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Principal Co-Investigators: R. W. Balluffi, Department of
Materials Science and Engineering

David N. Seidman, Department of
Materials Science and Engineering

Cornell University, Ithaca, New York

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DEFECTS IN METAL CRYSTALS

R. W. Balluffi and David N. Seidman

ABSTRACT

The objective of the present research effort was a broad investigation of the basic properties of crystal defects in metals. Attention was focused on point defects (vacancies, interstitials, and solute atoms), line defects (dislocations), and planar defects (dislocation arrays, grain boundaries and surfaces).

Progress was made in the following areas:

- (A) A Quantitative Study of Vacancy Defects in Quenched Platinum by Field Ion Microscopy (FIM) and Electrical Resistivity
- (B) FIM and Electrical Resistivity Study of Vacancy Defects in Tungsten
- (C) FIM Studies of Defects in Platinum Irradiated with Platinum Ions or Electrons
- (D) FIM Studies of Defects in Tungsten Irradiated with Tungsten Ions or Electrons
- (E) The FIM of Gold and In-Situ Studies of Gold Irradiated with Gold or Argon Ions
- (F) Computer Simulation of Contrast Effects Produced by Defects in FIM Specimens
- (G) On the Interpretation of Ledge "Bright Spot" Contrast Effects in Field Ion Microscope Images
- (H) On the Question of the Possibility of an Equilibrium Array of Voids in a Material Subject to Irradiation

- (I) FIM Studies of Solute Atoms in Dilute Platinum Alloys
- (J) Studies of the Structures of Low and High Angle Grain Boundaries
- (K) The Direct Measurement of the Range of a Focused Collision Replacement Sequence in Gold
- (L) Automatization of FIM Data Processing
- (M) The Atom Probe FIM

I. INTRODUCTION

During the past year a number of projects was completed, and significant progress was made in other areas. A brief account of the work is given in the following section.

The following personnel were involved:

Professor R. Balluffi, Principal Co-Investigator

Professor D. Seidman, Principal Co-Investigator

Professor P. S. Ho, Associate Investigator (left June 1972)

Dr. A. Berger, Research Associate

Dr. R. Benedek, Research Associate

Dr. J. Robinson, Research Associate

C. G. Chen, Research Assistant

H. Huang, Research Assistant

J. Park, Research Assistant

A. Wagner, Teaching Assistant

C. Y. Wei, Research Assistant

K. Wilson (Fellowship student)

K. Pratt, Film Scanner

R. Whitmarsh, Technician

II. EXPERIMENTAL WORK

A. A Quantitative Study of Vacancy Defects in Quenched Platinum by Field Ion Microscopy and Electrical Resistivity

The work on this subject discussed in our 1972 Research Renewal Proposal⁽¹⁾ has been completed and written up as two manuscripts^(2,3) which have been accepted for publication in Acta Metallurgica. The two manuscripts deal with the (i) experimental results and (ii) analysis and interpretation of the

research. A summary of this work is as follows.

(i) Experimental Results

Platinum wire specimens were brine quenched from $1700 \pm 1^\circ\text{C}$, and the 4.2°K resistivity of the quenched-in vacancy defects was measured. Field ion microscope (FIM) specimens were then prepared from these same wires, and the vacancy defects were imaged directly in an FIM as each specimen was dissected by pulse field evaporation at the rate of ~ 35 pulses per atomic layer. The surface image was recorded on ciné film after each pulse, and every atomic site in a large number of consecutive layers was recorded. The $\sim 32,000$ ft of film which were exposed were subsequently scanned for the defects. A total of 157 monovacancies and 9 divacancies were found in 593,794 sites in 3 as-quenched specimens, and 76 monovacancies and 1 divacancy were found in 321,298 sites in 5 specimens which were partially annealed after quenching. In order to correct for the presence of possible surface artifact vacancy defects, 319,552 sites were scanned in well-annealed specimens under identical conditions, and only 11 artifact monovacancies were found in the $\{135\}$, $\{023\}$, $\{124\}$, $\{123\}$, and $\{167\}$ planes used for the present study. These observations constitute direct evidence that the predominant equilibrium defect at elevated temperatures in platinum is the monovacancy. The value of the monovacancy concentration quenched-in from $1700 \pm 1^\circ\text{C}$ is $(2.64 \pm 0.14) \cdot 10^{-4}$ at.fr., with the recognition that this is less than the equilibrium value because of quenching losses. The combination of the measured concentrations and resistivity increments yielded a monovacancy resistivity of $(5.75 \pm 0.30) \cdot 10^{-4} \Omega\text{cm}$.

(at.fr.)⁻¹. An observed preferential loss of divacancies during the partial annealing of the 5 specimens provided direct evidence that the divacancy is more mobile than the monovacancy. The measured ratio of the divacancy concentration to the monovacancy concentration in the as-quenched specimens was (0.06 ± 0.02). The relationship of this value to the divacancy binding free energy is discussed in (ii) analysis and interpretation.

(ii) Analysis and Interpretation

Under (i) Experimental Results it was demonstrated by means of field ion microscopy that the measured ratio of divacancy concentration to monovacancy concentration (c_{2v}/c_{1v}) in high purity platinum specimens quenched from 1700 ± 1°C was 0.06 ± 0.02. As part of the analysis and interpretation a detailed series of quantitative calculations was first performed which showed that this ratio was not affected by specimen storage and electropolishing procedures subsequent to the quench and hence represented c_{2v}/c_{1v} at the "freeze-out" temperature (T^*). It was then shown that all combinations of the divacancy binding energy (E_{2v}^b) and binding entropy (S_{2v}^b) which lead to a quenched-in value of c_{2v}/c_{1v} equal to 0.06 ± 0.02 for a quench temperature of 1700°C were governed by the linear relationship

$$E_{2v}^b = [0.23 \pm 0.03] + 6.17 \cdot 10^{-2} \left(\frac{S_{2v}^b}{k} \right) \text{ eV ,}$$

where k is Boltzmann's constant. This linear equation implied that the divacancy binding free energy (G_{2v}^b) was 0.23 eV at $T^* = 443.4^\circ\text{C}$. This value of G_{2v}^b , calculated from our experimental data,

depended only on a knowledge of the monovacancy migration energy, and an approximate value of a frequency factor. The existing tracer diffusion data for platinum were re-examined in terms of a monovacancy and divacancy model in an attempt to decompose G_{2v}^b into E_{2v}^b and S_{2v}^b terms, and it was concluded that the available data are not sufficiently accurate to make this decomposition significant.

