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THE DIRECT DETERMINATION OF RADIATION
DAMAGE PROFILES IN THE ORDER-DISORDER
ALLOYS Ni_4Mo AND Pt_3Co IRRADIATED
WITH 500 to 2500 eV Ne^+ Ions

by

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 Pt_3Co IRRADIATED WITH 500 TO 2500 eV Ne^+ IONS

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Radiation damage produced in the order-disorder alloys Ni_4Mo and Pt_3Co by low-energy (500-2500 eV) Ne^+ ions has been observed directly, on an atomic scale, employing the field-ion microscope (FIM) technique. A new approach was developed to determine the radiation damage distribution in these alloys. In the case of the alloy Ni_4Mo the measurements were based on the contrast differences between the FIM images of an ordered alloy, which were completely regular, and a disordered alloy which appeared as a random array of image points. FIM specimens of the ordered alloy Ni_4Mo were irradiated in situ at 30 K, and the side of the specimen exposed to the beam became disordered as a result of the implantation of the neon atoms. The change from an ordered state to a disordered state was detected by the controlled dissection of the specimen, on an atom-by-atom basis, employing the pulse field-evaporation technique. The amount of disordered metal which was field-evaporated, to expose the perfectly ordered lattice, and some basic geometric relations gave the depth of radiation damage: the depth resolution was $\approx 2 \text{ \AA}$. A crystallographic directional dependence of the mean depth of radiation damage ($\bar{x}_d$) was observed and was attributed to the effect of channeling. For the non-channeling direction the value of $\bar{x}_d$ varied from ≈ 6 to $\approx 19 \text{ \AA}$ as the irradiation energy was increased from

500 to 2000 eV. The experimental $\bar{x}_d$ versus Ne^+ ion energy relation agreed with the theoretical predictions of Winterbon, Sigmund and Sanders, and the results of the Monte Carlo computer simulation program, of Biersack and Haggmark, called TRIM.

The determination of radiation damage profiles in the alloy Pt_3Co were based on the ability of the FIM technique to determine the local value of the Bragg-Williams long-range order parameter (S). Ordered Pt_3Co FIM specimens were irradiated in situ at 30 K; the implanted Ne^+ ions disordered the lattice and reduced the local value of S from its pre-irradiation value (S_0) to a value S_d which was a function of the distance (x) from the irradiated surface. The change in S , i.e., ΔS_d , was determined by measuring the value of S_d in each field-evaporated plane. The measurement of ΔS_d versus x constituted a radiation damage profile: the depth resolution of this profile was $\approx 2 \text{ \AA}$. All of the irradiations were parallel to high-index crystallographic directions except for one case where the beam was parallel to the $[110]$ direction. For the $[110]$ case the radiation damage profile extended to a greater depth than the profiles obtained for the other directions. This result was taken to be indicative of channeling in the $[110]$ direction. For the irradiations which were parallel to high-index directions the value of $\bar{x}_d$ varied from ≈ 9 to $\approx 28 \text{ \AA}$ and the straggling (Δx_d) varied from ≈ 6 to $\approx 18 \text{ \AA}$ as the irradiation energy was increased from 500 to 2500 eV. In general there was first order agreement between our experimental results and the predictions of Winterbon, Sigmund and Sanders, and Biersack and Haggmark models, although there were some quantitative differences in the values of the higher moments.

Isochronal recovery experiments were performed on both alloys after they had been irradiated in situ at 6 K with either 30 or 60 keV Kr⁺ or Xe⁺ ions. The results indicated that in both cases there was no long-range migration of self-interstitial atoms (SIAs) between 6 and 30 K. This assured us that the distribution of the radiation damage which was measured at 30 K was not affected by the long-range migration of SIAs. The isochronal annealing spectrum of irradiated Pt₃Co specimens was broader and slightly shifted to higher temperature, when compared with the annealing spectrum of electron-irradiated pure platinum.

1. INTRODUCTION

The precise determination of the distribution of radiation damage in solids irradiated with low-energy ions is related to problems concerning the first wall of plasma devices and of future fusion reactors,^(1,2) and has application in semiconductor device technology.⁽³⁾ This distribution, which represents the elastically deposited energy, is of primary interest in the modeling of radiation damage production. Experiments in the low-energy régime are especially valuable for testing the validity of the existing theories of stopping powers.⁽⁴⁻⁶⁾ However, it is extremely difficult to measure the distribution of radiation damage caused by low-energy ions, i.e., where the reduced energy parameter (ϵ)⁽⁶⁾ is less than 0.1. Although the range distribution of implanted ions has been obtained for values of ϵ as low as 0.0006⁽⁷⁾ employing the Rutherford backscattering (RBS) technique, radiation damage distribution data for $\epsilon < 0.1$ is scarce.^(8,9) The field-ion microscope (FIM) with its ability to resolve and to locate individual atoms in direct lattice space offers a unique approach for measuring the distribution of radiation damage in ordered alloys.

When an ordered alloy is irradiated it becomes disordered, i.e., the value of the Bragg-Williams long-range order-parameter (S) of the alloy decreases. The change in the value of S is caused by both atom displacement⁽¹⁰⁾ and replacement⁽¹⁰⁻¹²⁾ events. The replacement events create atoms that occupy the wrong sites in an ordered lattice, and the displacement events create vacancies and self-interstitial atoms (SIAs). The irradiation-produced vacancies and SIAs (Frenkel pairs) can enhance diffusion, and thus enhance the ordering kinetics of the alloy.⁽¹³⁾ The extent to which an ordered alloy will become disordered during an

irradiation depends upon the flux and the energy of the incident ions and the temperature of the target. There are still unanswered questions as to the temperature régimes in which the different point defects, i.e., vacancies and SIAs, are mobile and can contribute to the ordering process. A careful annealing treatment followed by an irradiation at temperatures where all the defects are immobile, can yield information on the relative mobilities of the point defects. Moreover, the use of an ordered alloy, as a target, combined with the unique capability of the FIM technique to distinguish between the different atoms in an alloy, enabled us to differentiate between displacement and replacement events.

1.1 Depth Distribution of Radiation Damage

When a fast charged particle (projectile) penetrates a solid it interacts with the constituents of the matter, i.e., with the target nuclei and with the target electrons. The projectile loses energy along its path in exciting the target electrons (inelastic process) and in transferring momentum (elastic process) to the target nuclei during collisions. When the projectile's velocity is greater than the orbital velocity of the target electrons, inelastic losses to the electronic system dominates. The projectile proceeds to interact with the target electrons until it reaches a velocity which is smaller than the orbital velocity of the least tightly bound target electron. At this stage it starts to interact with the target atoms via screened Coulomb fields. Finally the projectile comes to rest solely due to nuclear collisions. The different energy loss mechanisms, i.e., electronic and nuclear, are generally treated separately and are assumed⁽⁴⁻⁶⁾ to contribute additively to the total stopping power $\left[\left(-\frac{dE}{dx} \right)_{pl} \right]_T$ which is the differential energy

loss, thus

$$(-dE/dx_{pl})_{Total} = (-dE/dx_{pl})_{nuclear} + (-dE/dx_{pl})_{electronic} \quad (1)$$

where E is the energy of the projectile, and x_{pl} is the total path length of the projectile.

The inelastic energy loss to the target electronic system can cause structural damage in both insulators and semiconductors,⁽¹⁴⁾ whereas in the case of metals this energy loss is largely dissipated as heat without the creation of structural defects.⁽⁵⁾ In metals, the exclusive process leading to point-defect production is a result of nuclear collisions in which elastic energy is transferred to the atoms of the target material and displaces them if the transferred energy is greater than a minimum threshold displacement energy (E_d). Therefore, the depth distribution of the elastically deposited energy is of primary importance to this study.

I.1.1. Stopping Powers

The nuclear stopping power depends on the cumulative effect of a succession of independent elastic scattering events between the projectile and the atoms of the target. Theoretical approaches^(4,15) for predicting stopping powers are based on solving the classical scattering equation⁽¹⁶⁾ using different interatomic potentials. The rate of energy loss due to nuclear collisions can be written in terms of the differential scattering cross section $d\sigma_n(E,T)$ as:⁽⁴⁾

$$(-dE/dx_{pl})_n = N \int_0^{T_{max}} T d\sigma_n(E,T) dT \quad (2)$$

where N is the number of scattering centers per unit volume, T is the energy transferred in a collision, and T_{max} is the maximum transferable

energy in a collision at energy E . The analytical form of the nuclear stopping power depends on the form of the interatomic potential used in eqn. (2). Several potentials have been used in deriving the nuclear stopping power. Amongst the most heavily used interatomic potentials are the Molière⁽¹⁷⁾ and the Firsov⁽¹⁸⁾ approximations to the Thomas-Fermi potential. Each of these potentials is expressed as a Coulombic ($1/r$) term multiplied by a screening function, where r is the distance between the charge centers.

The rate of energy loss due to electronic collisions can also be written in terms of a differential scattering cross section $d\sigma_e(E,T)$, but because the different electrons in the solid contribute differently to the scattering process, an analytical form which covers all energies has yet to be formulated. However, for the low-energy régime ($\epsilon < 1$), models^(19,20) based on particles moving through a degenerate electron gas suggested that the electronic stopping power was proportional to the particle velocity. Also, based on a Thomas-Fermi statistical model, Firsov⁽²¹⁾ calculated the impact parameter dependence of the electronic stopping power and obtained an expression that was proportional to the particle velocity, as was also found by Robinson and Oen.⁽²²⁾

We want to emphasize that the theoretical stopping power describes the energy a projectile loses along its total path length. Experimentally the most significant quantity which can be measured is the projectile's projected range (x_{pr}). However, because the atomic scattering introduces deflections, the values of x_{pr} and x_{pl} will not necessarily be equal. Figure 1⁽²³⁾ illustrates the relation between the different types of range:

Figure 1: Illustration of the relationship between the total path length (x_{pl}), the vector range (x_v) and the projected range (x_{pr}). (Taken from Nelson⁽²³⁾).

1. The total path length (x_{pl}), which is the total path length traversed by the projectile.
2. The vector range (x_v), which is the vector between the starting point and the stopping point.
3. The projected range (x_{pr}), which is the normal projection of the vector range on the starting direction, and is therefore a measure of the penetration of a projectile along its initial direction.

Thus the stopping power, which is related to x_{pl} , cannot necessarily be directly compared with the experimental range determinations. For light projectiles with very high energy where the stopping power is almost exclusively electronic, the deviations from a straight path are small and $x_{pr} \approx x_{pl}$. On the other hand, for slower and heavier projectiles where large angle scattering events are more probable, especially for $M_1 < M_2$, where M_1 is the projectile's mass and M_2 is the mass of the target atom, the value x_{pr} can be much smaller than x_{pl} .⁽²³⁾

I.1.2 Theoretical Predictions of Elastically Deposited Energy Distribution

The two main calculational techniques used to obtain the spatial distribution of ranges and deposited elastic energy by the incident ions are the Monte Carlo^(24,25) method and the solution to the transport equations.^(6,26) Both techniques were used to calculate the distribution of energy deposited in atomic processes (atomic motion and displacements), and the resulting distributions were compared with our experimental data.

