Material Chemistry

- A. Chemical analysis of base material (see body of report)
- B. Chemical analysis after pre-irrad. treatment (see body of report)
- C. Chemical analysis after ion bombardment

C.1 - Analysis of entire specimen

Chemical analysis has not been done. A sample irradiated at 900°C and a sample held at 900°C in the irradiation target chamber are available for analysis.

C.2 - Analysis of damage zone

We do not feel this is required in the case of proton irradiation. Ion microprobe mass analysis of proton-irradiated Type 316 stainless steel and tantalum has shown the hydrogen content to be uniform throughout the sample, including the damage zone.

II. Pretreatment

- A. Electropolishing (solution, temperature, surface removal, etc.)
12% H₂SO₄ in methyl alcohol. -25 to -40°C. 2.5 x 10⁻³ cm.
- B. Other preparation (mechanical polish, chemical etch, etc.)
None
- C. Surface smoothness
Mirror

III. Irradiation Parameters

A. Specimen Environment

Spec. Ident.	Temp (°C)	Temp Variation ±	Time (pre-irrad)	Time (irrad)	Time (post-irrad)	Temp (post-irrad)	Vac.	Vacuum (low to high value)
M3	900	5	23 min	17 hr, 34 min	At 700°C within 1 min, at room temp in 8 min.			1.8×10^{-6} to 2.8×10^{-6}
M4	900	5	18 min	8 hr, 38 min	At 700°C within 1 min, at room temp in 8 min.			2.0×10^{-6} to 2.8×10^{-6}
M6	900	<5	19 min	4 hr, 30 min	At 700°C within 0.5 min, at room temp in 7 min.			3.2×10^{-6} to 5.0×10^{-6}
M10 + M11*	900	5**	7 min	10 hr, 53 min	At 700°C within 1.4 min, at room temp in ~7 min.			$\leq 3.0 \times 10^{-6}$
M12 + M13	950	50***	3 min	4 hr, 52 min				1.0×10^{-6} to 6.0×10^{-6}
M14	800	<5	18 min	16 hr, 2 min	At 600°C within 0.5 min, at room temp in 6 min.			4.8×10^{-6} to 8.0×10^{-6}
M15	800	<5	12 min	10 hr, 43 min	At 600°C within 0.5 min, at room temp in ~6 min.			$\leq 3.0 \times 10^{-6}$

Gas Analysis of Vacuum: None

B. Beam Variables

Spec. Ident.	Highest Current	Lowest Current	Average Current	Integrated Fluence	Beam Uniformity(?)
M3	1.78×10^{15} n/cm ² -s	1.59×10^{15} n/cm ² -s	1.71×10^{15} n/cm ² -s	1.09×10^{20} n/cm ²	$\leq \pm 10\%$ over 1.3 cm ² sample area.
M4	1.76×10^{15}	1.59×10^{15}	1.71×10^{15}	5.46×10^{19}	
M6	1.88×10^{15}	1.63×10^{15}	1.77×10^{15}	2.73×10^{19}	
M10 + M11	1.88×10^{15}	1.63×10^{15}	1.72×10^{15}	6.75×10^{19}	
M12 + M13	Typical beam fluctuations.		1.65×10^{15}	3.38×10^{19}	
M14	Typical beam fluctuations.		1.77×10^{15}	1.01×10^{20}	
M15	1.80×10^{15}	1.69×10^{15}	1.75×10^{15}	6.75×10^{19}	

*Sample M11 was shielded from the beam but kept at the irradiation temperature.

**Operator error caused severe temperature overshoot. Specimen spent ~40 s over 900°C and reached a maximum temperature of 1000°C momentarily.

***Heater and temperature sensing problems resulted in an estimated decrease in sample temperature of about 50°C before irradiation was terminated.

IV. Post-Irradiation Examination

A. Data or parameters common to all specimens

Magnification accuracy: Within 6%

Tilt stage accuracy: $\pm 0.5^\circ$

Average foil thickness + estimated error: Typical foil thickness is 1×10^{-6} cm. Estimated error is $\pm 25\%$.

Diffraction contrast conditions:

voids: absorption

dislocations: Imaging of dislocations was done as mandated by those areas of the sample which were transparent to 100 KeV electrons.

Focus conditions:

voids: overfocused

dislocations: sharp

Void dimension measured: Void diameters defined by the central portion of the Fresnel fringe.

B. Data Specific to Individual Specimens

Specimen Identification	Surface Removal($\pm$)	Calculated OPA($\pm$)	Mean Void Size and Nidi/NT	Total Number of Voids Sized	Number of Areas Sampled	Void Geometry	Void Density	Range of Density Values	Total Number of Voids Counted	Areas Sampled	Void Volume Fraction	Estimated Error*
M3	1.6×10^{-3} $\pm 2 \times 10^{-4}$ mm	16 ± 2 $- 1$	170A	1107	4	(100) with (110) Trunc.	$1.1 \times 10^{16}/\text{cm}^3$	0.96×10^{16} to 1.3×10^{16}	1107	4	4.9%	3.5-6.2%
M4	9.0×10^{-4} $\pm 2 \times 10^{-4}$	6.0 ± 0.4	140	622	4	(100) with (110) Trunc.	1.2×10^{16}	1.1×10^{16} to 1.3×10^{16}	622	4	2.8%	2.1-4.1%
M6	1.0×10^{-3} $\pm 2 \times 10^{-4}$	3.1 ± 0.2	140	749	3	(100) with (110) Trunc.	5.9×10^{15}	4.2×10^{15} to 8.6×10^{15}	749	3	1.1%	0.92-1.3%

*Numbers Quoted are Observed Variations from Area to Area

A.2 NAVAL RESEARCH LABORATORY

A beam of 2.7 MeV Mo ions was obtained from the NRL 5.5 MV Van de Graff accelerator using the modified Hill-Nelson source developed by NRL.⁽¹⁾ Suitable beam currents were obtained using a sputtering electrode of naturally occurring molybdenum. A magnetically analyzed beam of $^{98}\text{Mo}^+$ with a beam current of ~ 100 nA was defocused across the area to be irradiated (0.109 cm^2) to assure uniformity of flux across the sample. The beam current on the sample was 20 nA or $0.185\text{ }\mu\text{A/cm}^2$ and fluctuated less than $\pm 25\%$ during an irradiation.

Damage produced by this ion beam was calculated with the E-DEP-1 Code of Manning and Mueller⁽²⁾ assuming E_d of 63 eV, an efficiency of 0.8, and a density of 6.4×10^{22} atoms/cm³. Peak damage of 20 dpa was calculated for 157.5 μC total integrated charge. Maximum damage occurs at a depth of 0.3 μm and varied less than 20% from 0.13 μm to 0.40 μm . Dose rate was 2.5×10^{-3} dpa/sec.

The irradiation chamber previously employed was modified for these experiments to incorporate a specimen furnace of lower thermal inertia and higher temperature capability and the addition of a titanium sublimation pump. Vacuum at the sample at temperature during these runs was approximately 1×10^{-6} Torr. A single specimen was irradiated during each run and the temperature of the sample was lowered immediately upon completion of the bombardment to minimize possible annealing effects. A total of five specimens were irradiated as follows: 2 specimens at 900°C to 20 dpa; 1 specimen at 900°C to 2 dpa, 1 specimen at 800°C to 20 dpa; and 1 specimen at 800°C to 2 dpa.

The specimens were prepared for microscopy observation following ion bombardment in two stages. Removal of material from the front face was accomplished by pulsed electropolishing using a laser interferometer⁽³⁾ to monitor removal of 0.23 μ of material. The sample was then back-thinned to transparency with a jet electropolisher using a solution of 12.5% H_2SO_4 in methanol. The specimens were examined in a JEM-200A electron microscope equipped with a side entry goniometer tilting stage, with the microscope operated at 200 kV. Voids were imaged under weakly diffracting absorption contrast conditions using the out-of-focus phase contrast technique of Rühle. Foil thickness was determined from stereomeasurements on pairs of photographs taken at tilt angles of 10 to 14 degrees. Accuracy of the thickness measurements is estimated to be $\pm 20\%$.