B. FIM and Electrical Resistivity Study of Vacancy Defects in Tungsten

The study of vacancy defects in tungsten by field ion microscopy and electrical resistivity has been continued during the past year. The overall aim of this work is to obtain reliable information about the properties of monovacancies and divacancies in this BCC metal which is similar to that which we have obtained previously for FCC platinum (see II.A.). Tungsten was chosen, since it is particularly well suited for investigation by field ion microscopy. In addition, there is intense current interest in the properties of vacancy defects^(4,5) and their relationship to self-diffusion in BCC metals such as tungsten⁽⁶⁾.

As part of this program, FIM specimens of high purity tungsten have been prepared and have been bombarded with keV tungsten ions at room temperature in our ion accelerator in order to produce a distribution of vacancy defects containing monovacancies and possibly higher order vacancy clusters. Vacancy defect annealing occurs well above room temperature in tungsten, and these specimens have been annealed isothermally at a series of elevated temperatures in order to allow the vacancy defects either to

migrate out of the specimens or else form clusters. The defect structures in a considerable number of these specimens have been imaged during specimen dissection in the field ion microscope by the pulsed field evaporation technique, and the results have been recorded on ciné film. Detailed determinations of the concentrations and spatial distributions of the defects in various stages of the annealing are now being obtained from appropriate analysis of the films. These results should allow us to deduce properties of the observed point defects such as migration energies and binding energies.

A highly efficient and semi-automated film analysis technique has been recently developed which should greatly reduce the labor involved in the above work. The exact lattice position of each point defect is calculated by our on-line Nova computer (see Section II.L) after it has received appropriate input data from the film scanner. This information is accumulated in the computer, and final information about various aspects of the overall distribution of all the defects can be obtained from the computer after appropriate calculations using the stored data are carried out. Successful test runs of the various techniques have been performed, and we are about ready to apply them to the ciné film of the irradiated and annealed tungsten.

In another part of our study of the properties of vacancy defects in tungsten we are quenching high purity tungsten wires at high enough rates to retain a substantial fraction of the vacancy defects initially present in thermal equilibrium at elevated temperatures. Defect resistivity increments quenched

into these wires are then measured. FIM specimens are subsequently prepared from the same wires, and the concentrations of the vacancy defects present in them are measured using the same techniques which we have employed to investigate the vacancy defects retained in quenched platinum (see II.A.). The experimental results should lead to the monovacancy formation energy, the resistivity per monovacancy, and possibly the divacancy binding energy.

Thin 0.0075 cm dia single crystal wires of high purity (i.e., $\lesssim 1$ ppm impurities) have been prepared by a special electropolishing technique. A wire of this type is then mounted in a UHV quenching manifold at a pressure $\lesssim 10^{-10}$ torr. The wire is then resistively heated to near its melting point and radiation quenched by simply turning off the heating current. Quenching rates in the range $40,000^\circ\text{K sec}^{-1}$ are obtained by this method. This technique, therefore, constitutes an ultra-clean method for rapidly quenching tungsten from near its melting point. We have found that the base resistance of the specimen measured at 4.2°K remains constant during a number of successive heating and cooling cycles in this arrangement, indicating that no impurities are being picked up. Several quenching runs have been completed, and defect resistivity increments have been quenched in which are in reasonably good agreement with those obtained by Gripshover, et al. (4)

Considerable progress has been made in the construction of electronic circuitry which will enable us to obtain instantaneously the temperature versus time quenching curves of our thin specimen.

This system feeds two measuring voltages to our Nova computer where the data are processed and finally plotted out directly in the form of quenching curves. We have also constructed additional circuitry which will enable us to quench the specimen at different rates and also perform quenching and annealing runs over a wide range of thermal histories. This equipment will be used very shortly in an extensive series of vacancy quenching and annealing studies. The resulting annealing data should allow us to deduce the migration energy (energies) of the quenched-in defects.

In the field ion microscopy part of the work several quenched specimens have been dissected in the FIM by pulsed field evaporation, and the resulting ciné films are being scanned for the presence of quenched-in vacancies.

C. FIM Studies of Defects in Platinum Irradiated with Platinum Ions or Electrons

The radiation damage experiments on platinum involved three major topics. They were as follows:

- (1) A study of long range migration of self-interstitial atoms (SIA's) in Stage I of platinum⁽⁷⁾ irradiated with 20 or 30keV Pt⁺ ions.
- (2) A search for the Stage III SIA which may have formed as the result of a thermally activated conversion process in electron irradiated platinum.
- (3) A study of the structure of the depleted zones detected in the ion irradiated specimens.