In the transport equation approach^(6,26) it was assumed that the distributions can be represented by a continuously smooth function. The target was assumed to be amorphous, infinite and uniform. The surface was

set at the position where atom motion started. The Lindhard, Scharff and Schiøtt (LSS)⁽⁶⁾ formulation approached the solution to the transport equation by taking the spatial moments of the distributions and thus reducing the number of variables in the equations. The equations for the moments were solved and then the distribution was reconstructed from the moments.

The LSS formulation estimated the energy that fast ions lost by elastic collisions in an amorphous solid employing a Thomas-Fermi potential. A universal range versus energy curve was obtained in terms of the dimensionless reduced range (ρ) and energy (ϵ) parameters:

$$\rho = \frac{R n N a^2 M_1 M_2}{(M_1 + M_2)^2} \quad , \quad (3)$$

and

$$\epsilon = \frac{E a M_2}{Z_1 Z_2 e^2 (M_1 + M_2)} \quad , \quad (4)$$

where R is the range, Z_1 and Z_2 are the nuclear charges of the projectile ion and the target atom, respectively, and e is the charge on an electron. The quantity a is the screening radius given by

$$a = 0.8853 a_0 (Z_1^{2/3} + Z_2^{2/3})^{-1/2} \quad ; \quad (5)$$

where a_0 is the Bohr radius of the hydrogen atom (0.529 Å). The contribution of inelastic collisions was included by adding to the elastic stopping power the electronic stopping power which is proportional to the

velocity of the projectile.⁽¹⁹⁾ In the dimensionless units it has the form

$$S_e = \left(\frac{d\epsilon}{d\rho} \right)_e = k\epsilon^{1/2}, \quad (6)$$

where the electronic stopping power constant k was

$$k = \frac{0.0793 Z_1^{2/3} Z_2^{2/3} (M_1 + M_2)^{3/2}}{(Z_1^{2/3} + Z_2^{2/3})^{3/4} M_1^{2/3} M_2^{1/2}}. \quad (7)$$

Figure 2 shows the stopping powers as a function of energy, plotted in LSS-reduced units. Also shown in the figure are some typical values of ϵ and k for 1 keV projectile ions (Z_1) incident on different target materials (Z_2). The electronic stopping is usually a minor correction for $\epsilon < 1$, unless $Z_1 \leq 10Z_2$, when it may not be neglected.⁽⁶⁾ In the present work we report on irradiations in the energy régime where ϵ ranges from 0.005 to 0.05. Therefore, the nuclear stopping was the dominant mechanism as can be seen in Fig. 2.

Winterbon, Sigmund and Sanders (WSS)⁽¹⁵⁾ used the LSS formalism to obtain the projectile's total path length as a function of energy, for ϵ smaller than 0.2, by integrating the nuclear stopping power

$$S_n = \left(d\epsilon/d\rho_{pl} \right)_n = 0.98\epsilon^{1/3}, \quad (8)$$

they obtained:

$$\rho_{pl}(\epsilon) = 1.53\epsilon^{2/3}, \quad (9)$$

Figure 2: Universal nuclear and electronic stopping powers as a function of energy plotted in LSS-units.

where p_{pl} is the LSS dimensionless total path length. They used the nuclear stopping power in the integro-differential equation they derived for determining the distribution of the elastically deposited energy in amorphous targets. Winterbon⁽²⁶⁾ using the WSS approach has solved a set of transport equations and tabulated the values of the first four moments of the elastically deposited energy for a large number of projectile-target combinations as a function of energy.

There are two important simplifications in the transport theory:

(1) the transport equations were formulated for an infinite medium, and do not include the surface effect, i.e., in the calculations, particles can be scattered back and forth across the surface whereas physically, once they leave the medium they do not repenetrate the medium; (2) the finite value of E_d , i.e., the minimum energy needed to displace an atom permanently from its site, was not taken into account. Also, the use of Thomas-Fermi potential in the WSS formalism is inappropriate at energies below $\epsilon = 10^{-2}$.^(4,27) This potential overestimates the strength of the interaction between atoms at large separation (i.e., low energies). Thus, the nuclear stopping power is too high and the predicted ranges are too short.

The Monte Carlo technique^(24,25) follows a single ion as it penetrates the target and adds up the various quantities of interest, i.e., ranges and energy deposited, along the ion's path. The complete histories of the trajectories are obtained for a large number (typically two to three thousand) of incident ions, and the statistical average of the distributions is taken over these histories. Recently, an efficient Monte Carlo code (TRIM)⁽²⁵⁾ has been developed to simulate transport of energetic ions in amorphous targets. This program used a three-parameter

expression for the nuclear stopping power which was derived by Wilson, Haggmark and Biersack (WHB)⁽⁴⁾

$$S_n = \frac{A \ln(B\varepsilon)}{B\varepsilon - B\varepsilon^{-C}}, \quad (10)$$

where A, B and C were tabulated constants for different interatomic potentials. In the TRIM program the Molière interatomic potential was used, and the projectile was assumed to change direction as a result of a series of elastic collisions; its total energy was reduced as a result of both nuclear and electronic losses. The TRIM program accounted for the surface effect, i.e., once a particle left the medium it did not return. This program provided both an ion range profile and the distribution of the elastically deposited energy.

1.1.3. Previous Experimental Techniques and Results

Experimental data on the depth distribution of radiation damage in the low-energy régime, where ε is less than 0.1, has been limited⁽²⁸⁾ and not precise enough^(8,9) to determine an experimental damage range-energy relationship to compare with theory. The experimental techniques were based on: (1) the dependence of the sputtering on the amount of damage in the target; (2) the change in the Rutherford backscattering (RBS) yield in a channeling direction; and (3) the detection of defect clusters in transmission electron microscope (TEM) images. In the following, each technique will be described and its results in the energy régime where ε is less than 0.1 will be discussed.

MacDonald and Haneman⁽⁸⁾ have developed a method which was based on the differences in sputtering yield, for a given energy, between

irradiated and unirradiated material. They measured the sputtering yield of Ge, from specimens which were pre-bombarded with 400-100 eV Ar^+ ions ($3.5 \cdot 10^{-3} < \epsilon < 9 \cdot 10^{-3}$), as a function of the sputtered layer thickness. Typical pre-bombarded doses were of the order of 10^{16} to 10^{17} ion cm^{-2} . With these doses, the calculated dopant concentration within the penetration depth ($\sim 40 \text{ \AA}$) was of the order of one dopant atom (Ar) per target atom. Therefore, a distinction between range and damage distribution, using this technique, does not appear feasible. The peaks of the measured sputtering yield versus depth curves were in agreement with the LSS predictions of the mean projected range ($\bar{x}_{\text{pr}}$).

An unfortunate drawback of employing TEM to measure radiation damage distribution is that the yield factor (Y), where

$$Y = \frac{\text{observable vacancy contrast-effects (number cm}^{-2}\text{)}}{\text{ion dose (number of ions cm}^{-2}\text{)}}$$

measured by the TEM technique, is often considerably less than unity.⁽²⁹⁻³²⁾ The observation of radiation damage distribution by TEM depends heavily on the ability of the vacancy-rich core of a depleted zone (DZ) to collapse into dislocation loops which are observable in the TEM. English et al.⁽³⁰⁾ irradiated α -iron at room temperature with various ions in the energy range 40 to 240 keV and found that the defect yield was a strong function of the mass of the incident ions, increasing from zero for Fe^+ ions to $\sim 20\%$ for W^+ ions. Thus, a radiation damage distribution study with TEM cannot yield quantitative information.

Diehl et al.⁽⁹⁾ measured the depth distribution of small agglomerates in copper and gold foils which were bombarded with 1-5 keV Ar^+ ions ($0.004 < \epsilon < 0.05$). They determined that the majority of the observable agglomerates consisted of SIA-type defects which were in the form of dislocation loops. The average depth of these dislocation loops was approximately ten times greater than the depth predicted by the WSS approach. Based on their interpretation for the contrast effects, i.e., SIA-type dislocation loops, and on geometric considerations they attributed the difference in the depth values to the penetration of the SIAs into the lattice by means of focused replacement collision sequences (RCS). In contrast to this interpretation, Thomas and Balluffi⁽³³⁾ found that SIA loops were nucleated extensively in ion-irradiated gold only when impurities were present on the surface. Also Norris⁽²⁸⁾ studied the radiation damage distribution in nickel and gold foils irradiated with 80-150 keV Hg^+ and Au^+ ions ($0.1 < \epsilon < 0.03$). He concluded that the observed contrast effects consisted of vacancy-type defects. The value of Y was about one, but only approximately 20% of these defects were imaged sufficiently strong and well-defined to permit the measurements. The ratio of his results for the median cluster depth to the mean elastically deposited energy depth predicted by the WSS approach was 0.75 when the target was nickel and 1.5 when the target was gold.

The use of the RBS technique^(34,35) combined with channeling can provide information on lattice disorder⁽³⁶⁾ and atom location⁽³⁷⁾ in a single crystal target. The RBS technique is a useful method for analyzing the range profiles of implanted atoms which are heavier than the target atoms. In this method a monoenergetic beam of light ions,

i.e., ^4He at typically 1.5 MeV, is fired at a target, and the energy distribution of the backscattered ions at a given angle is measured. The energy distribution contains information on the mass and distribution of atoms in the scattering layers. In an undamaged crystal channeled ions, i.e., ions guided by the lattice rows, become dechanneled only due to collisions with the electrons and the thermally vibrating row atoms. When damage is introduced in the target dechanneling increases, and the number of backscattered ions is increased accordingly. Thus, the use of the RBS technique combined with channeling can provide the damage distribution caused by ions penetrating into single crystals. However, in metals the vacancies and SIAs which are created during an irradiation generally agglomerate into large dislocation loops with large stress field around them. Thus strain is generated which may extend further than the range of the damaging ions. Therefore, the radiation damage distribution obtained from the RBS technique combined with channeling has to be supplemented by other methods such as TEM.⁽³⁸⁾ Recently, Picraux et al.⁽³⁹⁾ used these techniques to determine the radiation damage distribution in silicon and aluminum at the reduced energy $\epsilon = 1.1$. The depth resolution of the RBS technique is typically 30-50 Å,⁽¹⁰⁾ which is the average depth of damage value in the low-energy region, i.e., $\epsilon \leq 10^{-2}$. Therefore, as yet these techniques have not been used to measure damage distribution in this low-energy régime.

However, the development of the RBS technique^(34,35) has enabled the measurements of x_{pr} for heavy ions incident into light targets with ϵ values between 0.0006 and 10. Kalbitzer et al.⁽⁴⁰⁾ (DKGF) derived an experimental nuclear stopping power by fitting their experimental data

to a power law expression

$$\left(\frac{d\epsilon}{d\bar{\rho}_{pr}} \right) = \frac{1.7\epsilon^{1/2} \ln(\epsilon + e)}{1 + 6.8\epsilon + 3.4\epsilon^{3/2}}, \quad (11)$$

where $\bar{\rho}_{pr}$ is the dimensionless mean projected range. In the case of a heavy projectile penetrating a light target $x_{pr} \approx x_{pl}$.