Voids were found in all the four samples examined and were homogeneously distributed. Voids in the 20 dpa samples appear to be truncated octahedra while those in 2 dpa samples appear to be nearly spherical. A void lattice was observed in the 20 dpa samples at both 800 and 900°C but not in the 2 dpa samples. A summary of the observations is provided in Table A.2-1. Each point represents the average of two determinations.

TABLE A.2-1. Void Microstructures in Ion-Bombarded Molybdenum

<u>Temp.</u> <u>(°C)</u>	<u>dpa</u>	<u>$N_V(\text{cm}^{-3})$</u>	<u>$\bar{D}^\circ$ (Å)</u>	<u>$\Delta V/V(\%)$ +</u>	<u>Void Lattice Spacing (Å)</u>
800	2	6.1×10^{16}	36	0.20	--
800	20	8.1×10^{16}	56	1.01	310
900	2	8.2×10^{16}	40	0.34	--
900	20	5.8×10^{16}	80	2.07	350

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A.3 UNIVERSITY OF WISCONSIN

I. Experimental Procedure

A. Irradiation Procedure

The material supplied to UW was in strip form 7 cm x 1.25 cm x 0.15 mm thick. To accomodate this material into the UW sample holder (Figure A.3-1), two 3.2 mm-diameter and five 4.8 mm-diameter holes were spark machined in the sample strips. The strips were also spark machined in half (making two 3.5 cm x 1.25 cm x 0.15 mm thick strips). This was done to facilitate the subsequent preparation of specimens for TEM examination. After the spark machining process but prior to irradiation, the specimen strips were electro-polished in a 15% H_2SO_4 , 85% methyl alcohol solution at -20°C and 10 volt to give a smooth surface. Table A.3-1 summarizes the pretreatment of the specimens.

TABLE A.3-1. Pretreatment of BCC Ion Correlation
Experiment Molybdenum Specimens

- A. Electropolishing (solution, temperature, surface removal, etc.). Polished in 15% H_2SO_4 , 85% methyl alcohol at -20°C and 10 V to smooth surface after strip preparation in B.
- B. Other preparation (mechanical polish, chemical etch, etc.). Spark machined seven holes in strips, then spark machined strips in half followed by polish in A.
- C. Surface smoothness.
Generally very smooth, with some preferential grain attack.

The specimens were irradiated using the UW tandem Van de Graaff accelerator as described elsewhere.⁽¹⁾ Briefly, the specimens were bombarded with ~ 25 particle nA of either 17.0 or 18.8 MeV Cu^{4+} ions. The charge state of the Cu beam was determined by elastic scattering of the beam off of a thin gold foil.⁽¹⁾ The energy E of the beam is given by $E = (q + 1)V$, where q is the charge state of the beam and V is the accelerator dome voltage (measured with a generating voltmeter).

FIGURE A.3-1. Detail of the Sample Folder Assembly

The samples were irradiated in three separate groups. The first group contained six specimens of which two were irradiated at 1000°C, two at 900°C, and two at 800°C to damage levels near 1 dpa (approximately 1 μ m from the surface). These irradiations were carried out at pressures near 1×10^{-8} Torr. The specimens were irradiated from highest to lowest temperature to minimize post-irradiation annealing effects. The second group also contained six specimens of which one was irradiated at 1000°C, two at 900°C and two at 800°C to damage level near 2 dpa. The sixth specimen in this group was irradiated at 700°C to ~ 1 dpa. The pressure during irradiation of these specimens was near 5×10^{-9} Torr. The last group contained only one specimen. It was irradiated at 900°C to 19 dpa at a pressure near 1×10^{-8} Torr. Partial pressure analysis prior to the first 1000°C run revealed that the residual gas in the sample chamber was composed of 57% H₂, 18% CO, 22% H₂O, 2% CO₂, and 1% CH₄.

The particle flux for all but the 19 dpa specimens was determined by periodically measuring the beam current striking an insertable Faraday cup. Knowledge of the charge state of the beam allows the beam particle current to be calculated. The total fluence was then calculated from knowledge of the irradiation time (about 2 hr for the 2 dpa specimens). The current striking the 19 dpa specimens was measured directly and integrated to obtain the fluence. The displacement damage versus depth curve as well as the position of the deposited copper ions is shown in Figure A.3-2. The dpa values were calculated with the E-DEP-1 Code⁽²⁾ assuming $E_d = 61.7$ ev, an efficiency of 0.8, and an atom density of $6.42 \times 10^{22}/\text{cm}^3$. The dpa rate at the region of analysis ($\sim 1 \mu\text{m}$ from the sample surface, see below) is $\sim 4 \times 10^{-4}$ dpa/sec.

To minimize the effects of the sample surface and the deposited Cu ions, TEM analysis of the damage was done $\sim 1 \mu\text{m}$ from the sample surface (Figure A.3-2). This analysis zone is $>1 \mu\text{m}$ from the deposited Cu atoms. A diffusion calculation shows that for the 2 dpa specimens the deposited Cu atoms move $<0.2 \mu\text{m}$ and for the 19 dpa specimens $<0.6 \mu\text{m}$ from their initial position during the course of the irradiation.

FIGURE A.3-2. Damage Curve for 17 MeV Cu on Molybdenum Calculated Using the E-DEP-1 Code of Ref. (2). Also indicated on the figure are the TEM analysis zone and the location of the deposited copper ions.

Therefore, impurity effects due to the use of a copper beam may not be a problem.* The ability to do the TEM analysis away from both the surface and the stopped ions is one of the advantages of using 17-19 MeV as opposed to 2-5 MeV heavy ion beams. Table A.3-2 summarizes the irradiation parameters for each of the six irradiated samples which were successfully analyzed on the electron microscope.

After irradiation, the same electropolishing conditions used in the pre-irradiation polish were used to remove $\sim 1 \mu\text{m}$ from the irradiated surface. The amount of surface removed was determined to within $\pm 0.1 \mu\text{m}$ by optical interference microscopy. The 3mm-diameter TEM specimens were then cut from the sample strip and back thinned to perforation in a Fischione jet electropolisher with the same polishing solution but at -50°C and 70 V.

B. Transmission Electron Microscopy Procedure

The specimens were examined in a Jeolco 100B electron microscope operated at 120 kV voltage. Voids were imaged under weak diffraction conditions at high magnification and focused as precisely as possible to minimize the surrounding dark fringes for void size measurement. The magnification of the electron microscope was determined to be accurate to within $\pm 5\%$. Dislocations and loops were imaged under a two beam condition in typical areas to get the average densities. Photographs of interesting grain boundary areas or any unusual features were also taken.

The foil thickness was determined by the stereo pair technique. The average tilt angle was about 9 degrees from the zero degree horizontal position. The accuracy of the tilt angle is about ± 10 minutes. Foil thicknesses ranged from $\sim 500 \text{ \AA}$ near the edge of the foil to a maximum of $2400 \text{ \AA}$. Photographs of stereo pairs were not taken for the 800°C , 2.2 dpa and 700°C , 1.0 dpa specimens because the images of the small voids and the background contrast were too variable when the specimens were tilted. The thicknesses of these two foils were estimated by the degree of transparency using the observed thickness range from $500 \text{ \AA}$ to $\sim 2500 \text{ \AA}$ in other specimens.

* A small amount of precipitation was observed in the 19 dpa specimen as discussed in Section 2.

TABLE A.3-2. Irradiation Parameters.