(i) Stage I Research

Direct visual evidence for uncorrelated long-range migration of self-interstitial atoms (SIA's) in Substage I_E of

irradiated platinum was obtained by a series of in-situ field ion microscope (FIM) experiments. High purity $[(2 \text{ to } 6) \cdot 10^{-5} \text{ and } (1 \text{ to } 3) \cdot 10^{-6} \text{ at.fr. impurity level}]$ platinum specimens were irradiated with 20 or 30 keV Pt^+ ions under ultra-high vacuum conditions at a specimen temperature of 10 or 11°K. The initial damage pattern at 10°K consisted of depleted zones within $\sim 100 \text{ \AA}$ of the irradiated surface and a distribution of immobile SIA's in the bulk of the specimen. Next the specimens were isochronally warmed from 10 or 11°K to 75°K [average linear warming rate = $(1/3)^\circ\text{K min}^{-1}$] at a constant value of the imaging electric field while the specimens' surfaces were photographed at a rate of 1 frame sec^{-1} . This experiment showed that a flux of SIA's crossed the specimens' surfaces between 13 and 27°K. A detailed series of control experiments were performed to demonstrate that the contrast effects observed were not due to impurity atoms or surface artifacts. Application of our diffusion model (Scanlan, Styris and Seidman) to the isochronal recovery spectrum yielded an enthalpy change of migration of $\sim 0.04 \text{ eV}$ and a diffusion coefficient of $\sim (0.08 \text{ to } 0.77) \exp(-0.04 \text{ eV}/kT) \text{ cm}^2 \text{ sec}^{-1}$ for the SIA. The FIM specimen was, of course, subjected to a negative hydrostatic pressure during the field ion imaging process which was caused by the required imaging field of 4 to $4.75 \text{ V}(\text{\AA})^{-1}$. Thus, it was possible to combine an existing value for the internal energy change of migration for recovery in Substages I_D and I_E with our enthalpy change of migration to yield a range of limiting values for the volume change of migration of a SIA $[(2.3 \text{ to } 3.2) \cdot 10^{-2} \text{ atomic volume}]$.

(ii) Stage II and Stage III Research

The main objective of this research was to search for a second SIA which had formed as a result of a thermal conversion process, since its presence or absence is crucial to the controversy concerning the "one-interstitial" model versus the "two-interstitial" model.

Consider the possibility that the fast SIA is injected into the specimen at some temperature T which is above Stage I, but below Stage III. The root mean square distance traveled by the fast SIA in a time t is then given by the equation

$$\sqrt{\bar{x}_i^2} = \sqrt{6D_{li}t} \quad (1)$$

The time t for the fast SIA to thermally convert to the slow SIA is given by

$$t = \frac{1}{\nu} \exp (H_{li}^C/kT) \quad (2)$$

where H_{li}^C is the conversion enthalpy, and ν is the standard attempt frequency ($\sim 10^{13} \text{ sec}^{-1}$). Thus, the mean distance traversed by the fast SIA before it converts is

$$\bar{x}_i = \left[\frac{6D_{li}^0}{\nu} \right]^{1/2} \exp \left[\frac{H_{li}^C - H_{li}^m}{2kT} \right] \quad (3)$$

where D_{li}^0 is the pre-exponential factor, and H_{li}^m the enthalpy of migration of the fast SIA. A necessary condition for the slow SIA to be of importance physically at any T is that $\bar{x}_i$ be less than the typical distance to a SIA sink. In the case of our FIM experiments the largest possible value of $\bar{x}_i$ is the radius of the tip, which is typically 100 to 300 Å. For H_{li}^C greater than H_{li}^m we see that $\bar{x}_i$ decreases as T increases. Thus, the chance of seeing

a converted SIA is enhanced by irradiating the specimen at as high a T as possible which is still below the onset of Stage III.

Three specimens were irradiated at 498, 504 and 508°K respectively, and a total of 782,384 atomic sites were examined for SIA's, and a total of 9 extra bright spots were detected among these sites. Eight of the spots were on the side close to the irradiated surface, and one was on the far side away from the irradiated surface. One of the problems with conclusively identifying these bright spots as SIA's is that we have recently observed anomalous field evaporation effects associated with the depleted zones which can give rise to extra bright spots. Since these bright spots were associated with depleted zones we have not as yet reached a decision as to whether or not they were converted (slow) SIA's.

To solve the above aspect of the problem we are and have been preparing high purity platinum single crystal wires for subsequent electron irradiation. During the past year a technique was developed for electropolishing zone-refined 1.5 mm diam single crystal rods of platinum down to 0.13 mm diam wires. This technique is important since it is difficult to draw wires from the high purity rod form into the wire form without contaminating the specimens. In addition, a study was made of the heating effects occurring in the tip region during the process of electropolishing a wire specimen into an FIM tip. The results of this experiment established an upper limit to the current density, in the electrolytic cell, which will prevent the electron irradiated tip region from being resistively heated into the Stage III regime.

The electron irradiation* of the bulk specimens will produce a simple distribution of isolated Frenkel pairs, and hence will avoid the problem of the presence of depleted zones in the specimens.

(iii) Structure of Depleted Zones

The structure of the depleted zones produced by the 20 or 30keV Pt^+ ion irradiations was analyzed in a manner similar to that previously described by Beavan, Scanlan and Seidman⁽⁸⁾. The depleted zones in platinum were found to be rich in vacant lattice sites which once again indicated the need for a mechanism to transport atoms away from the region where each depleted zone was created. The most likely mechanism for this is the focused collision replacement sequence.

In addition to mapping out the three dimensional spatial distribution of vacant lattice sites within the depleted zone the divacancy and monovacancy concentrations in the depleted zones were monitored as a function of annealing temperature⁽⁹⁾. That is, a series of specimens was irradiated at 10°K and then annealed at various temperatures between 35°K and 498°K before being examined at 25°K by the pulse field evaporation technique. The measured ratio of the monovacancy concentration to the divacancy concentration

* Arrangements have been made with Dr. J. DiCarlo of the NASA laboratory in Cleveland, Ohio to perform the electron irradiations of both platinum and tungsten (see II.D.) specimens. Dr. DiCarlo has extensive experience in field of radiation damage and has been extremely cooperative in helping us to irradiate these bulk specimens.