1.2. Effects of Radiation on Ordered Alloys

Particle radiation can have both the effect of ordering a disordered alloy and of disordering an ordered alloy. The extent to which an ordered alloy will be disordered during an irradiation depends upon the flux, the energy and the temperature of the irradiation.⁽⁴¹⁾ In particular, at elevated temperatures, but below the critical ordering temperature, thermal ordering of the disordered lattice can occur when there is appreciable migration of vacancies and SIAs which are produced continuously during the irradiation. In this case, there are competing effects between the disordering caused by the irradiation and the ordering resulting from migration of point defects. At very low temperatures where the point defects are barely mobile or do not migrate only disordering take place.

Disordering has been ascribed to three different mechanisms: (1) thermal spike;⁽⁴²⁾ (2) displacement spike;^(43,44) and (3) RCSs.^(11,16) In the thermal spike mechanism the energy is localized first on a single atom, the primary knock-on atom (PKA), which generates high energy secondary collisions that are completed about 10^{-13} sec after the creation of the PKA. This leaves a highly damaged region with an excess energy that can only be dissipated by lattice phonons which give rise to a local

heating. The effect of the thermal spike on its surrounding depends both on the spike temperature (T_p) and its lifetime (τ_p). The length of τ_p depends on the efficiency of heat conduction to the surrounding lattice. During this time the high temperature may allow diffusion to occur which disorders the lattice.

The displacement spike occurs in the lattice when the energy of the PKA falls to a value such that it displaces almost every atom it meets. The displacement spike consists of a high vacancy concentration core surrounded by SIAs. This mechanism is known to operate in pure metals.^(45,46)

In the RCS disordering mechanism, a collision sequence proceeds along a line of lattice atoms which in the ordered lattice has a prescribed sequential arrangement. Each atom successively replaces the atom ahead of it and thus disorders the alloy.

The first attempt to verify the existence of disordered zones was reported by Piercy,⁽⁴⁷⁾ who studied the magnetic properties of an ordered Pt_3Fe alloy after irradiation with fast neutrons. The ordered alloy was nonmagnetic while the disordered phase was ferromagnetic. He correlated the change in the magnetic properties to the size of the disordered zones and concluded that the thermal spike lasted a long enough time to allow diffusion to cause all of the observed disordering. From the variation in the Curie temperature he argued that there were regions of partial disorder surrounding each completely disordered region. Recently, disordered zones were observed in ion bombarded ordered Cu_3Au ^(48,49) and Zr_3Al ⁽⁵⁰⁾ alloys, employing the TEM technique.

To estimate T_p and τ_p the spike was approximated⁽¹⁶⁾ to be a spherical region with radius (r_p) where the energy distribution is

described by a Maxwell-Boltzman function: in this function the temperature appears as a parameter. The region was assumed to cool with a characteristic relaxation time of the order of $\tau_p r_p^2$, where $\tau_p = cd/4K$; c being the specific heat, d the mass density and K the thermal conductivity.

Jenkins et al.⁽⁴⁹⁾ estimated the values of τ_p and T_p for their experimental conditions. For 10 keV Cu^+ ions incident on ordered Cu_3Au $T_p \approx 7 \cdot 10^4 \text{K}$ and $\tau_p \approx 3.5 \cdot 10^{-13} \text{ sec}$, and for 100 keV Cu^+ ions $T_p \approx 7 \cdot 10^3 \text{K}$ and $\tau_p \approx 2.4 \cdot 10^{-11} \text{ sec}$. The duration of these spikes, within the validity of the Arrhenius equation at these very high temperatures, was sufficiently long to account for disordering by thermally activated diffusion. It is not clear, however, to what extent the concept of a spike is valid, particularly at such a high temperature. Nevertheless, the thermal spike might constitute an important disordering mechanism, particularly in the central region of the displacement cascades where the local spike temperature is the highest. However, the thermal spike mechanism was unable to explain the disordering of the long, thin extrusions that Jenkins et al. observed at high ($>30 \text{ keV}$) irradiation energies. They attributed these extrusions of disordered zones to a large number of relatively short RCSs and to quasi-channelled knock-ons.

The RCS disordering mechanism was proposed by Kinchin and Pease⁽¹¹⁾ and later also appeared in the many-body computer simulation model of low-energy collision events in Cu_3Au ⁽⁵¹⁾ and Fe_3Al ⁽⁵²⁾. Much indirect and model dependent evidence for the operation of this mechanism was reported in the literature. These reports were from experiments which used different alloy systems, different measurement techniques, different irradiation species and different energies. The n_r/n_d ratios, where n_r

is the number of atomic replacements and n_d is the number of atomic displacements, reported in the literature varied from $2.2 \pm 2.2^{(53)}$ to $132 \pm 10^{(54)}$

To summarize, no clear dominant mechanism was found to be responsible for the disordering of an ordered alloy. It is feasible that these three mechanisms operate simultaneously.

I.3 FIM Studies of Ordered Alloys

The FIM images of disordered alloys are nearly structureless, whereas the images of ordered alloys show a regular appearance comparable to that of pure metals. The first regular FIM images from ordered PtCo alloy were recorded by Muller⁽⁵⁵⁾ in 1962. Since then, many ordered alloys with different crystal structures have been studied with the FIM. A summary listing of these alloys can be found in two review articles by Lefèvre⁽⁵⁶⁾ and Ranganathan and Suryanarayana.⁽⁵⁷⁾

Southworth and Ralph⁽⁵⁸⁾ imaged ordered PtCo, which consists of alternate layers of Pt and Co atoms on the (002) planes. They found that the number of rings in the vicinity of the 002 pole was only half of that expected from the tip radius, calculated from the knowledge of the applied tip voltage and the ionization field required to form an image. They concluded that only one species was imaged. Tsong⁽⁵⁹⁾ suggested that the selective field-ionization was the mechanism responsible for the absence of one species in the FIM image. He argued that the mechanism of charge transfer was responsible for the image of the Pt atoms in the PtCo alloy. When the ionization potential of one atom in an alloy is higher than that of the other, it accumulates electrons. When the field is applied, the positive field above it is too low to give an image. He also pointed out that in the case of ordered Ni_4Mo alloy,⁽⁵⁶⁾ where the

Mo atoms are the imaging species, the authors showed pictures where the Ni atoms imaged very weakly. Thus he refuted the suggestion that the selective field-evaporation mechanism was responsible for the absence of one species in the FIM image of order alloy. Finally, experimental work⁽⁶⁰⁾ on ordered Pt_3Co alloy with the FIM atom probe showed that the selective field ionization was the operating mechanism.

I.4. Prospectus of the Thesis

Radiation damage produced in the order-disorder alloys Ni_4Mo and Pt_3Co by low-energy (500-2500) eV Ne^+ ions have been directly observed employing the FIM technique. Chapter III describes the determination of the depth of radiation damage in the alloy Ni_4Mo . The measurements were based on the contrast differences between the FIM images of an ordered alloy, which were completely regular, and a disordered alloy which appeared as a random array of image points. FIM specimens of the ordered alloy Ni_4Mo were bombarded in situ at 30 K, and the bombarded side of the specimen became disordered. The change from an ordered state to a disordered state was detected by the controlled dissection of the specimen on an atom-by-atom basis, employing the pulse field-evaporation technique. The amount of disordered metal evaporated to expose the perfectly ordered specimen, and the basic geometric relations for an FIM specimen, gave the depth of radiation damage. The depth resolution was of the order of $2 \text{ \AA}^0$. Three sets of irradiation experiments were performed. In each set the bombardment direction was parallel to a different crystallographic direction. A directional dependence of the mean depth of radiation damage ($\bar{x}_d$) was observed and was attributed to the effect of channeling. For the non-channeling direction the value of $\bar{x}_d$ varied from ≈ 6 to $\approx 19 \text{ \AA}^0$.

as the implantation energy was increased from 500 to 2000 eV.* The $\bar{\rho}_d$ versus ϵ expression obtained from the experimental results was

$$\bar{\rho}_d = (0.42 \pm 0.04)\epsilon^{0.68 \pm 0.04} \quad (12)$$

where $\bar{\rho}_d$ was the mean depth of radiation damage in LSS dimensionless units. The effective stopping power $[(d\epsilon/d\bar{\rho}_d)_{\text{eff}}]$ of Ne^+ in Ni_4Mo was

$$(d\epsilon/d\bar{\rho}_d)_{\text{eff}} = (3.50 \pm 0.4)\epsilon^{0.32} \quad (13)$$

We have used the term effective in connection with $(d\epsilon/d\bar{\rho}_d)_{\text{eff}}$ to distinguish between this stopping power and the theoretical stopping power which is related to ρ_{pl} . These relations were in a good agreement with the predictions of the WSS⁽¹⁵⁾ and Biersack and Haggmark⁽²⁵⁾ models.

Chapter IV describes the determination of radiation damage profiles in the alloy Pt_3Co . The measurements were based on the ability of the FIM technique to determine the local value of S . Ordered Pt_3Co FIM specimens were irradiated in situ at 30 K, the penetrating Ne^+ ions disordered the lattice and reduced the local value of S from its pre-irradiation value (S_0) to a value S_d which was a function of distance (x) from the irradiated surface along the bombardment direction. The change in S , i.e., ΔS_d , was detected by measuring the value of S_d in each field evaporated plane. The measurement of ΔS_d versus x constituted a radiation damage profile; the depth resolution of this profile was $\sim 2 \text{ \AA}$. Most

* The energy regime $500 \leq \epsilon \leq 2000 \text{ eV}$ Ne^+ ions penetrating into Ni_4Mo corresponds to $0.01 \leq \epsilon \leq 0.04$.

of the irradiations were parallel to high index crystallographic directions except in one case where the ion beam was parallel to the [110] direction. In this case the radiation damage profile extended to a greater depth than the profiles obtained for the other directions. This result was taken to be indicative of channeling in the [110] direction. For the irradiations which were parallel to high index direction the value of $\bar{x}_d$ varied from ≈ 9 to $\approx 28 \text{ \AA}$ and the straggling (Δx_d) varied from ≈ 6 to $\approx 18 \text{ \AA}$ as the implantation energy was increased from 500 to 2500 eV.[†] The expressions for $\bar{p}_d$ and $(d\epsilon/d\bar{p}_d)_{\text{eff}}$ obtained from the experimental results were:

$$\bar{p}_d = (0.29 \pm 0.04)\epsilon^{0.67 \pm 0.02}, \quad (14)$$

and

$$(d\epsilon/d\bar{p}_d)_{\text{eff}} = (5.15 \pm 0.5)\epsilon^{0.33 \pm 0.02}.$$

In general there was a first order agreement between our experimental results and the prediction of the WSS and Bierack and Haggmark models; although there were some quantitative differences in the higher moments of the profiles.

Chapter V describes the post-irradiation isochronal recovery experiments we performed on both alloys after they had been irradiated in situ at 6 K with either 30 or 60 keV Kr⁺ or Xe⁺ ions. The results indicated that in both cases there was no long-range migration of SIAs

[†] The energy régime $500 \leq \epsilon \leq 2500 \text{ eV}$ corresponds to $0.005 \leq \epsilon \leq 0.025$.

between 6 and 30 K. This assured us that the distribution of the radiation damage which we measured at 30 K was not affected by the long-range migration of SIAs.