A. Specimen Environment

Spec. Ident.	Temp (°C)	Temp. Variation (°C)	Time (pre-irrad)	Time (irrad)	Time at Temp. (post-irrad)	Vac. (Torr)	Vacuum (Torr) (low to high value)
2	1000	+3	Annealed by PNL at 1600°C for 2 hours near 10 ⁻⁶ Torr.	47 min (Beam on 39, off 8)	140 min at 900°C 160 min at 800°C	2.5 x 10 ⁻⁸	2.2 x 10 ⁻⁸ to 2.8 x 10 ⁻⁸
3	900	+4		112 min (Beam on 30, off 82)	55 min at 900°C 160 min at 800°C	9.2 x 10 ⁻⁹	8.1 x 10 ⁻⁹ to 1.0 x 10 ⁻⁸
9	900	+2		85 min (Beam on 84, off 1)	325 min at 800°C 50 min at 700°C	4.9 x 10 ⁻⁹	4.6 x 10 ⁻⁹ to 5.2 x 10 ⁻⁹
10	800	+5		195 min (Beam on 113, off 82)	120 min at 800°C 50 min at 700°C	4.3 x 10 ⁻⁹	3.8 x 10 ⁻⁹ to 4.7 x 10 ⁻⁹
12	700	+10		40 min (Beam on 29, off 11)	Cooled immediately after irradiation	4.4 x 10 ⁻⁹	3.8 x 10 ⁻⁹ to 4.8 x 10 ⁻⁹
13	900	+10	↓	1691 min (Beam on 1118, off 573)	145 min at 625°C 80 min at 575°C 65 min at 525°C	1.4 x 10 ⁻⁸	6.0 x 10 ⁻⁹ to 7.0 x 10 ⁻⁸

Gas Analysis of Vacuum 57% H₂, 18% CO, 22% H₂O, 2% CO₂, 1% CH₄ at 1000°C.B. Beam Variables (17-19 MeV Cu⁴⁺ ions)

Spec. Ident.	Highest Current (particle nA)	Lowest Current (particle nA)	Average Current (particle nA)	Integrated Fluence (particle nA-hr) (ions)		Beam Uniformity (%)
2	32	19	31	18	4.1x10 ¹⁴	+20
3	40	23	32	16	3.6x10 ¹⁴	↓
9	27	12	21	30	6.7x10 ¹⁴	
10	23	8	17	32	7.1x10 ¹⁴	
12	38	9	29	14	3.2x10 ¹⁴	
13	18	6	13	234	5.3x10 ¹⁵	↓

The accuracy of the foil thickness obtained from the stereo pairs is $\pm 15\%$ while the uncertainty of the thickness obtained from the transparency measurements is estimated to be $\pm 50\%$.

C. Electron Micrograph Analysis Procedure

The average void diameters and void size distributions were measured with a Zeiss particle size analyzer. For the 1000°C, 1.3 dpa specimen, edges of the cubic voids in the (100) foil orientation were measured. For specimens irradiated at 900, 800 and 700°C, the diameters were measured assuming spherical void morphology and using the center of the dark fringe surrounding the void. Because the edge of the voids on the surface were polished by the solution, voids intersecting the surface were not counted. A corrected foil thickness of $t - \bar{d}$ was used to determine the void density, where $\bar{d}$ is the average void diameter.

II. Results

We examined six samples irradiated in the temperature range 700-1000°C as discussed in Section 2-A. Voids were found in each of these specimens and in general were homogeneously distributed. Dislocation loops were observed only in the specimens irradiated to ~2 dpa at 700°C and 800°C and in the specimen irradiated to 19 dpa at 900°C.

The voids in the specimen irradiated at 1000°C were cubes of {110} faces with {110} facets. The voids became more spherical as the irradiation temperature decreased. The average void size varied from ~45 Å at lower temperature to ~250 Å at 1000°C. The volume swelling value at 1000°C included a correction for the observed truncations. Swelling values at all other temperatures were calculated by $\Delta V/V = \frac{\pi}{6} \rho_v \langle d^3 \rangle_{\text{avg}}$.

A summary of the damage microstructure observed in the examined specimen is contained in Table A.3-3. The uncertainties given in Table A.3-3 are arrived at in the following manner: surface removal, the uncertainty in the interference microscopy determination is $\pm 0.1 \mu\text{m}$; dpa value, $\pm 21\%$ due to $\pm 20\%$ beam profile nonuniformity⁽¹⁾ and $\pm \sim 5\%$ due to the uncertainty in the surface thickness removal (added in quadrature); mean void size $\pm 5\%$ due to the uncertainty in the measurement of the microscope magnification;

TABLE A.3-3. Damage Microstructure in the Irradiated Specimens

Specimen Number	Irradiation Temp. (°C)	Calculated dpa (%) ± Error	Surface Rem. (μm) Error (μm)	Mean Void Size (Å)	Total Number of Voids Sized	Number of Areas Sampled	Void Density (cm ⁻³)	Areas Sampled	Void Volume Fraction (%)
2	1000	1.3 ± 0.3	0.9 ± 0.1	252	354	2	5.8 × 10 ¹⁴	1	0.76
3	900	1.0 ± 0.2	0.9 ± 0.1	78	393	2	6.0 × 10 ¹⁵	1	0.17
9	900	1.9 ± 0.4	0.9 ± 0.1	98	230	1	6.6 × 10 ¹⁵	1	0.39
10	800	2.2 ± 0.5	1.0 ± 0.1	44	340	1	1.7 × 10 ¹⁶	1	0.11
12	700	1.0 ± 0.2	1.1 ± 0.1	48	259	1	1.0 × 10 ¹⁶	1	0.08
13	900	19 ± 4	1.2 ± 0.1	75	1305	2	3.0 × 10 ¹⁶	1	0.87

A-12

Specimen Number	Estimated Error (± %)	Mean Loop Diameter (Å)	Standard Deviation (Å)	Total No. of Loops Sized	Number of Areas Sampled	Loop Density (cm ⁻³)	Line Density (cm ⁻²)	Total Dislocation Density ^(a)
2	0.15	---	--	--	No Loops Detected	--	1 × 10 ⁹	1 × 10 ⁹
3	0.03	---	--	--	No Loops Detected	--	ND	ND
9	0.08	---	--	--	No Loops Detected	--	5 × 10 ⁹	5 × 10 ⁹
10	0.06	160	65	6	2	2 × 10 ¹³	8 × 10 ⁸	9 × 10 ⁸
12	0.04	76	27	72	1	2 × 10 ¹⁵	4 × 10 ⁹	9 × 10 ⁹
13	0.17	420	60	4	2	7 × 10 ¹²	1 × 10 ⁹	1 × 10 ⁹

ND - Not Determined

(a) - Loop + Line

void density $\pm 25\%$ due to $\pm 15\%$ uncertainty in foil thickness and $\pm 5\%$ in the microscope calibration (added twice) except for specimens 10 and 12 where this error is $\pm 60\%$ due $\pm 50\%$ uncertainty in foil thickness plus $\pm 5\%$ in the microscope calibration; and void volume fraction, $\pm 20\%$ due to the $\pm 15\%$ uncertainty in the foil thickness and $\pm 5\%$ in the microscopic calibration (added only once due to cancelation in the $\rho_v \langle d^3 \rangle_{\text{avg}}$ product) except for specimens 10 and 12 where this error is $\pm 55\%$ due to $\pm 50\%$ uncertainty in the foil thickness and $\pm 5\%$ in the microscope calibration. Figure A.3-3 contains histograms of the void size distributions.

Randomly distributed precipitates were observed in the 900°C , 19 dpa specimen. These precipitates could affect the void microstructure in this specimen. No precipitates were observed in any other specimen. Some void depleted zones were observed at grain boundaries with widths varying from 300 to $700 \text{ \AA}$ at 900°C . The change in the void size is reduced for narrower depleted zones. This can be explained by differences in the effectiveness of the grain boundaries as defect sinks. The beginning of a void superlattice was observed in the 800°C , 2.2 dpa specimen. A void lattice with better alignment was found in the 900°C , 19 dpa specimen though the alignment was still not perfect. The void lattice spacing is about $340 \text{ \AA}$ in both specimens.

Loop and network dislocation densities were also determined when the foil thickness was determined from stereo pair analysis. The values obtained are given in Table A.3-3. The loop density decreased from $2 \times 10^{15} \text{ cm}^{-3}$ at 700°C to $2 \times 10^{13} \text{ cm}^{-3}$ at 800°C . At 900°C , loops were observed only in the 19 dpa high dose specimen. No loops were found at 1000°C and 1.3 dpa.