Table I
STAGE II IRRADIATION EXPERIMENTS ON TUNGSTEN

Specimen	Irradiation Temperature (°C)	Radius (Å)	Dose ions (cm ⁻²) & (energy)	Number of atoms counted in the (111) region	Number of SIA's
1	100	100	$1.2 \cdot 10^{12}$ (20 keV)	10,000	0
2	200	100	$9.4 \cdot 10^{11}$ (20 keV)	11,322	0
3	200	115	$2.8 \cdot 10^{12}$ (20 keV)	26,400	0
4	280	200	$1.1 \cdot 10^{13}$ (30 keV)	23,585	² ($8.5 \cdot 10^{-5}$ at.fr)
5	285	160	$2.5 \cdot 10^{12}$ (30 keV)	10,700	0

increased strongly between 360 and 498°K. This result indicated that divacancies became mobile somewhere in this temperature range, and that the divacancies were more mobile than single vacancies. This result represents the first direct proof of this fact concerning the relative mobilities of mono versus divacancies.

D. FIM Studies of Defects in Tungsten Irradiated with Tungsten Ions or Electrons

This research represents a continuing effort to determine the atomic mechanisms responsible for recovery in Stages I through III of tungsten. The metal tungsten was chosen as a prototype BCC metal because of its amenability to the FIM technique as well as our ability to prepare extremely pure specimens of this material. We have previously shown that a SIA undergoes long range migration in substage I_E of tungsten [Scanlan, Styris and Seidman^(10,11) and Seidman⁽¹²⁾].

The present efforts on tungsten are concerned with the following problems:

(1) The question of the existence of a second SIA which is capable of long range migration in stage III.

(2) The geometric configuration of the SIA which migrates in substage I_E.

(3) The role of impurities in the recovery processes involved in stages I to III recovery.

(4) The effects of pressure on the SIA mobility.

(i) Stage III Recovery Mechanism

The theory of this experiment is identical to that described in II.C. for the case of platinum. Briefly, the experiment involves irradiating a specimen at a temperature (T) which is below Stage III, but at as high a T as possible in Stage II in order to decrease the distance the fast SIA travels before it thermally converts to a slow SIA. The results for five different specimens which were irradiated at temperatures between 100°C and 289°C and pulse field evaporated at 18°K are given in Table 1. To date only two possible SIA's have been found. These experiments tend to indicate either that the conversion enthalpy change (H_{1i}^C) is high, or else that the slow SIA does not exist.

To check critically as to the question of the existence of the slow SIA a series of bulk tungsten specimens (0.13 mm diam rod) were irradiated with 2.35 MeV electrons to a dose of 1×10^{20} electron cm^{-2} at the NASA Laboratory in Cleveland. The maximum temperature which the specimens reached during the irradiation was $440 \pm 30^\circ\text{K}$.^{*} Two types of specimens, with different impurity levels, were employed for this experiment. The first type was polycrystalline tungsten wire purchased from MRC which had a resistivity ratio [$R = R_{298^\circ\text{K}}/R_{4.2^\circ\text{K}}$] of 20. The second type was zone refined single crystal rod which had an R value of 3×10^4 when measured on a 0.38 mm diam rod. The R values were not corrected for any possible specimen size effects.

* This temperature is below the start of Stage III which is located at 500°K.

At the present time two $R \approx 20$ specimens have been examined in the FIM and a total of 3 contrast effects were found among the 205,000 atomic sites counted, for a concentration of $\sim 1.5 \cdot 10^{-5}$ at. fr. The one $R = 3 \times 10^4$ specimen examined contained 4 contrast effects among 130,000 atomic sites detected for a concentration of $\sim 3 \times 10^{-5}$ at. fr. None of the contrast effects observed involved the "bright spot" contrast effects we have used previously to identify SIA's, but instead consisted simply of vacant site type contrast effects. In view of the low concentration of contrast effects detected the future research will involve the dissection of many additional specimens in order to make the results statistically significant.

(ii) Geometric Configuration of the SIA

We are attempting to determine the geometry of the SIA employing the FIM contrast model developed by Seidman and Lie⁽¹³⁾. The procedure for this experiment is as follows:

- (1) Irradiate at a temperature which is below the onset of long range migration in Stage I ($< 20^\circ\text{K}$ in this case).
- (2) Dissect the specimen by the pulse field evaporation technique at the irradiation temperature in a manner such that the (111) plane is dissected one atom at a time.
- (3) Compare the dissection sequences to the calculated contrast patterns.

The calculated contrast patterns contain enough information to enable one to distinguish between a one-dimensional SIA (the crowdion) and a three-dimensional SIA (the split or dumb-bell interstitial).

(iii) The Role of Impurities in the recovery processes involved in stages I to III recovery

Preliminary evidence obtained in this laboratory [Scanlan, Styris and Seidman^(10,11) and Wilson and Seidman⁽¹²⁾] has shown that the recovery in both Stages I_E and II of tungsten is very sensitive to rather small impurity concentrations. A possible explanation of the long tail in the isochronal recovery spectrum of tungsten involves the detrapping of SIA's from impurity atom traps. Since this detrapping effect has been observed over the temperature range ~50 to 220°K it is most likely that Stage I of tungsten corresponds to the range 0 to ~40°K, and not 0 to ~100°K as suggested previously⁽¹⁵⁾. The trapping effects are strong enough such that SIA's can be retained in the lattice to temperatures as high as room temperature.

In view of the above effects we will try to determine the specific impurity atoms which bind to SIA's in tungsten employing the atom probe FIM constructed recently in our laboratory (see Section II.M.).

(iv) Effects of Pressure on SIA Mobility

We have previously shown [Scanlan, Styris and Seidman^(10,11)] by means of a series of control experiments that the volume change of migration of a SIA in tungsten must be ≤ 0.1 atomic volume. This problem has now been considered in more detail in order to demonstrate that the recovery previously observed at $T < 50^\circ\text{K}$ was not due to the stressed-induced migration of a SIA which was characteristic of a process that would only be observed at $T > 50^\circ\text{K}$ if the large negative hydrostatic pressure caused by the electric field was absent. The experiment consisted of the following steps:

(1) Irradiation of the specimen at 50°K in the absence of the electric field.