II. EXPERIMENTAL DETAILS

The irradiation experiments were performed in two different systems. In one system a low-energy (0.2 - 2.5 keV) ion gun was attached to an FIM, and in the other system the FIM was attached to a high-energy (10 - 60 keV) ion accelerator. This chapter contains a description of both systems, and the description of the data recording system and the cine film analysis equipment.

II.1 The Ultra-High Vacuum FIM and the Low-Energy Ion Gun

The FIM employed for the low-energy Ne^+ ions irradiations was an all stainless steel microscope which was operated in a dynamic mode with a background pressure in the range $(5 \text{ to } 10) \cdot 10^{-10}$ Torr.* The temperature of the FIM specimen was varied between 18 K and room temperature by means of a continuous-transfer liquid-helium cryostat.⁽⁶¹⁾ The specimen temperature was monitored with a miniature platinum resistance thermometer (PRT) mounted near the tail of the cryostat. The FIM specimens were irradiated in situ at 30 K in this bakable ultra-high vacuum (UEV) FIM employing a specially-constructed low-energy ion gun. Figure 3 shows the experimental setup and the position of the FIM specimen relative to the ion gun. This ion gun is a modified version of the ion gun that we have previously described.^(62,63) In the ion gun's present form the plasma chamber and the Einzel lens elements were both floated above earth potential. This design permitted the irradiation to be performed with the FIM specimen maintained at earth potential, and thus avoided the

* All pressures reported in this thesis have been converted from gauge to real pressure.

problem of field-emission induced melting and subsequent failure of the FIM specimen.⁽⁶²⁾ The gas was ionized in the plasma chamber by a magnetically confined 10 mA beam of 90 eV electrons emitted from a resistively-heated tungsten filament. By maintaining the energy of the ionizing electrons below 90 eV, more than 98.5% of the Ne^+ ions produced were singly-charged.⁽⁶⁴⁾ This ion gun, when operated at a pressure of $1.2 \cdot 10^{-3}$ Torr neon, delivered a current density of $0.5 \mu\text{A cm}^{-2}$ Ne^+ ions at an energy of 500 eV and $1.6 \mu\text{A cm}^{-2}$ Ne^+ ions at an energy of 2000 eV.

The energy distribution of the ions was measured using the retarding-field collector method.⁽⁶⁵⁾ The measurements were performed at a constant pressure of $1.2 \cdot 10^{-3}$ Torr neon, and the difference between the filament voltage (V_f) and the plasma chamber voltage (V_p) was constant and equal to 90 Vdc. A schematic representation of the ion gun system and the voltages involved are shown in Fig. 3. Measurements were performed to detect a leakage current; these measurements were performed with -300 Vdc on the secondary electron-retarder; this procedure prevented the escape of secondary electrons that were produced inside the Faraday cup. The voltage on the Faraday cup was varied between 0 and 1000 Vdc, and no leakage currents greater than 10^{-13} A were detected. This value (10^{-13} A) corresponded to 10^{-5} of the typical ion current employed in the irradiation experiments. The energy spread of the ions was determined by progressively increasing the dc potential on the Faraday cup and measuring the total ion current as a function of this retarding potential. A typical experimental curve is shown as a solid line in Fig. 4. And the negative first derivative of this current density curve is shown as a broken line. The maximum ion energy, where a cutoff in the current was observed, was found to be 10 to 30 Vdc less than the plasma potential.

Figure 3: A schematic diagram of the experimental setup.

Figure 4: Ion-beam cutoff characteristic and energy spectrum. (a) Is the ion-current density as a function of the retarding potential, and (b) is the negative first derivative of ion-current density curve.

No ions with a greater energy than the cutoff voltage hit the FIM specimen and about 80% of the ions striking the specimen were within 50 Vdc of this cutoff voltage.

In order to preserve UHV conditions when the ion gun was operated, it was necessary to thoroughly out gas the ion source. Therefore during the bakeout of the FIM, the ion gun was operated in the following mode: (1) the Einzel lens elements and V_p were maintained at ground potential; (2) V_f was maintained at -500 Vdc; and (3) the filament current was increased until about 30 mA of emission current was obtained. The entire system was then baked for 12 hours. After the bakeout, V_p was maintained at -90 Vdc with a filament current of 10 mA. The ion gun was left in this state until the system had to be opened to air to exchange specimens.

The irradiations were performed with the following standard conditions: (1) an emission current of 10 mA; (2) V_p was varied between 500 and 2500 Vdc; (3) $V_f = (V_p - 90)$ Vdc; and (4) the central element of the Einzel lens sat at a voltage of $0.80 V_p$. In most of the irradiation experiments the sum of the irradiation time and the time the electric imaging field was off was less than or equal to ten minutes. The calculated time required to form an adsorbed monolayer of neon, assuming a sticking coefficient of unity, at a background pressure of $6 \cdot 10^{-10}$ Torr at 30 K is approximately 15 minutes. Control experiments indicated that less than a monolayer of residual gas was adsorbed on the specimen's surface during a typical irradiation period of ten minutes.

II.2. The Ultra-High Vacuum FIM and the High-Energy Ion Accelerator

An all stainless steel FIM was attached to a Hill-Nelson sputtered metal ion source via a three-stage differentially-pumped flight-tube. For a detailed description and operational procedure see our earlier

publications.^(66,67) This arrangement allowed for the in situ irradiation of the FIM specimen at a background pressure in the range $(6 \text{ to } 10) \cdot 10^{-10}$ Torr with a pressure of Kr ranging between $(2 \text{ to } 10) \cdot 10^{-6}$ Torr measured above the diffusion pump which evacuated the Hill-Nelson ion source. The ion beam was magnetically analyzed so that only the desired ion struck the specimen. A beam-steering system was used to adjust the position of the ion beam relative to specimen. A special Faraday cup⁽⁶⁸⁾ was employed to simultaneously monitor the profile and intensity of the ion beam. The FIM specimens were irradiated at ~ 6 K with 30 or 60 keV Kr^+ ions to a dose of $\sim 1 \cdot 10^{12}$ ion cm^{-2} and the flux of the ions at the specimen position was $\sim 3 \cdot 10^{10}$ $\text{cm}^{-2} \text{sec}^{-1}$. Control experiments showed that at this value of flux the time required to form an adsorbed monolayer was greater than the irradiation period. The temperature of the FIM specimen was monitored with a miniature PRT, and it was varied between ~ 6 K and room temperature by means of a continuous-transfer liquid-helium cryostat.

II.3. Data Recording System and Ciné Film Analyzer

The FIM images were observed with the aid of an internal image-intensification system based on a Galileo 76 mm diameter channel electron multiplier array (CEMA).⁽⁶⁹⁾ The FIM images produced in this manner were recorded on 35 mm ciné film employing a semi-automated Automax 35 mm ciné camera equipped with a 1000 ft. film chamber⁽⁷⁰⁾ which was operated in two modes. The first mode was ciné, and was used during the isochronal warming experiments. The second mode involved the synchronization of the voltage output of a pulse amplifier⁽⁷¹⁾ with the shutter of the ciné camera, so that the specimen was alternately pulsed to field evaporate a few atoms and then photographed at best image voltage (BIV) between the voltage pulses.⁽⁶⁶⁾ The recorded and developed ciné film

was analyzed with a Vanguard motion analyzer which was equipped with an x-y atom position readout⁽⁷⁰⁾ and a zeroing device. The zeroing device, capable of zeroing the position read-out of any point on the ciné film, allowed us to read directly the coordinates of any point on the ciné film with respect to any zeroed point. The position read-out of the motion analyzer was interfaced to a Houston Omnigraphic 200 x-y recorder. The positions of the atomic sites detected in the FIM micrographs, after zeroing each micrograph, were transferred to a sheet of graph paper with the aid of the x-y recorder. In this way a map of all the atom positions in each plane was obtained.⁽⁷²⁾

III. DEPTH OF RADIATION DAMAGE IN THE ORDER-DISORDER ALLOY Ni_3Mo

The depth of radiation damage, produced in the ordered alloy Ni_3Mo , as a result of bombardment with low-energy Ne^+ ions, was measured directly employing the FIM technique.⁽⁷³⁾ The measurements were based on the contrast differences between the FIM images of an ordered alloy, which were completely regular (like those of pure metal), and a disordered alloy which appeared as random array of image points.^(56,74) Specimens of the ordered alloy Ni_3Mo were bombarded in situ at 30 K with 500 to 2500 eV Ne^+ ions; this corresponded to a range of the ISS reduced energy ϵ between 0.01 and 0.05. As the incident ions penetrated the ordered alloy, they lost their energy as a result of a series of inelastic and elastic collisions. The elastic collisions produced radiation damage, i.e., point defects and replacement events, and hence disordered the bombarded side of the specimen. The change from an ordered state to a disordered state was detected by the controlled dissection of the specimen on an atom-by-atom basis, employing the pulse field-evaporation technique.^(32,75) The amount of metal evaporated in order to remove the disordered region and expose the perfectly ordered specimen, and the basic geometric relations for an FIM specimen gave the depth of radiation damage.

III.1 Experimental Techniques

III.1.1 Specimen Preparation

An ingot of the alloy Ni_3Mo was prepared from 99.97 wt.% pure nickel (obtained from Huntington Alloys) and 99.97 wt.% pure molybdenum (obtained from Climax Molybdenum Metal) by arc melting in an argon atmosphere. In order to obtain a dense, gas-free alloy,⁽⁷⁶⁾ the ingot was subsequently swaged into a 0.5 cm diameter rod and then remelted six times in an

electron beam zone-refining system which was operated at a background pressure of $5 \cdot 10^{-6}$ Torr. The rod was inverted between each pass to ensure a uniform distribution of the constituents. The final homogenization treatment consisted of annealing the rod in a vacuum of $5 \cdot 10^{-6}$ Torr for 100 hours at 1200°C. The alloy was then drawn into a 0.0254 cm diameter wire. The composition of the wire was determined by atomic absorption spectroscopy analysis which was performed in the Chemical Analytical Facility of the Cornell Material Science Center. The final composition of the wire was found to be 80.6 ± 0.4 at.% nickel and 19.4 ± 0.4 at.% molybdenum. The wire was cut into 10 cm long specimens and these were then sealed into evacuated quartz tubes at a pressure of better than 10^{-5} Torr. The ordering treatment^(77,78) consisted of initially annealing the specimen at 800°C for two days. Next the specimen temperature was gradually decreased over a period of two weeks to 600°C; and finally the specimen was cooled to room temperature during the course of one day.

Sharply-pointed FIM specimens were prepared by electroetching 12 mm long specimens in a freshly made solution of one part H_2SO_4 :20 parts methanol (by volume) at room temperature; a potential of 5 Vac was used and the counter-electrode material was stainless steel. The etching procedure consisted of dipping a 5 mm length of the specimen into the solution and then applying the voltage and electroetching away a 2 mm length of the specimen. When examined with an optical microscope at a magnification of 400X, a good specimen had the appearance of a well-sharpened pencil. The electroetching procedure was repeated using short (~ 0.5 sec) 5 Vac pulses, until a satisfactory sharpness was achieved. The specimen was then

transferred to the FIM which was evacuated to a pressure of $6 \cdot 10^{-10}$ Torr. Finally the specimen was pulse field-evaporated at 30 K to a final end-form⁽⁷⁹⁾ in the presence of $1 \cdot 10^{-4}$ Torr neon.