FIGURE A.3-3. Void Size Distributions in Five of the Six Irradiated UW Molybdenum Specimens

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A.4 PACIFIC NORTHWEST LABORATORY BCC ION CORRELATION
EXPERIMENT DATA SHEET FOR EXPERIMENTAL PARAMETERS

- I. Material Chemistry - See Body of Report
- II. Pretreatment
 - A. Electropolishing (solution, temperature, surface removal, etc.)
12% H₂SO₄ in Methyl Alcohol, -55°C, 20 volts for 2 minutes
 - B. Other preparation (mechanical polish, chemical etch, etc.)
Ground flat and mechanically polished prior to electropolish
- III. Post-Irradiation Examination
 - A. Data or parameters common to all specimens
 - Magnification accuracy
+5%
 - Tilt stage accuracy
+5%
 - Average foil thickness + estimated error
 - voids:
500 Å +20% @ 93% confidence level
 - dislocation:
2000 Å +20% @ 93% confidence level
 - Diffraction contrast conditions
 - voids:
kinematic $S \gg \sigma$
 - dislocations:
near dynamical condition (s positive)
 - Focus conditions
 - voids:
under focus
 - dislocations:
at focus
 - Void dimension measured
<100> cube edge

B. Data specific to individual specimens

Specimen Identification	Surface Removal (°)	Calculated DPA (±)	Mean Void Size (λ_{VD}/n_I)	Standard Deviation	Total Number of Voids Sized	Number of Areas Sampled	Void Density	Range of Density Values	Total Number of Voids Counted
A-64	4600Å ± 300Å	18 ± 1	53Å	23Å	1157	3	8.9×10^{16}	$8.7 - 9.2(\times 10^{16})$	1192
A-65	5500Å ± 500Å	21 ± 2	55Å	25Å	519	2	6.6×10^{16}	$6.0 - 7.2(\times 10^{16})$	1894
A-67	4100Å ± 600Å	17 ± 2	43Å	14Å	1334	3	7.4×10^{16}	$7.2 - 7.5(\times 10^{16})$	1459
A-68	5400Å ± 400Å	2.0 ± .1	34Å	10Å	707	3	9.3×10^{16}	$8.9 - 9.6(\times 10^{16})$	1579
A-69	5700Å ± 800Å	21 ± 3	63Å	20Å	1081	3	5.4×10^{16}	$4.1 - 6.5(\times 10^{16})$	4403
A-70	4400Å ± 400Å	17 ± 1	66Å	23Å	511	2	4.5×10^{16}	$1.6 - 7.4(\times 10^{16})$	1738
A-71	4600Å ± 400Å	1.8 ± .1	35Å	10Å	1416	4	4.3×10^{16}	$3.1 - 4.9(\times 10^{16})$	3224
A-72	5700Å ± 600Å	2.2 ± .2	39Å	16Å	460	2	3.9×10^{16}	$3.4 - 6.8(\times 10^{16})$	2242
A-73	5600Å ± 100Å	21 ± .5	53Å	20Å	775	3	2.1×10^{16}	$1.7 - 2.5(\times 10^{16})$	1360
A-74	5800Å ± 300Å	22 ± 1	65Å	75Å	1090	3	7.6×10^{16}	$6.7 - 9.2(\times 10^{16})$	2550
A-75	4500Å ± 200Å	18 ± .5	63Å	20Å	843	3	7.2×10^{16}	$6.9 - 7.5(\times 10^{16})$	1283
A-76	5000Å ± 100Å	1.9 ± .05	36Å	9Å	1121	3	8.7×10^{16}	$5.5 - 11(\times 10^{16})$	2057
A-77	5000Å ± 200Å	1.9 ± .05	40Å	12Å	734	3	1.2×10^{17}		287
A-78	5000Å ± 500Å	1.9 ± 1.5	44Å	17Å	434	2	5.1×10^{16}	$4.4 - 5.8(\times 10^{16})$	811
A-79	4400Å ± 800Å	17 ± 2							
A-80	5200Å ± 500Å	20 ± 2							
A-81	4600Å ± 100Å	1.8 ± .05							
A-82	5100Å ± 400Å	1.9 ± .1							

Low Density and Small Size Precluded Measurement

Specimen Identification	Areas Sampled	Void Volume Fraction	Mean Loop Diameter (Å)	Standard Deviation (Å)	Total Number of Loops Sized	Number of Areas Sampled	Loop Density	Line Density	Total Dislocation Density (Loop & Line)	Dose Rate
A-64	2	.0222	350	170	96	3	1.8×10^{13}	5.4×10^9	5.6×10^9	2.8×10^{-3}
A-65	3	.0180						4.4×10^9		3.3×10^{-3}
A-67	2	.0098								6.2×10^{-3}
A-68	2	.0047	345	220	243	3	4.3×10^{13}	3.4×10^9	3.9×10^9	3.2×10^{-3}
A-69	7	.0190	270	225	88	3	2.6×10^{13}	5.8×10^9	6.0×10^9	1.5×10^{-3}
A-70	4	.0180	180	120	129	1				1.2×10^{-3}
A-71	3	.0025	165	70	327	2	3.8×10^{14}	2.7×10^9	4.7×10^9	1.1×10^{-2}
A-72	4	.0034								1.3×10^{-2}
A-73	4	.0045						1.7×10^9		3.3×10^{-2}
A-74	3	.0320						4.7×10^9		1.5×10^{-2}
A-75	2	.0250						5.3×10^9		1.2×10^{-2}
A-76	3	.0048	350	245	90	4	6.0×10^{12}	2.3×10^9	2.4×10^9	1.2×10^{-2}
A-77	1	.0096	232	120	230	3	4.8×10^{13}	2.5×10^9	2.8×10^9	9.8×10^{-2}
A-78	2	.0065	100	25	330	2	1.4×10^{16}	2.0×10^9	6.4×10^9	3.0×10^{-3}
A-79			145	85	549	4	2.8×10^{14}	8.9×10^9	1.0×10^{10}	1.2×10^{-2}
A-80			120	60	304	2	1.2×10^{15}	2.1×10^{10}	2.5×10^{10}	1.4×10^{-2}
A-81			150	75	641	4	6.7×10^{14}	5.6×10^9	6.6×10^9	1.2×10^{-3}
A-82										1.3×10^{-3}

IV. Irradiation Parameters

A. Specimen Environment

Spec. Ident.	Temp °C	Temp Variation ± °C	Time (pre-irrad) Hours	Time (irrad) Hours	Time (post-irrad) Hours	Temp. (post-irrad) °C	Vac. $\times 10^{-6}$
A-64	900	5	1.9	2.0	8.0	900	1.5
A-65	900	5	3.9	1.9	6.1	900	1.4
A-67	900	5	7.7	.7	3.5	900	1.2
A-68	900	5	3.7	.2	3.0	900	1.2
A-69	900	5	10.9	.4	.5	900	1.0
A-70	900	5	11.3	.4	.1	900	1.0
A-71	900	5	11.3	.05	.05	900	1.0
A-72	900	5	11.8	.05	0		1.0
A-73	1000	5	0	1.8	1.0	1000	2.3
					3.3	300	
A-74	1000	5	1.8	.4	.5	1000	1.5
					3.5	300	
A-75	1000	5	2.1	.5	.2	1000	1.3
					3.5	300	
A-76	1000	5	2.6	.05	.2	1000	1.6
					3.5	300	
A-77	1000	5	2.7	.05	3.5	300	1.5
A-78	1000	5	2.7				
	300	5	1.3	2.1	0		1.0
A-79	1000	5	2.8				
	800	5	0	.5	2.7	800	.9
A-80	1000	5	2.8				
	800	5	.5	.5	2.2	800	.9
A-81	1000	5	2.8				
	800	5	.9	.05	2.2	800	.9
A-82	1000	5	2.8				
	300	5	1.0	.05	2.1	800	.8

B. Beam Variables

Spec. Ident.	Highest Current(ma)	Lowest Current(ma)	Average Current(ma)	Integrated Fluence ₂ ions/cm ²	Beam Uniformity(?)
A-64	.038	.020	.028	3.3×10^{16}	Unknown
A-65	.035	.020	.028	3.3×10^{16}	
A-67	.040	.028	.030	1.5×10^{16}	
A-68	.028	.028	.028	3.2×10^{15}	
A-69	.120	.120	.120	3.3×10^{16}	
A-70	.120	.120	.120	3.3×10^{16}	
A-71	.120	.120	.120	3.3×10^{15}	
A-72	.120	.120	.120	3.3×10^{15}	
A-73	.035	.028	.028	3.2×10^{16}	
A-74	.120	.130	.122	3.3×10^{16}	
A-75	.120	.120	.120	3.2×10^{16}	
A-76	.120	.142	.131	3.6×10^{15}	
A-77	.120	.085	.111	3.5×10^{15}	
A-78	.033	.005	.053	3.1×10^{16}	
A-79	.120	.120	.120	3.3×10^{16}	
A-80	.120	.120	.120	3.3×10^{16}	
A-81	.120	.120	.120	3.3×10^{15}	
A-82	.120	.120	.120	3.3×10^{15}	

APPENDIX B

DATA ACQUISITION FROM PART 2
ANALYSIS OF A COMMON MICROGRAPH IN THE
BODY-CENTERED CUBIC ION CORRELATION PROGRAM

APPENDIX B

DATA ACQUISITION FROM PART 2 ANALYSIS OF A COMMON MICROGRAPH IN THE BODY-CENTERED CUBIC ION CORRELATION PROGRAM

This appendix contains detailed description of the data acquisition from Part 2 - Analysis of a Common Micrograph in the BCC-ICE program. Some editing of the material has been done but the context remains basically as supplied by the participants.