(2) Specimen cooled to 18°K in the absence of the electric field.

(3) Specimen warmed isochronally from 18°K to 105°K in the presence of the best image field characteristic of 18°K, while the specimen surface was photographed continuously at a rate of 1 frame sec⁻¹.

The results of this experiment showed that 92% of the total number of defects observed (37) crossed the specimen surface at 50°K or higher. The remaining 8% (2 defects) crossed the surface between 40 and 50°K. This result demonstrated that the peak observed by Scanlan et al.⁽¹⁰⁾ at about 30°K was not due to a SIA which was characteristic of $T > 50^\circ\text{K}$.

It is to be noted that 32.5% of the defects observed consisted of multiple bright spots (two or more spots). This is to be compared to 13.15% for the 18°K irradiation followed by an isochronal anneal in the Scanlan et al. experiments. This result strongly suggests that we have observed some type of elementary cluster in these recovery experiments. Since the fast SIA is mobile during the irradiation, the most probable type of cluster consists of an impurity atom and a SIA. The probability of observing a di-SIA is considerably smaller. It is planned to determine the character of these clusters employing the Atom Probe FIM (See II.M.).

E. The FIM of Gold and In-Situ Studies of Gold Irradiated with Gold or Argon Ions

During the past few years we have demonstrated that quasi-stable images of the relatively soft non-refractory metal gold can be obtained [Ast and Seidman^(16,17), and Schmid and Balluffi⁽¹⁸⁾] via field ion microscopy. In the last year we have developed a technique which allows the production of stable images of gold in the temperature range 11 to 30°K. The development of a stable end form of gold consisted of the following two stage procedure. In the first stage the FIM was baked to 100°C, and then when the pressure was less than 5×10^{-9} Torr the specimen was cooled to approximately 55K. Gauge pressures of 2×10^{-8} Torr air and 3×10^{-5} Torr neon were then admitted dynamically into the FIM via Varian leak valves. The neon was Linde research grade gas (~99.999 vol % pure). Next the tip voltage (V_T) was increased slowly until both the symmetry of the lattice could be recognized and a few net planes were resolved. The tip temperature (T_T) was then decreased to approximately 28K in approximately 5K intervals (V_T was increased at each step). Finally at 28K the tip was developed to the point where all but the low field regions (e.g. {111}) were resolved. The low field regions usually displayed considerable pitting due to field etching. The value of V_T was then decreased to zero, and the FIM was rebaked. This first stage of the development of an end form usually required about 1 hour. The images obtained in this first stage were remarkably symmetric, and they exhibited no streaks or distorted ring structure. Very few specimens failed during this first stage of development, and approximately 10 to 20%

of those which failed behaved anomalously prior to failure. The specimen failure may have been due either to the tip having been damaged during mounting, or to some residual substance in the tip region which had not annealed out.

The second stage of the development of an end form began after the FIM had been rebaked and after the pressure was typically 5×10^{-10} Torr (although pressures as high as 3×10^{-9} Torr were used). The tip was then recooled to 30K and pure neon gas was admitted dynamically to a gauge pressure of 3×10^{-5} Torr. The image usually reappeared with an average tip radius which was 20% greater than the value at the end of the first stage of development. Approximately 10% of the specimens failed during development in second stage. It is noted that without the first stage of development in a neon-air mixture (or perhaps equivalently in an unbaked FIM) we had virtually no success.

In addition to the development of the above technique the characteristic curves between ~21 and 49K were studied photographically and also employing light intensity measurements. A study of the surface artifact vacancy concentration for a number of different {hkl} planes showed that their concentration was ~0.3 at. %. This concentration was approximately an order of magnitude lower than the value found by Ast and Seidman and Schmid and Balluffi. This result implies that the use of ultra-high vacuum conditions is a necessity for the imaging of gold. We are now applying the experience we have accumulated in studying gold to the question of the existence of a low temperature (Stage I) interstitial in irradiated gold.

Of all the FCC metals the Stage I recovery spectrum is the most anomalous, and also the most controversial. The recovery spectra of electron, fast neutron and deuteron irradiated gold have been reviewed by Schilling et al.⁽¹⁹⁾, and it is speculated that the peak which occurs at about 19°K is caused by the long range migration of a SIA, although the indirect evidence is very weak. For example, Bauer and Sosin⁽²⁰⁾ found that quenched-in vacancies had no effect on the recovery of electron irradiated specimens, while Swanson and Piercy⁽²¹⁾ measured an enhanced recovery in pre-quenched fast neutron irradiated specimens which was distributed uniformly over the range of Stage I recovery. If the 19°K peak were truly due to long range migration of a SIA then one would expect an enhanced recovery rate for this peak, but no enhancement of the lower temperature peaks. Thus, in spite of the speculation that this peak is due to the long range migration of a SIA, there is still considerable controversy over the existing data as testified to by the lively round table Panel Discussion at the "International Conference on Vacancies and Interstitials in Metals"⁽²²⁾. Thus, the direct observation of the Stage I SIA by the experiments that we have already employed for studying Stage I in tungsten [Scanlan, Styris and Seidman^(10,11)], and platinum [Pétroff and Seidman^(7,9)] would be useful in solving this outstanding problem.

To date three different gold specimens have been irradiated with 5 or 8keV Ar⁺ ions to doses of $\leq 1 \times 10^{13}$ ion cm⁻². The specimens have been warmed isochronally from 21K and a total of 15 SIA's have been observed to cross the surface of these specimens. A number of

control specimens have also been warmed isochronally from 21°K, and none of these specimens exhibited contrast effects which were similar to the ones which we have attributed to SIA's in the irradiated specimens. Hence these preliminary results indicate uncorrelated long range migration of an SIA in stage I of gold. At present a big effort is being made to improve the statistics of this experiment in order to convert this preliminary result into a more definitive one.