III.1.2 Irradiation and Data-Recording Procedures

Each specimen was bombarded in situ at 30 K, at a constant pressure of $1.2 \cdot 10^{-3}$ Torr neon, with the specially constructed ion gun. The specimens were bombarded with low-energy (500-2500) eV Ne^+ ions at a constant flux (ϕ) of $5 \cdot 10^{12}$ ion cm^{-2} sec^{-1} to a constant dose (ϕ_d) of $3 \cdot 10^{15}$ ion cm^{-2} . The sum of the irradiation time and the time the electric imaging field was off was less than or equal to ten minutes. In the case of the 500 eV Ne^+ ions irradiations the value of ϕ was $2.5 \cdot 10^{12}$ ion cm^{-2} sec^{-1} and therefore a ϕ_d of $3 \cdot 10^{15}$ ion cm^{-2} was achieved in 20 minutes. At these values of ϕ and background pressure, i.e., $6 \cdot 10^{-10}$ Torr, and temperature (30 K), control experiments showed that the time required to form an adsorbed monolayer of gas was greater than the irradiation period.

After an irradiation each specimen was carefully pulse field-evaporated and the FIM image was photographed between each pulse employing the semi-automated 35 mm ciné camera. The pulse height and duration were adjusted so that one atom, on the average, was removed per four pulses on the (121) plane. The pulse field-evaporation dissection of a specimen was continued until most of the radiation damage had been removed from the specimen. A total of 1400, 2500, 3500, 4600 and 5500 frames of film were photographed for the 500, 1000, 1500, 2000 and 2500 eV bombardments, respectively. The number on each frame of film was keyed to the amount of metal removed; approximately 240 frames were photographed during the removal of each (121) plane. The developed ciné film was analyzed with

the aid of the Vanguard motion analyzer. The position of the atomic sites detected in the FIM micrographs were transferred to a sheet of graph paper with the aid of an x-y recorder. In this way a map of all the atomic positions in each plane was obtained.

III.2 Procedure for Analysis

III.2.1 The Indexing of FIM Micrographs of Ordered Ni_4Mo Alloy

In analyzing the data, the first step was to index the poles that appeared in the field-ion micrographs. The poles were indexed employing symmetry considerations, the zone rule, and the relative prominence rule.⁽⁸⁰⁾ The resulting indexed micrograph was confirmed by comparing it to computer simulated images of ordered Ni_4Mo .⁽⁸¹⁾ The indexed micrograph also was consistent with the work of Yamamoto *et al.*⁽⁷⁷⁾

The crystal structure of an ordered Ni_4Mo alloy has been determined by Harker employing standard x-ray crystallographic techniques.⁽⁸²⁾ The disordered face-centered cubic (fcc) structure undergoes a slight contraction along one cube axis during ordering and becomes tetragonal with $c/a = 0.986$. The ordered structure belongs to the D_{4h} space group and has a body-centered tetragonal (bct) unit cell with $a = 5.727 \text{ \AA}$, $c = 3.566 \text{ \AA}$, and $c/a = 0.623$. The ordered bct unit cell shown in Fig. 5 consists of eight nickel and two molybdenum atoms.* Two sets of planes exist in an ordered Ni_4Mo alloy; the fundamental planes and the superlattice planes. In a set of fundamental planes, for which $h+k+l = 2n$ and $2h+k = 5n$,†

* The position of the ten atoms in the bct unit cell are as follows:
Mo at $xyz = 000$ and $1/2 \ 1/2 \ 1/2$
Ni at $xyz = 2/4 \ 4/10 \ 0; 8/10 \ 4/10 \ 0; 6/10 \ 2/10 \ 0; 8/10 \ 6/10 \ 0;$
 $1/10 \ 7/10 \ 1/2; 9/10 \ 7/10 \ 1/2; 9/10 \ 3/10 \ 1/2; 3/10 \ 1/10 \ 1/2.$

† The Miller indices given in this chapter refer to the bct structure.

Figure 5: Schematic representation of a body-centered tetragonal unit cell showing the location of the Ni and Mo atoms in the ordered alloy Ni_4Mo .

where $n = 0, 1, 2, \dots$, and h, k , and l indices refer to the bct lattice, successive layers have identical mixed packing, with every fifth atom being molybdenum. In the superlattice set of planes, for which $h+k+l = 2n$ and $2h+k \neq 5n$, the planes are layered, with every fifth plane consisting of only molybdenum atoms, and the intervening four planes consisting of only nickel atoms. The interplanar distance d_{hkl} for the ordered Ni_4Mo alloy was calculated using the equation:

$$\frac{1}{d_{hkl}^2} = \left(\frac{h^2 + k^2}{a^2} \right) + \left(\frac{l^2}{c^2} \right) \quad (16)$$

The crystallographic angle β between two poles $h_1 k_1 l_1$ and $h_2 k_2 l_2$ was calculated using the equation:

$$\cos \beta = \frac{\frac{h_1 h_2 + k_1 k_2}{a^2} + \frac{l_1 l_2}{c^2}}{\left\{ \left(\frac{h_1}{a} \right)^2 + \left(\frac{k_1}{a} \right)^2 + \left(\frac{l_1}{c} \right)^2 \right\} \cdot \left\{ \left(\frac{h_2}{a} \right)^2 + \left(\frac{k_2}{a} \right)^2 + \left(\frac{l_2}{c} \right)^2 \right\}}^{1/2}$$

The overall symmetry in the FIM micrographs of the ordered Ni_4Mo alloy was that of a bct lattice. This has been shown, by measurements of the step height⁽⁸³⁾ and by comparison with computer simulation images⁽⁸⁴⁾, to be due to the molybdenum atoms being more prominent than the Ni atoms; therefore, one essentially sees only one of the bct sublattices. If the nickel and the molybdenum atoms were to appear with equal brightness, one would see the symmetry of the fcc lattice. In the case of Ni_4Mo , where the molybdenum atoms were the imaging species, it was found⁽⁸⁵⁾ that nickel atoms imaged very weakly in the most prominent superlattice planes.

It has been shown⁽⁸⁶⁾ that the size and the prominence of a plane in a field-ion image decreases with decreasing d-spacing. The most prominent superlattice plane was the (110). The next most prominent planes, with decreasing d-spacing, were the (101), (011), (200), (020), (121), (211) and (002). The only plane with fourfold symmetry was the (002). The {110}, and the (200) and (020) planes had twofold symmetry. Referring to the FIM micrograph of the ordered Ni_4Mo alloy in Fig. 6, one can see that the plane A has fourfold symmetry. The planes B, C, D and E have twofold symmetry. Because plane A has fourfold symmetry, we assumed that it was the (002) plane, and because planes B, C, D, and E have twofold symmetry we assumed that they were the (011), (101), (0 $\bar{1}$ 1), and ($\bar{1}$ 01) planes, respectively. The method presented by Newman et al.⁽⁸⁷⁾ for indexing FIM images was used. They related the linear distance between any two planes on the micrograph, d , to the curvilinear distance along the surface of the tip between the same two planes, $S = R\beta$, as $d = k\beta$, where β was the crystallographic angle between these planes, and k was a proportionality constant.

The crystallographic angle between the (002) and (011) planes was $\beta = 31.91$ deg, and the linear distance between A and B, measured from Fig. 6, was $d = 47$ nm. Inserting these values in the equation $d = k\beta$, we found $k = 1.473$ nm deg⁻¹. Using this k value and the distance between A and C, $d = 47$ nm measured from Fig. 6, the angle β between A and C was found to be 31.9 deg, which was the calculated crystallographic angle between the (002) plane and the (101) plane. The distance between A and each of the B, C, D, and E planes, measured from Fig. 6 was the same and equal to 47 nm; therefore the angle between A and each of the B, C, D, and E planes was

Figure 6: A neon FIM micrograph of an ordered Ni_4Mo alloy recorded at a specimen temperature of 30 K.

the same and equal to 31.9 deg, which was the calculated crystallographic angle between the (002) plane and each of the planes (011), (101), ($0\bar{1}1$) and ($\bar{1}01$). This was consistent with our choice for the above planes.

To index planes that did not have any symmetry elements, the (001) stereographic projection for the ordered Ni_4Mo was used. The stereographic projection was obtained using the procedure described by Kelly and Groves⁽⁸⁸⁾ and is represented in Fig. 7. For example, the plane G in Fig. 6, after inspection in the stereographic projection, was assumed to be the ($1\bar{2}1$) plane. To confirm this the distance between A and G on the micrograph was measured and found to be 83 mm. The angle between these two planes, using the k value derived before, was 56.4 deg. The calculated crystallographic angle between the planes (002) and ($1\bar{2}1$) was 54.3 deg, which showed that plane G was indeed the ($1\bar{2}1$) plane. In the same way the planes F, H, and K were indexed as (121), ($\bar{2}11$) and ($\bar{1}\bar{2}1$), respectively. Once the major planes were identified, the other planes could be identified using the zone rule. For example, the plane L, according to the zone rule should be the (112) plane. We confirmed this by measuring the distance between L and A, $d = 32$ mm, and finding the angle between these two planes, $\theta = 22.1$ deg. This was consistent with the crystallographic angle between the planes (002) and (112), which was 23.7 deg. In the same manner, using the zone rule and the stereographic projection, the remaining planes on the FIM micrograph were indexed. Fig. 8 shows the resulting crystallographic map of the FIM micrograph shown in Fig. 6.

III.2.2 Determination of the Depth of Radiation Damage

The ion beam was perpendicular to the axis of the FIM specimen, and as a result of an irradiation the bombarded side, i.e., the side exposed to the ion beam, became disordered. Figure 9 is a schematic representation

Figure 8: The crystallographic map of the FIM micrograph shown in Fig. 6.

of an FIM specimen after an irradiation (initial surface) and after removing $n(h_1 k_1 l_1)$ atomic planes to obtain a final surface which contained an ordered $(h_2 k_2 l_2)$ plane. The disordered region was removed by the pulse field-evaporation technique, on an atom-by-atom basis until the perfectly ordered crystal was exposed.