B.1 ATOMICS INTERNATIONAL ANALYSIS OF PHOTOMICROGRAPHS AT ATOMICS INTERNATIONAL

A Zeiss Particle-Size Analyzer is used to count and size voids. The perimeter of each void is assumed to lie in the center of the encircling overfocus fringe. In those instances where the voids are irregularly shaped, the area of the light spot on the analyzer is adjusted until the operator feels the area matches that of the void image on the photomicrograph. Whenever possible, the analyzer is operated in the "reduced" linear mode so that the intervals on the analyzer represent the minimum uncertainty in size.*

The void populations in the analyzer intervals are fed into a computer program. This program confirms the number of voids in each interval and supplies the total number of voids counted, the void diameter represented by the center of each size interval, the fraction of voids falling within the interval, the void density, the average void diameter, the foil volume, the total void volume, the void volume fraction ($\Delta V/V$) and the swelling ($\Delta V/V - \Delta V = \Delta V/V_0$). When more than one photomicrograph is being analyzed, the program supplies the above information for each photograph and for the entire sample represented by all photographs.

*In counting the NRL photograph this mode was used, and each analyzer interval spanned about 4.6 Å.

The computer program is based on the following relationships. The void density, ρ_v , is given by

$$\rho_v = \frac{n_t M^2}{L_1 L_2 t} (\text{cm}^{-3}) \quad (1)$$

where n_t is the total number of voids counted, M is the magnification of the photomicrograph, L_1 and L_2 are the dimensions in cm of the photomicrograph area analyzed, and t is the foil thickness in cm. On the analyzer, the center of the i^{th} interval, $\bar{x}_i$, is given in mm. The diameter of a void falling within this interval is given by

$$\bar{d}_i (\text{cm}) = 3.33 \times 10^{-2} \bar{x}_i / M \quad (2)$$

if the analyzer is operated in the reduced mode. The average diameter for all voids in the photomicrograph, $\bar{d}$, is then

$$\bar{d} = \sum_i \frac{n_i}{n_t} \bar{d}_i = \frac{3.33 \times 10^{-2}}{M n_t} \sum_i \bar{x}_i n_i \quad (3)$$

where n_i is the number of voids in the i^{th} interval. The average volume, $\bar{V}_i$, of a void falling within the i^{th} interval is

$$\bar{V}_i (\text{cm}^3) = \frac{\pi}{6} (\bar{d}_i)^3 = 1.94 \times 10^{-5} \bar{x}_i^3 / M^3. \quad (4)$$

$\bar{V}_i$ is summed for all intervals to obtain ΔV . The void volume fraction and swelling are then obtained from the known values of ΔV and V , the foil volume represented by the photomicrograph. Relationships identical to (2), (3), and (4) except for numerical constants are used when the analyzer has been operated in the "standard" mode.

Void Parameters Measured on Common Micrograph:

Number of voids counted	1842
Average diameter	79 Å
Standard Deviation	22 Å
Void Density	5.96×10^{16} voids/cm ³
$\Delta V/V_0$	2.00%

Void size distribution is shown in Figure B.1-1.

FIGURE B.1-1. Histogram of Void Size Distribution
Measured on the "Common Micrograph"

B.2 ARGONNE NATIONAL LABORATORY

The following data on measurements of the common micrograph was supplied by ANL:

No. of voids counted	1703
Average diameter	82.1 Å
Standard deviation	27.5 Å
Void density	6.14×10^{16} voids/cm ³
Void Volume Fraction	2.2%

B.3 MASSACHUSETTS INSTITUTE OF TECHNOLOGY

Procedures:

A. Void counting

The void size distribution was measured with a Zeiss TGZ3 Particle Size Analyzer using a portion of the micrograph (19.98 x 19.08 cm.). Every void that was visible was counted; using a reduced, exponential scale. The true diameter of the void was taken as equivalent to the outer portion of the dark contrast ring surrounding each image. Edge effects were accounted for by counting only the voids whose centers lie within the field of view. Surface effects and overlap were ignored for the present.

B. Data Reduction and Analysis

The reduction of raw data was straightforward. The counts from each size class were multiplied by the appropriate exponential mode correction factors given in the Zeiss manual and a histogram and cumulative distribution curve were generated in the conventional manner. A correction accounting for the voids whose centers lie outside of the plane of the foil was used:

$$N_i^C = N_i^O / (1 + IC_i/t)$$

where N_i^C = corrected number of voids for each size class,

N_i^O = observed number of voids for each size class,

IC_i = Interval Center for each size class,

and t = foil thickness

As a check for normality, the cumulative curve was plotted on normal probability paper. Descriptive parameters for the distribution curve--mean, standard deviation, skewness and kurtosis--were calculated by the standard formulas. The volume fraction was found from the following relation:

$$V_v = \frac{\Sigma V}{lwt/M^2}$$

Where ΣV = Summation of individual void volumes; $l \& w$ = enlarged dimensions of examined region, t = actual thickness, M = magnification.

Discussion:

A problem exists in determining a size distribution--the observers tendency to place more weight on the larger objects than on the smaller. Using linear class intervals will result in higher accuracy for the larger objects than the smaller. When the scale is changed to exponential intervals, there is constant measuring accuracy for all sizes. As is pointed out by Rühle,⁽¹⁾ the true diameter of the void may correspond to different contrast features depending upon the foil thickness, void size, foil orientation and focussing conditions. For this micrograph, the true diameter was taken as the dark ring surrounding each image, with the voids assumed spherical.

A difficulty in determining volume fraction arises with edge and surface effects. The edges of the field of view cut many of the voids--requiring a convention for taking this into account. The convention used here was to count only those images whose centers lie within the boundaries. The voids which are physically cut by the surface lead to the same problem. The previous convention cannot be adopted because of the impossibility of determining which voids have centers outside of the foil thickness. Since, on the average, an equal number of voids have centers outside of the foil as within, their effects on the volume fraction may be ignored. There will be a broadening of the distribution curve due to this effect (especially at the low end). It should also be recognized that the surface voids will be somewhat enlarged by the etching process, but this may be essentially ignored, since the surface voids (in this sample) occupy approximately .13% of the volume.

Inspection of the micrograph showed that loss of voids through overlap could be essentially ignored--although the methods of Hilliard⁽²⁾ to account for overlap are being applied to the data. Due to the small loss through overlap, the relation for volume fraction was extremely simple.

The void parameters are shown below and the void size distribution in Figure B.3-1.

Number of Voids	=	1280.04
Average Diameter	=	77.60 Å
Average Area	=	0.52E+04 Å ²
Average Volume	=	0.32E+06 Å ³
Thickness	=	1050.0 Å
Volume Fraction	=	0.016335
Voids/cc	=	0.51E+17
Variance	=	578.18 Å ²
Standard Deviation	=	24.05 Å
Skewness	=	0.57
Kurtosis	=	3.30

FIGURE B.3-1. Histogram of Void Size Distribution and Cumulative Distribution Measured on the "Common Micrograph"

REFERENCES

1. M. R. Rühle, Radiation Induced Voids in Metals, AEC Symposium Series 26, 1971.
2. J. E. Hilliard, "The Counting and Sizing of Particles in Transmission Microscopy", Trans. AIME, 224: 906, 1962.

B.4 NAVAL RESEARCH LABORATORY

Data on Measurements of the Common Micrograph.

Foil Thickness	1049 Å
Mean Diameter	80.23 Å
Number Density	5.85×10^{16} voids/cm ³
Void Swelling	2.07%
Number of Voids Counted	1664

The void size distribution is shown in Figure B.4-1.