F. Computer Simulation of Contrast Effects Produced by Defects in FIM Specimens

A series of calculations of displacement fields for SIA's in tungsten has now been completed. These calculations were performed using a modification of the Green's function approach⁽²³⁾ which enables an accurate treatment of the large relaxations occurring within the "interstitial core". A description of this method is given in (24). To assess the sensitivity of the results to the assumed interatomic potential the calculation was repeated for (i) a potential fitted to the tungsten phonon spectra (described in last year's report⁽¹⁾) and (ii) the tungsten potential of Johnson and Wilson⁽²⁵⁾. It was found that while the energy and volume of formation are rather sensitive to the potential the displacement fields were nearly the same for both (i) and (ii). (Detailed results are given in (5).) This is encouraging since the interstitial displacement fields are the major input in the model proposed by Seidman and Lie⁽¹³⁾ for the interpretation of image contrast produced in the field ion microscope.

The next stage in this work will be an attempt to assess the influence of a surface on the lattice distortion produced by an

SIA. This is a somewhat difficult problem because of the lack of knowledge of the effect of the surface on the inter-atomic interactions. As a first approximation, however, the surface may be simulated by "breaking" the atomic bonds above a given crystallographic plane and assuming the interatomic potential is unchanged from that in the bulk. To calculate the lattice relaxation in this situation we intend using a variational approach similar to that of Beeler⁽²⁶⁾.

G. On the Interpretation of Ledge "Bright Spot" Contrast Effects in Field Ion Microscope Images

A detailed pulse field evaporation study⁽²⁷⁾ was made of the field evaporation characteristics of atoms from the first five ledges of (011) planes of high purity ($\leq 1.5 \times 10^{-6}$ at.fr. impurity level) tungsten of well annealed specimens and specimens irradiated with 35 or 40keV W^+ ions at room temperature (middle of Stage II) in vacuua of $\sim 10^{-6}$ Torr. The detailed examination of $> 1.8 \times 10^4$ frames of ciné film of the well annealed control specimens and $> 2.6 \times 10^4$ frames of film of the irradiated specimens showed that the "bright spots" identified by Buswell⁽²⁸⁾ as vacancies decorated by surface contamination, were in reality normal tungsten atoms associated with the ledges of (011) planes. The identification of these "bright spots" as tungsten ledge atoms was achieved by employing a field evaporation increment of $\sim 2.5 \times 10^{-3}$ of an (011) plane per evaporation pulse. The concentrations of ledge "bright spots" in the control and the irradiated specimens were almost identical to one another. The vacancy concentrations, as measured in 7 different families of high index planes [$\{111\}$, $\{233\}$, $\{122\}$, $\{133\}$, $\{433\}$, $\{342\}$ and $\{532\}$ planes] changed by a factor of 10^2

as a result of the irradiation. Hence, it was concluded that Buswell's model for the "bright spots" in the ledges of (011) plane is incorrect. An example was given of the field evaporation of a small vacancy cluster in a (011) terrace plane which gave rise to a double ledge "bright spot" contrast effect. A hard sphere ball model of a cluster of three vacancies in an (011) terrace showed that these ledge "bright spots" were the by-product of the field evaporation process. Thus, this special type of contrast effect must be taken into account when analyzing ciné film of irradiated specimens. The existence of the phenomenon of ledge "bright spots" does not affect our prior interpretation of SIA's as also causing "bright spot" contrast, as the two effects are readily distinguishable from one another.

H. On the Question of the Possibility of an Equilibrium Array of Voids in a Material Subject to Irradiation

In a recent note Brown⁽²⁹⁾ presented a calculation from which he concludes that "an equilibrium array of voids may be a very general consequence of irradiation to produce a steady-state." This conclusion was refuted⁽³⁰⁾ since it was based on an erroneous application of equilibrium thermodynamics to a highly non-equilibrium situation. In particular, it was pointed out that the mathematical minimum which Brown calculates does not correspond to the minimum Gibbs free energy of the total system, since his procedure yielded a lattice containing concentration gradients. The presence of point defect concentration gradients corresponds to the presence of chemical potential gradients, and the basic definition of

thermodynamic equilibrium demands that for the whole system to be in equilibrium the chemical potential of each species must be the same throughout. Thus, the "equilibrium" properties of the void lattice which Brown calculates are without physical significance. The same conclusion was also reached by a simple kinetic argument. A brief discussion of some of the kinetic factors involved in reaching a steady-state situation were also discussed.

I. FIM Studies of Solute Atoms in Dilute Platinum Alloys

This program has several objectives. Firstly, it is providing us with experience in observing dilute distributions of solute atoms in the FIM. In this respect we are interested in the detection of individual solute atoms. For example, the type of imaging contrast that they produce will be of considerable interest. Secondly, from observations of the distributions of the solute atoms, we should be able to obtain valuable information about solute atom-solute atom interactions in the alloys. Physical properties of interest are short range order parameters and the concentrations of solute atom clusters of various sizes. Thirdly, the work should set the stage for eventual studies of the interactions between solute atoms and vacancy and SIA point defects. It should be noted that the Atom Probe FIM which is currently under construction (see II.M.) will also be of use in the study of solute atom-point defect interactions. It is hoped that these investigations will mutually complement one another.

So far, we have prepared the following alloys:

(Au-Pt	(0.6 at. % Au)
(Au-Pt	(4.0 at. % Au)
(Ni-Pt	(0.5 at. % Ni)
(Ni-Pt	(4.0 at. % Ni)
(W-Pt	(0.5 at. % W)
(W-Pt	(4.0 at. % W)

According to preliminary work by Dubroff and Machlin⁽³¹⁾ the gold and nickel atoms should produce "dark spot" contrast, whereas the tungsten atoms should produce "bright spot" contrast. Furthermore, thermodynamic data indicate that we should expect "clustering" in the Au-Pt solution and "ordering" in the Ni-Pt solution.