The local tip radius (r) between any two poles was obtained by applying the ring-counting method developed by Drechsler and Wolf⁽¹⁹⁾; their expression for the tip radius is

$$r = \frac{nd_{hkl}}{(1 - \cos\beta)} \quad (17)$$

where n is the number of rings between the two poles, d_{hkl} is the step height, and β is the crystallographic angle between the two poles. The local tip radii (r_i) between all the poles for which we could count the number of rings between were measured before the irradiation. The average of all these r_i values measured on the bombarded side of the specimen was used as the average tip radius ($\bar{R}$); the quantity $\bar{R}$ was defined by

$$\bar{R} = \frac{1}{m} \sum_{i=1}^m r_i \quad (18)$$

Employing Fig. 9 we obtained the following equations which described the geometrical relationships embodied in this diagram:

$$R_1^2 = y^2 + \xi^2 \quad (19)$$

$$nd_{n_1 k_1 l_1} + y = R_2 \cos\beta \quad (20)$$

Figure 9: A schematic representation of an FIM specimen after an irradiation (initial surface) and after removing $n(h_1k_1l_1)$ planes to obtain a final surface which contained an ordered $(h_2k_2l_2)$ plane.

$$x = \xi - R_2 \sin \beta ; \quad (21)$$

and

$$\xi = (R_1^2 - y^2)^{1/2} = [R_1^2 - (R_2 \cos \beta - n d_{h_1 k_1 l_1})^2]^{1/2} . \quad (22)$$

Thus, the general expression obtained for the depth of radiation damage (x) was

$$x = [R_1^2 - (R_2 \cos \beta - n d_{h_1 k_1 l_1})^2]^{1/2} - R_1 \sin \beta , \quad (23)$$

where R_1 and R_2 were the average tip radii before the irradiation and after the removal of $n(h_1 k_1 l_1)$ central planes, respectively. The local tip radii were remeasured after the radiation damage had been removed by the pulse field-evaporation technique and it was found that the r_1 , and hence $\bar{R}$, had not changed as a result of the removal of the radiation damage. Usually the removal of about 50 Å or less of metal, depending on the bombarding energy, was sufficient to remove most of the radiation damage. Thus the value of R_2 used in eqns. (21-23) was the same as R_1 . Under these conditions eqn. (23) became:

$$x = [R_1^2 - (R_1 \cos \beta - n d_{h_1 k_1 l_1})^2]^{1/2} - R_1 \sin \beta . \quad (24)$$

An FIM specimen is not an ideal spherical cap but has a complicated end form, which is a result of the field-evaporation process.⁽⁹⁰⁾ The measurement of the r_1 values along the main zone axes is known to give a good description of the shape of the end form.⁽⁸⁹⁾ Such measurements were

performed on each of the specimens before an irradiation and the measured values showed that the shape of the end form was best approximated by a hemisphere. We also checked to see if the shape of the end form had changed as a result of the pulse field-evaporation process. We recorded the number of atomic planes removed from each pole during the controlled pulse field-evaporation process. These numbers and the knowledge of the crystallographic angles between the poles, showed that the amount of material that was removed from each pole was the same. This showed that the shape of the end form had not changed. Therefore, the shape of the end form was taken to be a hemisphere.

Schematic top and side views of an FIM specimen are shown in Fig. 10. The vector $\vec{A}$ is the normal to the central $(h_1 k_1 l_1)$ plane and the vector $\vec{B}$ is the normal to the $(h_2 k_2 l_2)$ plane. The centers of both planes lie along the bombardment direction; $\vec{B}$ passes through the center of the $(h_2 k_2 l_2)$ plane. In order to determine the bombardment direction, first the vector cross-product $(\vec{A} \times \vec{B} = \vec{C})$ was calculated; then the vector cross-product $\vec{A} \times \vec{C}$ was calculated and the resultant vector $\vec{D}$ indicated the bombardment direction.

In order to obtain a complete radiation damage profile one must determine the local atomic arrangement in the bombarded specimen. By a radiation damage profile we mean the total amount of disorder produced in the lattice, i.e., the number of displaced and replaced atoms, by the incoming ions as a function of the depth from the surface of the specimen along the bombardment direction. The value of the long-range order parameter (S) can be obtained from the local atomic arrangement; this has been done in partially ordered Pt_3Co using the FIM technique.⁽⁹¹⁾ If one starts

Figure 10: Schematic top and side views of an FIM specimen showing the geometric relations between the bombardment direction and the poles on the FIM specimen. (a) A top view of an FIM specimen with the central $(h_1k_1l_1)$ plane and the $(h_2k_2l_2)$ plane. The center of these two planes lie along the bombardment direction. (b) A cross sectional view along the central axis of an FIM specimen showing the direction vectors.

with a fully-ordered alloy ($S = 1$), then after a bombardment the radiation damage profile will be reflected in the S values that are a function of the depth from the irradiated surface. A small value of S indicates that a region has been heavily damaged. We made a strong effort to determine the positions of atoms in each plane in various stages of the pulse field-evaporation process to determine S , but it was unsuccessful in the case of Ni_4Mo . The most likely reason we were unsuccessful centers around the fact that Ni atoms, in the Ni_4Mo alloy, were invisible.⁽⁸³⁾ The Ni atoms constituted 80% of the total number of atoms, whereas in Pt_3Co the Pt atoms were visible and they constituted 75% of the total number of atoms.

Nevertheless, we were able to distinguish the ordered and the disordered regions. Fig. 11 shows an approximate representation of a radiation damage profile; the regions where the specimen appeared disordered and ordered during the pulse field-evaporation dissection of a specimen are indicated schematically. The specimen appeared disordered as long as the radiation damage was above a certain level (level D in Fig. 11), and below level D the specimen appeared ordered; the depth (x) indicates the position of this transition.

In order to determine the depth of the radiation damage we had to determine the number of central atomic planes ($h_1k_1l_1$) removed before an ordered plane ($h_2k_2l_2$) was exposed on the bombarded side of the specimen (see Fig. 9). Since the interface between the ordered and the disordered regions was diffuse we had to establish criteria for determining when an atomic plane ($h_2k_2l_2$) was ordered. Two criteria were used. The first criterion defined an atomic plane ($h_2k_2l_2$), that was exposed during the removal of atoms from the surface, to be ordered when the atoms in the

Figure 11: A schematic representation of radiation damage profile, x_d indicates the depth at which the radiation damage level goes below the level D , and the FIM image of the specimen changes from a disordered one to an ordered one.

center of the plane were ordered; i.e., the positions of the atoms in the center of the plane were identical to those observed in the perfectly ordered crystal. This criterion was checked by mapping the atomic positions of a certain $(h_2k_2l_2)$ plane in the perfectly ordered crystal before an irradiation and by comparing this map to maps of the atomic positions of the same $(h_2k_2l_2)$ planes after an irradiation. Fig. 12(a) shows the center of a (031) atomic plane of an ordered Ni_4Mo specimen before an irradiation, and Fig. 13(b) after an irradiation and after the removal of the disordered region. The positions of the atoms in the center of the (031) plane are the same in these two figures. In order to ensure that the atoms in the center of the plane $(h_2k_2l_2)$ were not accidentally ordered, we also require that the next $(h_2k_2l_2)$ atomic plane had to be ordered. Figure 13(c) shows that the center of the next (031) plane was also ordered.

In the second criterion we defined a plane, exposed during the pulse field-evaporation process, to be ordered when the ledge-atoms of the plane at its maximum size were ordered; the innermost ring of image points on the plane $(h_2k_2l_2)$ at its maximum size had to be ordered giving the contrast of a perfect ring of image points. Figure 12(b) shows the innermost ring of image points of a (031) plane of an ordered Ni_4Mo specimen before the irradiation, and Fig. 13(d) after the irradiation and the removal of the disordered region. The ledge-atoms in these two figures exhibited the contrast of a perfect ring of atoms. The second criterion was more stringent than the first one; because a larger fraction of the plane had to be ordered. Figures 13(b) and 13(c) show that while the centers of the plane were ordered, i.e., fulfilling the first criterion, the ledge-

Figure 12: Two FIM micrographs of the (031) atomic plane of an ordered Ni_4Mo specimen recorded during the pulse field-evaporation process. The center of the plane is shown in (a) in the white circle, and the ledge-atoms of the plane at its maximum size are shown in (b) indicated by an arrow. The position of the atoms are illustrated below each micrograph.

Figure 13: Five FIM micrographs showing the dissection of the (031) atomic planes of a Ni_4Mo specimen, which had been irradiated with 1 keV Ne^+ ions, recorded at a specimen temperature of 30 K during the pulse field-evaporation process. The position of the atoms are illustrated below each micrograph: (a) recorded immediately after the irradiation; (b) the atoms in the center of the (031) planes appeared ordered for the first time; (c) after the removal of one (031) atomic plane from the specimen which was recorded in (b); (d) the ledge-atoms appeared ordered for the first time; and (e) after the removal of one (031) atomic plane from the specimen which was recorded in (d).

atoms were still disordered, thus not fulfilling the second criterion. After the removal of more atoms from the surface of the specimen (Fig. 13 (d)) the second criterion was fulfilled. Therefore, the value of x obtained using the second criterion was greater than the one obtained using the first criterion.

The resulting number of central planes ($h_1 k_1 l_1$) removed (n) was inserted into eqn. (24) and the quantity x was calculated. We repeated this procedure, of finding the number of central planes removed in order to detect when a plane was ordered, for each of the disordered ($h_2 k_2 l_2$) planes. We calculated x_i for each plane and then calculated the mean value ($\bar{x}$) and the sample standard deviation σ^* using all the x_i values obtained for each bombardment.

III.3 Experimental results

III.3.1 Control Experiments

The isochronal recovery experiments, which are described in Chapter V, showed that there was no long-range migration of SIAs between 6 and 30 K. In order to minimize the adsorption of residual gases during the irradiation period, and to prevent any change in the depth of the radiation damage as a result of the migration of SIAs, the temperature of the irradiation experiments was chosen to be 30 K.

Three types of control experiments were performed in order to determine the effects of the adsorption of residual gases on the surface of the specimen during the irradiation period. Each control experiment was performed

* The standard deviation σ was calculated employing the expression $\sigma = \{[\sum_{i=1}^N (x_i - \bar{x})^2] / (N-1)\}^{1/2}$ (92) where N was the total number of disordered ($h_2 k_2 l_2$) planes checked and x_i was the measured depth of damage for each of these ($h_2 k_2 l_2$) planes.

for a period of ten minutes; this was the time required for achieving $\Phi_d = 3 \cdot 10^{15}$ ion cm^{-2} , under the same vacuum and temperature conditions as the irradiation experiments, i.e., $1.2 \cdot 10^{-3}$ Torr Ne and 30 K. In the first experiment the ion gun was not turned on and our observations indicated that no change in the appearance of the specimen's surface occurred. During the second experiment the ion gun was turned on and the potentials on the lenses and the plasma chamber were maintained at zero voltage. In the third control experiment the ion gun was operated in its normal mode, producing 2 keV Ne^+ ions, but the specimen was held at a positive potential of 4 keV; therefore only neutral neon atoms were able to reach the specimen. In the second and the third control experiments some small amount of adsorption was observed; although the field evaporation of less than one monolayer of atoms was required to detect the perfectly ordered crystal. No radiation damage was detected in the specimen in all the control experiments; this latter result indicated that our experiments were not affected by the small neutral component of neon atoms in the ion beam. It was also concluded that the surface adsorption of residual gases on each specimen's surface, during the irradiation period, did not affect our measurements.