FIGURE B.4-1. Histogram of Void Size Distribution Measured on the "Common Micrograph"

B.5 OAK RIDGE NATIONAL LABORATORY

Void Sizing of Common Micrograph

Information supplied:

Foil Thickness = 1050 Å

Magnification = 400,000×

From the contrast exhibited by the voids the micrograph was underfocussed slightly and the true size of the voids is given by the diameter measured to the inside of the dark Fresnel-like fringe. (Normally with such small voids better delineation is obtained in the slightly overfocussed condition, say, ~1 μm.)

Void size distributions were obtained using a calibrated Zeiss particle size analyzer (for three areas and a total of 713 voids). The data were processed to yield the following parameters:

(1) Mean void size $\sum_i n_i d_i / \sum_i n_i = 78.4 \text{ Å}$

(First moment of size distribution.)

Standard deviation = 2.0 Å^*

(2) Volume averaged mean void size $(\sum_i n_i d_i^3 / \sum_i n_i)^{1/3} = 86.5 \text{ Å}$

(Third moment of size distribution)

Standard deviation = 3.2 Å^*

(3) Void density = $6.04 \times 10^{16} \text{ cm}^{-3}$

Standard deviation = $0.15 \times 10^{16} \text{ cm}^{-3}^*$

(4) Void volume fraction = 2.04%

Standard deviation = 0.20%^{*}

* Standard deviation of the measurements.

NOTE: Some difficulty was experienced in fitting the size of the aperture on the particle size analyzer to the inside of the dark Fresnel-like fringe and a comparison with measurements using a 10× eyepiece and graticule indicates that the measured sizes are probably approximately 2 or 3% larger ($\Delta V/V \sim 6$ to 9% too large).

B.6 PACIFIC NORTHWEST LABORATORY VOID SIZE AND VOID DENSITY
DETERMINATION OF THE BCC-ICE "STANDARD" PHOTOMICROGRAPH

The standard photomicrograph provided by NRL has been analyzed for void size and void density. The void size was measured by three persons using two techniques, namely a calibrated 7X measuring eyepiece and a Ziess particle-size analyzer. Approximately 600 voids were measured from representative areas of the micrograph with the same areas being measured by each person. The void density was determined by counting the voids in two 100 cm² areas of the micrograph. The results from two independent measurements show the void density to be $5.91 \pm .06 \times 10^{16} \text{ cm}^{-3}$.

The void size data and the calculated swelling values are given in Table B.5-1. The combined data show mean void diameters of 82 Å and 87 Å, respectively, for the measurements made with the Ziess particle-size analyzer and the measuring eyepiece. The mean void diameters obtained by the three individuals show maximum differences of 12 Å when the particle size analyzer was used and 6 Å with the measuring eyepiece. In all cases, the void perimeter was assumed to coincide with the inside of the dark underfocus ring and thus the variations observed reflect the different interpretations of this dimension.

TABLE B.5-1. Void Data from the BCC-ICE
"Standard" Photomicrograph

	<u>Measuring Eyepiece</u>			<u>Particle-Size Analyzer</u>		
	<u>Void Size</u>	<u>Standard Deviation</u>	<u>Swelling</u>	<u>Void Size</u>	<u>Standard Deviation</u>	<u>Swelling</u>
	<u>$\bar{d}$ (Å)</u>	<u>S (Å)</u>	<u>$\frac{\Delta V}{V_0}$ (%)</u>	<u>$\bar{d}$ (Å)</u>	<u>S (Å)</u>	<u>$\frac{\Delta V}{V_0}$ (%)</u>
Person A	83	25	2.4	79	21	2.0
Person B	89	24	2.8	89	25	2.9
Person C	89	25	2.9	77	23	1.9
Combined Data	87	25	2.7	82	23	2.2

Void Density = $5.91 \pm .06 \times 10^{16} \text{ cm}^{-3}$

B.7 UNIVERSITY OF CINCINNATI

I. Experimental Procedure

The void sizes and numbers in one of the two identical micrographs supplied by NRL were measured and counted using a TGZ 3 Zeiss Particle Size Analyzer operated at a linear-distribution-reduced mode. All visible voids with images lying wholly within the field of area (19.05 x 24.80 cm) and those with images intersected by the edges of the micrograph but with their centers lying within the boundaries were measured and counted. The actual void size was taken as the inner diameter of the dark contrast ring for small voids (approximately up to 80 Å), and as the outer diameter of the dark contrast ring for larger voids. For nonspherical voids the equivalent-area diameters were determined.

The true number of voids for each size class was calculated as follows:

$$n_i^t = \frac{n_i}{1 + \bar{d}_i/t} \quad (1)$$

where n_i^t = true number of voids for i-th size class

n_i = observed number of voids for i-th size class

$\bar{d}_i$ = interval center of i-th size class

t = thickness of the specimen

The following equations were used to determine void parameters:

$$N_v = \frac{\sum n_i^t M^2}{LWt} \quad (2)$$

$$\bar{d}_v = \frac{\sum n_i^t \bar{d}_i}{\sum n_i^t} \quad (3)$$

$$\bar{D}_v = \left(\frac{\sum n_i^t (\bar{d}_i)^3}{\sum n_i^t} \right)^{1/3} \quad (4)$$

$$\frac{\Delta V}{V} = \frac{\pi}{6} (\bar{D}_v)^3 \cdot N_v \quad (5)$$

$$S = \left\{ \frac{\sum n_i^t (\bar{d}_i - \bar{d}_v)^2}{\sum n_i^t} \right\}^{1/2} \quad (6)$$

where N_v = void number density

M = magnification of the micrograph

L, W = length and width of the micrograph, respectively

$\bar{d}_v$ = average void diameter

$\bar{D}_v$ = diameter of the void with the average volume

$\Delta V/V$ = void volume fraction

S = standard deviation

III. Results and Discussion

The observed number of voids, n_i , represents the total number of voids in the i -th size class whose images lie wholly within the field of view including those sectioned at the surface of the foil, and those whose images are intersected by the edges of the micrograph but with the centers lying within the boundaries of the micrograph. Since n_i contains the voids whose centers lie outside the specimen surface as well as inside, the true number of voids, n_i^t , is represented by relationship (1) in the previous section. The sizing and counting of surface voids do not introduce a significant source of error for the following reason: Although it should be realized that there is a possibility of preferential attack to the surface voids by the electrolyte during the thinning process, which could lead to enlarged void sizes (i.e. surface effect), the surface voids also suffer a truncation effect; that is, the diameters of the segments of the voids truncated by the specimen

surface are generally smaller than the major diameters of the voids. Therefore, these two effects act in the opposite sense and cancel each other to some extent.

A histogram and a cumulative frequency-distribution curve are shown in Figure B.7-1. The histogram was generated by plotting the true number of voids against the interval centers, while the cumulative frequency-distribution curve was obtained by plotting the cumulative percentages against the upper interval limits. The void parameters were calculated using the relationships (2) through (6) and are given below. In general the number of small voids is underestimated because of an overlap effect and because of the reduced

FIGURE B.7-1. Histogram and Cumulative Frequency-Distribution Curve for Void Sizes Measured on "Common Micrograph"

sampling probability on the specimen surface (sampling effect). Although these effects are neglected in this study, it seems that these are contribution factors for the positively skewed void size distribution, as shown in the histogram in Figure B.7-1 and the calculated skewness value ($= 0.382$).

Void Parameters Obtained From Common Micrograph

Void Number Density, $N_v = 5.57 \times 10^{16}$ voids/cm³

Mean Void Diameter, $\bar{d}_v = 76.76 \text{ \AA}$

Diameter of the Void With the Average Volume, $\bar{D}_v = 83.18 \text{ \AA}$

Void Volume Fraction, $\Delta V/V = 1.678\%$

Standard Deviation, $S = 22.28 \text{ \AA}$

Median Diameter = $73.92 \text{ \AA}$

Skewness = 0.382

Specimen Thickness, $t = 1050 \text{ \AA}$

Magnification, $M = 400,000 \times$

B.8 UNIVERSITY OF WISCONSIN

Measurements on duplicate copies of a common micrograph supplied by NRL were made using the techniques of counting and sizing voids that was previously described (see Appendix A.3). The foil thickness determined by NRL ($1050 \text{ \AA}$) for this common micrograph was used to calculate the void volume fraction. The results of this experiment are shown below. The void size distribution is shown in Figure B.8-1.