Specimens of the 0.6 and 4.0 at. % Au-Pt alloys have been dissected by the pulsed field evaporation technique, and it has been confirmed that the gold atoms do indeed produce "dark spot" contrast. However, a phenomenon is present which tends to complicate the situation. We have found that most of the dark spots are visible as soon as the atoms adjacent to them in the net planes associated with them become resolved during the pulsed field evaporation technique. These dark spots then remain visible until the planes in which they are embedded are removed by the field evaporation. The behavior of these dark spots is consistent with the assumption that they are vacancies which are left behind when gold atoms are removed by preferential field evaporation. The concentrations of these dark spots averaged over all {hkl} planes examined in each specimen correspond closely with the concentrations of gold atoms as determined by chemical analysis. For this reason we term these dark spots "intrinsic dark spots". However,

we have found that a number of extra dark spots appear suddenly in well-resolved net planes during observation. The number of newly-created dark spots corresponds to about 30% of the number of intrinsic dark spots. It seems possible that the "newly-created dark spots" may be platinum atoms which are suddenly removed from regions adjacent to gold atoms when the applied electric field becomes sufficiently high. This possibility will be investigated in the near future by carrying out detailed studies of the spatial distribution of both the intrinsic dark spots and the newly-created dark spots. This will be done using the semi-automated on-line computer technique described in Section II.L. With this technique the exact lattice position of each dark spot will be generated in our Nova computer and a computer analysis of correlation between the positions of the various dark spots will be carried out.

Corresponding studies of the Ni-Pt and W-Pt alloys are in progress.

J. Studies of the Structure of Low and High Angle Grain Boundaries

Several analyses of the structure of grain boundaries were carried out.

A discussion was given⁽³²⁾ of the extent to which all high angle grain boundaries can be described by the grain boundary dislocation (GBD) model which recently has been shown to apply to special boundaries having misorientations near high coincidence site density misorientations. In this model singular interfaces, which are dimensionally periodic on a fine scale, and which are of relatively low energy, are formed at special high coincidence site

density misorientations. Deviations from these misorientations are then compensated for by networks of discrete GBD's having Burgers vectors which are characteristic of the periodic structure of the singular interface. The structure therefore consists of a network of discrete GBD's embedded in the singular interface.

Particular attention was paid to the following two factors, both of which tend to increase the range of applicability of the GBD model, and both of which have been ignored previously in the published literature:

- (1) For each singular interface there is a hierarchy of GBD Burgers vectors of continuously increasing magnitude which possibly may be pressed into service in order to stabilize GBD type boundary structures.
- (2) There are considerable numbers of misorientations which give rise to near-coincidence situations in the boundary plane. A high coincidence site density situation can then be achieved over large patches of the boundary by introducing a suitable network of misfit GBD's. The result is a GBD type boundary which is quite similar to that which is achieved when the misorientation is close to an exact coincidence misorientation.

In further work⁽³³⁾ models for line defects in grain boundaries were considered. The structures of GBD's and ledges were examined, and line defects which correspond to either pure GBD's, pure grain boundary ledges or else defects possessing both dislocation and ledge character were distinguished. The concept of intrinsic grain boundary dislocations (IGBD's) was described, and circuits were defined which

yield the Burgers vector of both GBD's and IGBD's. Two distinct types of circuits were found to be useful and were presented. Virtual grain boundary dislocations (VGBD's) were also considered. What have been called VGBD's were shown in some cases to be true GBD's. In other cases, defects exist which could logically be termed VGBD's, but these can often alternatively be regarded as the result of the superposition of GBD's or IGBD's. GBD's formed in grain boundary cutting or impingement processes were found to be identical to secondary GBD's in boundaries of near-coincidence orientation. Generalized secondary GBD's were found to represent an intermediate representation between the Frank model and the surface dislocation model.

The recent analysis of grain boundary structure⁽³⁴⁾ carried out by Pumphrey, using the so-called plane matching (PM) method, was examined⁽³⁵⁾. The relationship between the PM approach and the GBD approach to grain boundary line structure observed in the electron microscope was shown explicitly. Various limitations of the PM approach were pointed out. It was demonstrated that the PM approach is actually not an independent "model" for the line structure but is merely another way of representing the line structures of certain boundaries which are already well accounted for by the more general GBD model.

In our experimental work we have continued to exploit the technique which we have developed earlier⁽³⁶⁾ for producing any type of planar grain boundary in thin-film bicrystal specimens of gold, copper or aluminum under controlled conditions. In this technique single crystal thin films of the desired orientations are

first grown epitaxially on appropriate substrates from the vapour phase. Two films, while still on their substrates, are then welded together face-to-face at any desired misorientation to produce a thin-film bicrystal containing a planar grain boundary at the welded interface. The substrates are then dissolved away to allow examination of the grain boundary at normal incidence by transmission electron microscopy. With the aid of this technique, work in the following areas was carried out:

- (1) Several near-coincidence type high angle boundaries were produced and were examined for GBD type structure.
- (2) Coincidence type boundaries following low index planes of the coincidence lattice were prepared and were examined for the presence of faceting. There is reason to believe that such boundaries should break up into small facets in order to reduce their energy.
- (3) Continued efforts were made to employ high resolution electron microscopy techniques in the examination of GBD structures in high angle grain boundaries.
- (4) Further efforts were made to use the extra electron diffraction spots arising from the periodic structure of grain boundaries⁽³⁷⁾ as a tool for the study of the fine structure of grain boundaries.

K. The Direct Measurement of the Range of a Focused Collision Replacement Sequence in Gold

The range of a focused collision replacement sequence (RCS) in gold has been established to be as small as $50\text{\AA}$ ⁽³⁸⁾ and as large as $1500\text{\AA}$ ⁽³⁹⁾. To try to settle this controversy an experiment has

been designed and the apparatus constructed to measure directly the range of an RCS as a function of temperature. The experiment consists of irradiating a thin gold foil of a known thickness with low energy (100 to 1000Vdc) argon ions and detecting the gold atoms that emerge from the back surface of the gold foil with a channel plate multiplier detector assembly.