Exploratory experiments, in which the specimen was bombarded to different doses, were performed in order to optimize the conditions required for separating the ordered region from the disordered one. The irregular contrast pattern observed after an irradiation was attributed to the random displacement of the Ni and Mo atoms from their normal sites, i.e., the disordering of the crystal. The implanted Ne atoms were not considered to be a source of the observed contrast effect because the process of image formation in the FIM indicated that they were not imaged.^(93,94) As a

specimen was pulse field-evaporated the implanted Ne atoms intersected the surface. They were either field-desorbed instantaneously without producing any contrast effect(s)⁽⁹³⁾ or they were field-adsorbed on the surface as a result of the high electric field, but this adsorption is not expected to change the image pattern of the metal.⁽⁹⁴⁾ When the specimen was bombarded to a ϕ_d of less than $1 \cdot 10^{14}$ ion cm^{-2} we were not able to distinguish a disordered region because the radiation damage was not extensive enough. But as ϕ_d was increased to $> 5 \cdot 10^{14}$ ion cm^{-2} we were able to detect a disordered region.

III.3.2 Depth of Radiation Damage Measurement

Three sets of irradiation experiments were performed. For each set the bombardment direction was different: in the first, second and third sets the bombardment directions were parallel to the $[\bar{7}6\bar{2}]$, $[100]$, and $[\bar{1}\bar{2}2]$ crystallographic directions of the Ni_4Mo crystal, respectively. In the first and second sets the specimens of the ordered Ni_4Mo were irradiated with 500 to 2000 eV Ne^+ ions, and in the third set with 500 to 2500 eV Ne^+ ions. A typical example of a FIM micrograph of the perfect lattice recorded during the pulse field-evaporation dissection of a specimen before an irradiation is shown in Fig. 14. Figure 15 shows a crystallographic map of the micrograph shown in Fig. 14 with the local tip radii along the $[100]$, $[\bar{1}11]$, $[001]$, $[11\bar{1}]$, $[\bar{1}\bar{1}1]$, and $[\bar{1}01]$ zone axes indicated. As stated before, the measured values of r_i along these zone axes showed that the shape of the end form could be best approximated by a hemisphere. Typical examples of FIM micrographs recorded during the pulse field-evaporation dissection of a specimen which was irradiated with 1000 eV Ne^+ ions, are shown in Figs. 16 to 19; these micrographs were taken after the removal of

Figure 14: A neon FIM micrograph of an ordered Ni_4Mo alloy recorded at a specimen temperature of 30 K before an irradiation with 1000 eV Ne^+ ions.

Figure 15: The crystallographic map of the FIM micrograph shown in Fig. 14. The numbers between the poles are the local tip radii values along the zone axes which are indicated by the arrows.

Figure 16: A neon FIM micrograph recorded at a specimen temperature of 30 K after an irradiation with 1 keV Ne^+ ions, and after the removal of two central (121) planes. The irradiated side is completely disordered.

Figure 17: A neon FIM micrograph recorded at a specimen temperature of 30 K after the removal of two central (121) planes from the specimen which was recorded in Fig. 16.

Figure 18: A neon FIM micrograph recorded at a specimen temperature of 30 K after the removal of two central (121) planes from the specimen which was recorded in Fig. 17. The (020) plane starts to appear more ordered.

Figure 19: A neon FIM micrograph recorded at a specimen temperature of 30 K after the removal of four central (121) planes from the specimen which was recorded in Fig. 18. Most of the disordered lattice has been removed. The ledge-atoms of the (020) plane appear ordered.

two, four, six and ten complete (121) planes, respectively. Figure 16 shows that the planes on the irradiated side of the specimen were completely disordered, whereas the planes on the unirradiated side were ordered. As the removal of atoms from the specimen's surface was continued more ordered planes were exposed (Figs. 17-18). The removal of atoms was continued until most of the disordered planes were removed as shown in Fig. 19.

The results for the three sets of irradiation experiments are summarized in Tables I to VI, where x_d^I is the depth of radiation damage obtained using the first criterion, and x_d^{II} using the second criterion. The largest error in the number (n) of central planes removed, in order to expose an ordered plane, was one central plane; in the majority of the cases it was less than one. The resulting error in the depth of radiation damage was of the order of 10% for small β ($< 20^\circ$) and small n (3), and of the order of 5% for large β ($\sim 50^\circ$) and large n (10). A straight forward error analysis showed that an error of 10% in the average radius R_1 values resulted in a less than 1% error in the depth of radiation damage values.

Figures 20 to 22 show the experimental values of $\bar{x}_d^I$ and $\bar{x}_d^{II}$ for the three sets of irradiations. The error bars on the experimental data points indicate $\pm$ one σ . The experimental depth of radiation damage versus energy curves in the figures were obtained employing the method of least squares fit⁽⁹⁵⁾ of the data points to the power-law expression

$$\bar{x}_d = fE^c. \quad (25)$$

The fitted constants f and c for the quantities $\bar{x}_d^I$ and $\bar{x}_d^{II}$ for the different irradiation directions are presented in Table VII.

Table I: Summary of results of damage depth x_d^I for the four irradiations parallel to the $[\bar{7}62]$ direction.

Bombarding Energy (eV) Average R_1 (Å)		500 700		1000 710		1500 727		2000 742	
exposed ordered planes	β (deg)	nd_{121} (Å)	x_d^I (Å)	nd_{121} (Å)	x_d^I (Å)	nd_{121} (Å)	x_d^I (Å)	nd_{121} (Å)	x_d^I (Å)
(433)	21.89	a				10.0	23.67	13.69	31.86
(323)	27.18			7.81	14.78			7.07	14.1
(321)	28.17					6.8	12.41	8.5	15.45
(211)	29.77	4.3	7.44	7.87	13.37	8.84	15.20	8.06	13.79
(424)	31.79	4.73	7.52	8.53	13.43			14.6	22.61
(312)	35.05	4.91	6.90	7.86	10.98	10.49	14.57	18.7	25.49
(613)	42.55	7.18	7.71	11.26	11.99	18.72	19.64	26.57	27.46
(512)	42.87	6.06	6.45	9.14	9.66	13.0	13.65	17.20	17.92
(411)	45.23	6.84	5.72	12.47	12.08	10.2	9.92	14.56	14.05
(101)	45.69	7.37	6.88	10.39	9.64	11.95	11.05	18.04	16.51
(503)	48.21	7.79	6.86	10.14	8.89	11.39	9.97	15.05	13.08
(402)	49.57	8.31	6.97	10.39	8.68	12.8	10.65	21.81	17.88
(301)	53.45	9.4	6.85	9.24	6.79	13.8	9.98	29.2	20.58
(501)	58.38	6.75	4.10						
(3 $\bar{1}$ 2)	60.25	5.38	3.04	13.35	7.44	18.4	10.17	21.94	11.92
(6 $\bar{1}$ 2)	60.54	11.09	6.13	10.39	5.76	28.82	15.43	40.01	20.98
(4 $\bar{1}$ 1)	67.92							43.63	16.13
(6 $\bar{1}$ 3)	56.67	10.39	6.7						
Mean and one standard deriva- tion (Å)		6.38 $\pm$ 1.31		10.27 $\pm$ 2.78		13.56 $\pm$ 4.21		18.74 $\pm$ 5.71	

^a Where there are no entries, no measurements were taken.

Table II: Summary of results of damage depth x_d^{II} for the four irradiations parallel to the $[762]$ direction.

Bombarding Energy (eV) Average R_p (Å)		500 700		1000 710		1500 727		2000 742	
exposed ordered planes	θ (deg)	nd_{121} (Å)	x_d^{II} (Å)	nd_{121} (Å)	x_d^{II} (Å)	nd_{121} (Å)	x_d^{II} (Å)	nd_{121} (Å)	x_d^{II} (Å)
(433)	21.89	a				10.74	25.34	16.85	38.65
(323)	27.18							13.28	24.68
(321)	28.17					8.52	15.47	11.87	21.3
(211)	29.77	5.11	8.79	7.97	12.75	8.32	14.2	9.8	18.53
(424)	31.79	7.52	8.08	10.48	16.40			23.33	35.3
(312)	35.05	6.48	9.09	8.73	12.17	9.54	13.27	22.22	30.0
(613)	42.55	8.96	9.58	14.54	15.37	20.8	21.74	31.0	31.77
(512)	42.87	7.23	7.67	11.40	12.00			21.81	22.52
(411)	45.23	6.67	6.53	10.38	10.09				
(101)	46.59	4.43	4.15	7.73	7.20				
(503)	48.21	8.31	7.31	12.47	10.88	22.19	19.04	26.23	22.35
(402)	49.57	6.91	5.81	13.71	11.38	17.51	14.47	29.0	23.45
(301)	53.45	8.5	6.2	8.31	6.07	14.0	10.52	30.85	21.66
(501)	58.38	9.28	5.61						
(3 $\bar{1}$ 2)	60.25	7.12	4.02	14.75	8.20	23.26	12.74	33.28	17.89
(5 $\bar{1}$ 2)	60.54	12.57	6.93	13.25	7.30	36.28	19.16	42.02	21.95
(4 $\bar{1}$ 1)	67.92			33.24	12.52				
Mean and one standard deviation (Å)		6.91 $\pm$ 1.76		10.95 $\pm$ 3.13		16.60 $\pm$ 4.50		25.38 $\pm$ 6.48	

^a Where there are no entries, no measurements were taken.

Table III: Summary of results of damage depth x_d^I for the four irradiations parallel to the [100] direction.

Bombarding Energy (eV) Average R_1 (Å)		500 607		1000 624		1500 652		2000 760	
exposed ordered planes	β (deg)	$n \cdot d_{002}$ (Å)	x_d^I (Å)	$n \cdot d_{002}$ (Å)	x_d^I (Å)	$n \cdot d_{002}$ (Å)	x_d^I (Å)	$n \cdot d_{002}$ (Å)	x_d^I (Å)
(112)	23.76	a		5.16	11.42	7.70	16.67		
($\bar{1}\bar{1}2$)	23.76			6.18	13.60	6.17	13.58		
(213)	24.90			7.07	14.71	8.11	16.83	7.17	15.01
(2 $\bar{1}$ 3)	24.90			5.87	12.28	6.04	12.66	7.79	16.27
(101)	31.91	5.93	9.33	9.41	14.66	10.26	15.95	11.40	17.75
(334)	33.44	6.94	10.28	7.13	10.56	10.24	15.05	10.70	15.77
(3 $\bar{3}$ 4)	33.44	6.57	9.74	7.25	10.73	10.70	15.70	10.55	15.55
(323)	36.81	8.72	11.38	7.33	9.60	11.22	14.56	10.33	13.50
(3 $\bar{2}$ 3)	36.81	8.34	10.89	7.49	9.81	9.92	12.91	11.63	15.14
(222)	41.37	7.70	8.58	6.07	6.80	8.70	9.69	13.74	15.18
(2 $\bar{2}$ 2)	41.37	8.06	8.97	7.46	8.32	9.82	10.90	16.75	18.40
(312)	44.55	9.43	9.37	11.84	11.71	11.03	10.94	15.27	15.08
(3 $\bar{1}$ 2)	44.55	8.08	8.06	10.70	10.61	10.81	10.73		
(503)	46.06	10.49	9.87	13.49	12.62	12.37	11.61	16.60	15.52
(402)	51.24	11.14	8.74	14.26	13.32	17.75	13.75	21.80	16.86
(613)	51.62	13.79	10.61	18.58	14.15	20.52	15.60	29.33	22.10
(6 $\bar{1}$ 3)	51.62	13.39	10.30	23.18	17.50	24.64	18.59	25.46	19.30
(211)	54.31	10.97	7.69	15.19	10.57	15.58	10.85	26.75	18.36
(2 $\bar{1}$ 1)	54.31	14.26	9.94	14.26	9.94	14.26	9.95		
(512)	57.79	16.05	9.76	16.05	10.11	23.50	14.12	29.61	17.73
(5 $\bar{1}$ 2)	57.79	15.66	9.54	21.08	12.70	25.92	15.50	30.93	18.47
(301)	61.84	19.79	10.13	30.86	15.44	37.44	18.53	41.01	20.39
Mean and one standard deviation (Å)		9.62 ± 0.96		11.87 ± 2.52		14.97 ± 3.45		17.02 ± 2.21	

^a Where there are no entries, no measurements were taken.