Average void size	$84 \text{ \AA}$
Void density	$5.86 \times 10^{16} \text{ voids/cm}^3$
Void Volume Fraction	2.2%

FIGURE B.8-1. Histogram of Void Size Distribution Measured on the "Common Micrograph"

APPENDIX C

INTERLABORATORY COMPARISON OF ROUND ROBIN TEM FOIL
OF THE
BODY-CENTERED CUBIC ION CORRELATION PROGRAM

APPENDIX C

INTERLABORATORY COMPARISON OF ROUND ROBIN TEM FOIL OF THE BODY-CENTERED CUBIC ION CORRELATION PROGRAM

This appendix contains detailed description of the techniques and experimental results from Part 3 - Interlaboratory Comparison of Round Robin Tem Foil of the BCC-ICE program. Some editing of the material has been done but the content remains basically as supplied by the participants.

C.1 ATOMICS INTERNATIONAL

The data are presented in Table C.1-1, where they are categorized according to photographs taken of four different foil areas. Composite values are also given in the table; these should be used in comparison of our results with those obtained by other laboratories. A histogram showing the void size distribution is given in Figure C.1-1, along with a normal distribution fitted to the data.

Stereo analysis was used to determine the foil thickness; voids were used to establish the upper and lower surfaces of the foil. Counting and sizing of voids was done with a Zeiss particle-size analyzer operated in the reduced, linear mode.

TABLE C.1-1 Data for Mo "Round Robin" Tem Foil

<u>Photo Plate Number</u>	<u>Foil Thickness (Å)</u>	<u>Number of Voids Counted</u>	<u>Void Density (cm⁻³)</u>	<u>Average Void Size (Å)</u>	<u>Void Volume Fraction (%)</u>	<u>Swelling (%)</u>
2-414	850	613	4.55x10 ¹⁶	89	1.98	2.02
4-414	500	499	3.71x10 ¹⁶	77	1.20	1.22
6-414	400	529	5.20x10 ¹⁶	82	1.98	2.02
12-414	400	482	4.74x10 ¹⁶	81	1.68	1.71
Composite	-	2123	4.55x10 ¹⁶	82	1.71	1.74

FIGURE C.1-1. Histogram of Void Size Distribution Measured on the TEM "round robin" Specimen.

C.2 ARGONNE NATIONAL LABORATORY

Void parameters determined on "round robin" specimen:

Void size	$64 \text{ \AA}$
Void density	1.2×10^{17}
Void swelling	2.1%

C.3 MASSACHUSETTS INSTITUTE OF TECHNOLOGY

Procedures:

A. Electron Microscopy:

The sample was examined using a Phillips EM300 Electron Microscope with a tilting stage. An accelerating potential of 100 KV was used. To maximize contrast, the incident beam was deviated slightly from the exact diffracting condition for (100) planes and a small degree of underfocus. Care was taken that the optical axis coincided with the tilt axis. This removes the necessity to account for a change in magnification during tilt. Credit for the electron microscopy is due to Professor J. Vander Sande.

B. Void Counting:

The void size distribution was measured with a Zeiss TGZ3 Particle Size Analyzer. Every visible void within a reduced area of the micrograph was counted, excluding only those whose centers were outside the field of view. The reduced-exponential-distribution mode was used for more accurate counting. The true diameter of the void was taken as equivalent to the outer portion of the dark contrast ring surrounding each void.

C. Thickness Determination:

The foil thickness at the location of interest was determined using the stereo parallax technique. When the sample is properly aligned, the height difference between two voids lying on opposite surfaces is given by:

$$\Delta h = P/2M \sin \gamma/2 \quad (\text{eq'n 1})$$

where Δh = height difference,
M = magnification,
P = measured parallax and
 γ = total tilt angle.

A large number of parallax measurements were made using a vernier caliper. For reasons explained in the discussion, a distribution in height was found. The true thickness was taken to be the tail of the distribution curve shown in Figure C.3-1.

FIGURE C.3-1. Histogram of Distribution of the Measurements of Foil Thickness on the TEM "Round Robin" Specimen

D. Data Reduction and Analysis:

The raw data from the particle size analyzer were input to a Fortran Code which applied the appropriate correction factors, performed a statistical analysis and plotted the results using a Calcomp plotter. A correction accounting for the voids whose centers lie outside of the plane of the foil was used:

$$N_i^C = N_i^O / (1 + IC_i / t) \quad (\text{eq'n } 2)$$

where N_i^C = corrected number of voids for each size class,

N_i^O = observed number of voids for each size class,

IC_i = Interval Center for each size class,

and t = foil thickness

Discussion:

A problem existed in determining the foil thickness as is evidenced by the distribution in Figure C.3-1. The spread in the distribution is due to two causes: 1) Difficulty in determining voids which were definitely on the surfaces--possibly due to the relatively large tilting angle of 15° , and 2) the dependence of the apparent height on the orientation of the void pair. It is obvious that if the projection of a line connecting the void pair is parallel to the tilt axis, the parallax will be greatly reduced. Another complication, both to this and to void counting was the disappearance of voids between the two elements of the stereo pair. A number of voids weakly visible in one photograph, were not visible in the other. This was most probably due to a change in contrast as the specimen was tilted. The alternate technique of thickness determination utilizing thickness fringes was not applicable due to the presence of radiation damage as well as a large degree of bending evidenced by the presence of bend contours. The most appropriate solution to this problem was to take the thickness as the tail of this curve.

An error estimate was made by making duplicate measurements on one photograph to determine inaccuracies in measurement. This, coupled with an estimated inaccuracy in the tilt stage of $\pm 0.5^\circ$ leads to an estimate of the error in the thickness $\pm 15\%$. This will also be the possible error in the calculated volume fraction. Within a 95% confidence level, the measured void distribution is within 9% of the true distribution.

There is definite evidence of the formation of a void superlattice. This has been observed by many investigators. As might be expected, the

visibility of the void superlattice is highly dependent upon the orientation of the specimen. The void data are summarized below and the void size distribution plotted in Figure C.3-2.

Number of Voids	=	875.73
Average Diameter	=	71.41 Å
Average Area	=	0.49E+04 Å ²
Average Volume	=	0.30E+06 Å ³
Thickness	=	425.00 Å
Volume Fraction	=	0.029633
Voids/cc	=	0.10E+18
Variance	=	668.56 Å ²
Standard Deviation	=	25.86 Å
Skewness	=	0.42
Kurtosis	=	3.08

FIGURE C.3-2. Histogram of the Void Size Distribution Measured on the TEM "Round Robin" Specimen

C.4 NAVAL RESEARCH LABORATORY

NRL supplied the "round robin" specimen. The contribution from NRL would be given in Appendix A.2 in the discussion of Part 1 of the overall Body-Centered Cubic Ion Correlation Program.

C.5 OAK RIDGE NATIONAL LABORATORY

"Round Robin" Specimen

The thickness of the foil area used was determined by two independent methods. The area was imaged in dark-field at exact Bragg position using a 110-type reflection. An area at $4\xi_{110}$ was selected. The value of ξ_{110} used was 200 Å (Bentley, 1974; Howie and Basinski, 1968). The second measurement was made from a bright-field stereo pair obtained using a 200 reflection. Dislocation image termination points were used to define the foil surfaces and the parallax was measured using a Hilger-Watts floating spot mirror stereoscope. The mean of many parallax measurements yielded a foil thickness of 824 Å.

The voids were imaged using a $\bar{3}30$ reflection near a 115 pole and from a through focus series a micrograph which was slightly overfocussed (~ 8000 Å) was selected. The void diameter is measured to the inside of the bright Fresnel-like fringe.

Void size distributions were obtained using a calibrated Zeiss particle size analyzer for four areas and a total of 687 voids. The data were processed to yield the following parameters:

(1) Mean void size $\sum_i n_i d_i / \sum_i n_i = 87.4 \text{ Å}$

Standard distribution = 2.0 Å

(2) Volume averaged mean void size $(\sum_i n_i d_i^3 / \sum_i n_i)^{1/3} = 96.4 \text{ Å}$

Standard deviation = 2.5 Å

(3) Void density = $6.04 \times 10^{16} \text{ cm}^{-3}$

Standard deviation = $0.12 \times 10^{16} \text{ cm}^{-3}$

(4) Void volume fraction = 2.83%

Standard deviation = 0.15%

* Standard deviation of the measurements

The void lattice parameter was also determined. From measurements on micrographs at [100] beam direction, $a_v = 375 \text{ \AA}$. From measurements of the spacing of the extra spots on diffraction patterns caused by diffraction by the void lattice (Sass and Eyre, 1973) and using 110 matrix spots as a calibration, $a_v = 378 \text{ \AA}$.