At a given ion energy and specimen temperature the pattern of atoms that emerge from the back surface of the foil are observed visually with the aid of the channel plate multiplier detector (gain of $\sim 10^6$) and a phosphor screen. The range will be determined by simply examining the observed pattern as a function of foil thickness. At a given specimen temperature there will be a critical value of the foil thickness at which the intensity of this pattern will go to zero. This value of the foil thickness corresponds to the range of the RCS at this specimen temperature.

L. Automatization of FIM Data Processing

(i) General remarks regarding the Nova 1220 computer

Progress is well under way in integrating the Nova 1220 computer into our laboratory. We have been somewhat slowed by the failure of vendors to meet delivery dates and by the unavailability of many of the integrated circuits needed for building the various computer interfaces. We have, as a consequence, completed the construction of only one interface system, but all systems have been designed and software programming is currently being generated.

(ii) Atom Probe FIM

The atom probe-computer system has been built and will shortly be tested. Operation is as follows; (1) Computer initiates the field evaporation pulse and starts the high speed, multichannel

timer; (2) Timer signals computer of the completion of a cycle; (3) Computer reads all timing channels and two analog to digital converters (A/D) which monitor the dc and pulse voltage levels; (4) computer calculates the charge to mass ratio of the field evaporated species and compares this to a library of possible chemical species; (5) computer plots or prints out results for immediate identification.

(iii) Quenching and annealing of tungsten wires

The Nova 1220 in conjunction with a Data General high speed A/D converter system will provide an extremely sophisticated system for the introduction and analysis of thermally induced defects in tungsten. This system will completely control and record the thermal history of fine tungsten wires with an accuracy of $\pm 1^\circ\text{K}$. It will also provide complete data reduction and on-line plotting of the temperature versus time curve for each specimen. This system has been designed and we are currently waiting for delivery of the needed components.

(iv) Analysis of recorded images

The computer will allow the rapid analysis of films of FIM specimens by instantaneously digitizing the coordinates of an atom on the screen of the Vanguard motion analyzer, recording it, calculating its real position and immediately plotting its location on a point plotter. This system is designed and we are waiting for components. Future plans include purchasing an Instronics digitizer which uses a touch pen system to speed data acquisition even further.

M. The Atom Probe Field Ion Microscope

The basic atom probe FIM has been constructed and interfaced to the Nova computer. The microscope that was designed has a number of novel features which make it particularly well suited for the study of point defects, point defect clusters, point defect-line defect interactions and point defect-planar defect interactions. These features are as follows:

- (1) An ultra-high vacuum FIM.
- (2) A two-axis goniometer stage with a tilt of $\pm 45^\circ$ in two orthogonal directions. The tilt angles can be readily increased to $\pm 90^\circ$ if this is found necessary.
- (3) Continuously variable liquid helium cooling of the specimen holder between $\sim 6^\circ\text{K}$ and room temperature.
- (4) A Bendix micro-channel plate for internal image intensification of the FIM pattern. The tip to micro-channel plate distance is continuously variable over a factor of about 4 in linear distance, hence the area magnification of the image size is variable by a factor of about 16.
- (5) An electronic readout device⁽⁴⁰⁾ for measuring the flight times of up to eight atoms per field evaporation pulse. The measured flight times will be displayed in digital form, and we have also made provision to divert the flight times to a small computer for further data processing.
- (6) A specimen exchange device which will enable us to change specimens under ultra-high vacuum conditions.

III. LIST OF DOCUMENTS GENERATED DURING THE CONTRACT PERIOD

1. "A Quantitative Study of Vacancy Defects in Quenched Platinum by Field Ion Microscopy and Electrical Resistivity: II. Analysis", D. N. Seidman, A. S. Berger and R. W. Balluffi, COO-3158-5
2. "On the Structure of High Angle Grain Boundaries with Particular Reference to a Recent Plane Matching Approach", R. W. Balluffi and T. Schober, COO-3158-6
3. A Critique of L. M. Brown's "A Simple Explanation of Voids in Materials under Irradiation", D. N. Seidman and R. W. Balluffi, COO-3158-7
4. "On Some Problems of the Detection of Self-Interstitial Atoms in Field Ion Microscope Images", J. T. Robinson, K. L. Wilson and D. N. Seidman, COO-3158-8
5. "The Direct Observation of Point Defects in Irradiated or Quenched Metals by Quantitative Field Ion Microscopy", D. N. Seidman, COO-3158-9
6. "Comments on the Range of Applicability of the Grain Boundary (Secondary) Dislocation Model to High Angle Grain Boundaries", R. W. Balluffi and T. Tan, COO-3158-10
7. "Calculation of Correlation Effects for Grain Boundary and Dislocation Pipe Self-Diffusion", J. T. Robinson and N. L. Peterson, COO-3158-11
8. "On Grain Boundary Dislocations and Ledges", J. P. Hirth and R. W. Balluffi, COO-3158-12
9. "Binding Energies and Entropies of Solute Atoms and Vacancies in Metals", R. W. Balluffi and P. S. Ho, COO-3158-13
10. "A 10 Nsec Resolution Counter for Multiparticle Atom Probe Time-of-Flight Measurements", A. S. Berger, COO-3158-14
11. "A Modified Green's Function Technique for the Calculation of Lattice Distortion Surrounding Interstitials", Roy Benedek and P. S. Ho, COO-3158-15
12. "Application of Green's Function Technique to Self-Interstitial Atoms in Tungsten", Roy Benedek and P. S. Ho, COO-3158-16
13. Annual Progress Report, "Defects in Metal Crystals", R. W. Balluffi and D. N. Seidman, COO-3158-17

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