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C.6 PACIFIC NORTHWEST LABORATORY

Foil thickness is determined by making parallax measurements from stereo pairs of electron micrographs using a Hilger-Watts floating spot stereoscope. The parallax measurements are converted to foil thickness by the relation:

$$t = \frac{\Delta P}{2M \sin(\frac{\Theta}{2})}$$

where t = the foil thickness,

ΔP = the parallax,

M = the magnification of the micrograph,

Θ = the tilt angle between the stereo pairs.

The magnification and the tilt angle depend on the void size and void density in the specimen being examined. The magnification and tilt angles normally used range from 30 to 80 KX and 13° to 19°, respectively.

The procedure for obtaining the stereo pairs is as follows: First, the specimen is tilted to a positive value and the direction of tilt reversed sufficiently to remove any slop in the tilt stage. The electron transparent area is then examined to find a suitable region for the stereo pair. The criteria used for determining a suitable area include, 1) voids in good contrast, 2) the foil thickness appears uniform, 3) the number of voids in the region is sufficiently large to be statistically significant but not so large that excessive overlap occurs, and 4) a marker (i.e. ppt., etchpit, etc.) exists in the region such that the identical area can be located after tilting.

The parallax measurements are made, using the electron image negatives, on a Hilger-Watts floating spot stereoscope. An area is marked off on a photographic print of one of the stereo pairs and at least five parallax measurements are taken from this region with the average value taken as the foil thickness. The parallax measurements normally correspond to the separation between one of the foil surfaces and a void near the opposite surface. If a surface is not clearly resolvable, the maximum void to void separation is measured and the foil thickness is determined by adding one mean void diameter to the calculated void to void separation.

The error associated with the foil thickness determinations is estimated to be less than $\pm 22\%$. This value represents the maximum uncertainty due to the propagation of errors in the magnification (5%), tilt angle (5%), and parallax measurements (10%).

The following void parameters were determined on the "round robin" specimen:

Average void diameter	83.9 Å
Volume average void diameter	92.2 Å
Standard deviation	24.9 Å
Skewness	0.25
Total voids measured	1167
Average void density	6.35×10^{16} voids/cm ³
Void volume fraction $\Delta V/V$	2.62%
Void swelling $\Delta V/V_0$	2.67%
Void lattice parameter	365 Å

C.7 UNIVERSITY OF CINCINNATI

I. Experimental Procedure

The round robin specimen supplied by NRL was examined in a JEM 200A transmission electron microscope equipped with 30° tilt - 360° azimuth goniometer stage, operated at 200 kV. The goniometer had been calibrated for tilting angle against the Kikuchi line shifts. Also the tilt axis of the goniometer was known fairly accurately.

Stereo-pictures of the voids were taken at different areas of the specimen with a tilting angle of 10° at a magnification of 50,000 X. The pictures were taken with the voids imaged both in underfocused and overfocused conditions. Diffraction patterns of the specimen before and after the tilting were also obtained for a Kikuchi line analysis. The pictures were enlarged by three times; i.e., the magnification of the micrographs is 150,000 X, and the void size and density were determined by using a TGZ 3 Zeiss Particle Size Analyzer as described in the previous report.

The thickness of the specimen was calculated using the relationship:

$$t = \frac{P}{2M \sin \Theta} \quad (1)$$

where t = specimen thickness
 P = parallax
 M = magnification
 Θ = one half of the total tilting angle

The parallax, P , was measured at several different areas of the micrograph by using a mirror stereoscope (stereo-viewer) equipped with a micrometer head and floating mark units. The voids which appeared to be on or near the surfaces of the specimen were chosen as reference points in measuring the parallax, and the average value of the measurements obtained from several different areas of the micrograph was used to calculate the specimen thickness in the region from which the micrograph was obtained.

The tilting angle was determined primarily by the goniometer readings, and subsequently by indexing the Kikuchi patterns before and after the tilting and thus calculating the angle between the two zone axes.

II. Results and Discussion

The true number of voids was calculated using the following relationship, described previously (see appendix B.7)

$$N_i^t = \frac{N_i}{1 + \bar{d}_i/t} \quad (2)$$

where N_i^t = true number of voids for i-th size class.
 N_i = observed number of voids for i-th size class.
 $\bar{d}_i$ = interval center of i-th size class.
 t = thickness of the specimen.

The above correction is necessary because the image of the sectioned voids with their centers outside the specimen surface lies within the field of view. As can be seen in Equation (2), the correction is significant especially for a large void size and/or a thin specimen. For a void size of 100 Å and a specimen thickness of 500 Å, for example, the correction will make a difference of ~20% in the void number density.

The histogram and the cumulative frequency-distribution curve of the corrected void data are shown in Figure C.7-1. The void parameters calculated using both the corrected and uncorrected data are presented in Table C.7-1. However, it should be noted that the void number density varies noticeably from one area of the specimen to another, and this could effect the results significantly, especially when the void pictures are taken at a high magnification. Moreover, one tends to take a picture from an area where the void population is dense, and this, too, can effect the results significantly: in this case the results represent higher-than-true average values.

FIGURE C.7-1. Histogram and Cumulative Frequency-Distribution Curve of the Void Data Obtained from the NRL "Round Robin" Mo Specimen

In general, most of the uncertainty is in the determination of tilting angle, when stereomicroscopy is used for determining the void parameters. If one allows 0.5° uncertainty in a goniometer tilting stage and 0.2° in Kikuchi pattern analyses, the tilting angle determined in this study is consistent with each other: 10° vs. 10.6° . Also, if the tilt axis is not perpendicular to the optical axis, the goniometer reading will be always larger than the true tilting angle, and again this is not the case in this study.

TABLE C.7-1. Void Parameters Obtained from NRL Round Robin Sample

	Sample Area #1		Sample Area #2	
	Before Correction	After Correction	Before Correction	After Correction
Void Number Density, N_v , Voids/cm ³	$4.4 \times 10^{16} \text{ #/cm}^3$	$3.8 \times 10^{16} \text{ #/cm}^3$	$4.6 \times 10^{16} \text{ #/cm}^3$	$3.8 \times 10^{16} \text{ #/cm}^3$
Mean Void Diameter, $\bar{d}_v$, Å	86.1 Å	85.4 Å	84.0 Å	83.0 Å
Diameter of the Void with Average Volume, $\bar{D}_v$, Å	91.2 Å	90.6 Å	89.6 Å	88.5 Å
Void Volume Fraction, $\Delta V/V$, %	1.75%	1.48%	1.73%	1.38 Å
Standard Deviation, S, Å	21.0 Å	20.8 Å	22.0 Å	21.8 Å
Median Diameter, Å	82.3 Å	81.6 Å	81.4	80.2 Å
Skewness	0.54	0.55	0.35	0.39
Specimen Thickness, t, Å	615 Å	615 Å	415 Å	415 Å
Magnification, M	150,000 X	150,000 X	150,000 X	150,000 X
Void Image Condition	Under	Under	Over	Over
No. of Voids Counted	1180	1030	1300	1080

C.8 UNIVERSITY OF WISCONSIN

The same procedure of microscopy analysis and void size measurement as described previously (see Appendix A.3) was applied to examine this specimen at the University of Wisconsin. The results are shown below.

Average void size	$85 \pm 4 \text{ \AA}$
Void density	$6.4 \pm 1.6 \times 10^{16} \text{ voids/cm}^3$
Void volume fraction	$2.6 \pm 0.5\%$

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Lawrence Livermore
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Laboratory
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Livermore, CA 94550

W. Bauer
Division Supervisor of
Physical Research
Sandia Labs Livermore
Livermore, CA 94550

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Los Alamos Scientific
Laboratory
CTN Research
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Los Alamos, NM 87544

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Los Alamos Scientific
Laboratory
CTN Research
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