Table IV: Summary of results of damage depth x_d^{II} for the four irradiations parallel to the [100] direction.

Bombarding Energy (eV) Average R_p (Å)		500 607		1000 624		1500 652		2000 760	
exposed ordered planes	θ (deg)	n.d. 002 (Å)	x_d^{II} (Å)	n.d. 002 (Å)	x_d^{II} (Å)	n.d. 002 (Å)	x_d^{II} (Å)	n.d. 002 (Å)	x_d^{II} (Å)
(112)	23.76	a		5.83	12.85	8.92	19.38		
(1 $\bar{1}$ 2)	23.76			6.67	14.63	5.92	13.05		
(213)	24.90			7.93	16.46	8.77	18.15	9.75	20.22
(2 $\bar{1}$ 3)	24.90			7.67	15.93	10.26	21.10	8.38	17.47
(101)	31.91	6.87	10.78	9.41	14.66	8.74	13.65	14.68	22.68
(334)	33.44	8.72	12.85	7.98	11.79	10.26	15.07	12.69	18.61
(3 $\bar{3}$ 4)	33.44			8.54	12.59	11.33	16.59	12.06	17.71
(323)	36.81	9.15	11.92	9.10	11.86	12.12	15.70	11.63	15.14
(3 $\bar{2}$ 3)	36.81	9.27	12.06	9.28	12.09	10.89	14.14	11.89	15.47
(222)	41.37	8.92	9.90	7.13	7.69	9.34	10.37	12.67	14.03
(2 $\bar{2}$ 2)	41.37	10.70	11.83	8.92	9.91	10.34	11.46	17.33	19.02
(312)	44.55	10.30	10.21	13.98	13.76	10.81	10.73	20.07	19.65
(3 $\bar{1}$ 2)	44.55	9.85	9.78	13.19	13.00	12.04	11.92	14.92	14.75
(503)	46.07	15.47	14.40	17.83	16.52	27.29	24.84	20.48	19.02
(402)	51.24	14.84	11.54	16.05	14.93	24.01	18.38	25.51	19.61
(613)	51.62	13.04	9.11	27.08	20.28	22.43	16.99		
(6 $\bar{1}$ 3)	51.62	17.58	12.16	26.22	19.67	26.34	19.80		
(211)	54.31	22.73	17.15	19.88	13.71	16.54	11.50	26.73	18.32
(2 $\bar{1}$ 1)	54.31	15.88	12.16	18.54	12.82				
(512)	57.79	20.92	12.60	24.96	14.92	33.88	19.94	39.23	23.10
(5 $\bar{1}$ 2)	57.79	22.84	13.69	22.34	13.43	27.45	16.37	45.00	26.24
(301)	61.84	26.23	13.24	35.66	17.65				
Mean and one standard deviation (Å)		12.08 $\pm$ 1.94		14.16 $\pm$ 2.9		15.96 $\pm$ 3.93		18.82 $\pm$ 3.26	

^a Where there are no entries, no measurements were taken.

Table V: Summary of results of damage depth x_d^I for the five irradiations parallel to the $[1\bar{1}2]$ direction.

Bombarding Energy (eV) Average R_1 (Å)		500 427		1000 498		1500 483		2000 492		2500 567	
exposed ordered planes	θ (deg)	$n \cdot d_{121}$ (Å)	x_d^I (Å)	$n \cdot d_{121}$ (Å)	x_d^I (Å)	$n \cdot d_{121}$ (Å)	x_d^I (Å)	$n \cdot d_{121}$ (Å)	x_d^I (Å)	$n \cdot d_{121}$ (Å)	x_d^I (Å)
(231)	13.2	2.1	8.48	3.8	12.26	2.5	10.25	4.0	13.51	4.9	19.36
(341)	20.1	3.2	8.54	4.2	10.93	3.1	8.33	6.1	15.35	6.1	15.90
(141)	18.1	2.1	6.21	4.2	12.15	3.7	11.03	6.7	19.11	6.5	18.74
(251)	19.5	2.1	5.73	3.6	9.74	5.4	14.44	5.7	15.46	6.8	18.13
(361)	22.2	3.7	8.83	3.4	8.02	4.2	9.87	8.3	19.19	9.5	21.83
(431)	29.5	3.9	6.44							9.2	15.67
(110)	39.6	7.6	8.96	9.4	10.95	11.6	13.55	16.8	17.93	22.2	23.31
(240)	35.7	4.2	5.72	6.9	9.33	7.6	10.30	13.1	17.44	20.0	26.25
(130)	36.5	5.5	7.31	6.9	9.08	8.6	11.24	12.6	16.69	21.1	26.77
(020)	43.4	a		8.3	8.57	9.2	9.41	11.4	13.55	24.4	24.23
(431)	59.1	14.8	8.49	16.6	9.48	28.7	15.87	24.9	17.45	33.5	18.52
(341)	54.4	11.7	8.09	15.8	10.86	20.0	13.61	23.7	19.40	32.5	21.61
(051)	59.1	19.0	10.74	16.0	9.16	19.3	10.94				
(271)	49.2	11.5	9.58	13.3	11.04	18.0	14.82	17.1	16.77	28.8	23.28
(161)	53.0	8.8	6.46	12.5	9.08	17.0	12.22	17.6	15.26	26.2	18.59
(141)	58.2	9.4	5.64	16.7	9.90	20.3	11.92	29.1	20.80	36.7	20.93
(251)	52.5	11.6	8.60	14.6	10.72	19.0	13.89	24.9	21.52	30.1	21.59
(031)	68.6	18.2	6.66	22.3	9.16					48.3	21.97
(231)	60.1	9.4	5.23	18.6	10.16	32.2	16.95	25.9	17.52	43.7	22.65
Mean and one standard deviation (Å)		7.54 ± 1.99		10.03 ± 1.18		12.27 ± 2.42		17.28 ± 2.34		21.05 ± 3.15	

^a Where there are no data, no measurements were taken.

Table VI: Summary of results of damage depth x_d^{II} for the five irradiations parallel to the $[1\bar{1}2]$ direction.

Bombarding Energy (eV) Average R_1 (Å)		500 427		1000 458		1500 483		2000 492		2500 567	
exposed ordered planes	θ (deg)	$n \cdot d_{121}^{II}$ (Å)	x_d^{II} (Å)	$n \cdot d_{121}^{II}$ (Å)	x_d^{II} (Å)	$n \cdot d_{121}^{II}$ (Å)	x_d^{II} (Å)	$n \cdot d_{121}^{II}$ (Å)	x_d^{II} (Å)	$n \cdot d_{121}^{II}$ (Å)	x_d^{II} (Å)
(231)	13.2	2.5	10.10	3.3	13.01	3.8	14.92	5.2	20.23	6.3	24.41
(341)	20.1	3.2	8.47	5.5	14.56	5.4	14.06	6.3	16.31	7.4	19.14
(141)	18.1	3.1	9.13	4.2	12.56	4.5	13.26	7.5	21.28	7.4	21.21
(251)	19.5	3.2	8.73	4.2	11.25	6.4	17.05	7.5	19.91	7.4	19.72
(361)	22.2	4.0	9.47	5.5	12.90	7.4	17.25	7.5	17.41	10.0	23.03
(431)	29.5	5.6	9.60	6.1	10.68	9.7	16.38	10.5	17.69	12.6	21.18
(110)	39.6	8.2	9.62	10.5	12.10	13.5	15.60	17.4	20.09	18.3	21.04
(240)	35.7	6.2	8.41	8.3	11.2	11.5	15.35	12.3	16.39	15.1	20.06
(130)	36.5	4.6	6.10	10.5	13.53	11.6	14.98	13.2	17.04	17.3	22.20
(020)	43.4	a		11.3	11.67	14.0	14.21	18.2	18.26	23.2	23.15
(431)	59.1	20.0	11.25	20.3	11.50	27.2	15.11				
(341)	54.4	14.6	9.98	19.3	13.06			29.4	19.69	32.2	21.43
(051)	59.1	21.2	11.39	23.0	12.82	26.3	14.65	30.3	16.72	38.6	21.11
(271)	49.2	13.3	11.02	18.5	15.10	19.2	15.73	24.6	19.89	31.2	25.07
(161)	51.0	14.5	10.46	16.6	11.96	19.2	13.75	24.6	17.38	30.2	21.25
(141)	58.2	12.5	7.45	23.0	13.30			29.6	16.96		
(291)	32.5	13.7	10.09	18.8	13.60	21.2	15.37	23.4	16.29	33.2	23.63
(031)	68.6	27.0	9.57								
(231)	60.1	20.2	10.90	22.8	12.30	28.6	15.19	31.2	16.48	45.3	24.78
Mean and one standard deviation (Å)		9.57 $\pm$ 1.40		12.62 $\pm$ 1.17		15.18 $\pm$ 1.10		18.14 $\pm$ 1.63		22.03 $\pm$ 1.81	

^a Where there are no data, no measurements were taken.

Figure 20: Mean damage depths $\bar{x}_d^I$ and $\bar{x}_d^{II}$ and one standard deviation of the experimental data as function of Ne^+ ion energy for the irradiations parallel to the $[\bar{7}6\bar{2}]$ direction.

Figure 21: Mean damage depths $\bar{x}_d^I$ and $\bar{x}_d^{II}$ and one standard deviation of the experimental data as function of Ne^+ ion energy for the irradiations parallel to the $[100]$ direction.

Figure 22: Mean damage depths x_d^I and x_d^{II} and one standard deviation of the experimental data as function of Ne^+ ion energy for the irradiations parallel to the $[1\bar{2}2]$ direction.

Table VII: The constants f and c fitted, by methods of least squares fit, to the expression $\bar{x}_d = fE^c$.

Depth of radiation damage in Å	$\bar{x}_d^I$			$\bar{x}_d^{II}$		
	[$\bar{7}6\bar{2}$]	[$\bar{1}\bar{2}2$]	[100]	[$\bar{7}6\bar{2}$]	[$\bar{1}\bar{2}2$]	[100]
Constants						
f	0.10 ± 0.04	0.10 ± 0.03	0.71 ± 0.10	0.05 ± 0.02	0.35 ± 0.05	1.50 ± 0.20
c	0.68 ± 0.04	0.67 ± 0.05	0.42 ± 0.05	0.80 ± 0.04	0.50 ± 0.05	0.33 ± 0.02

III.4 Discussion

The depth of radiation damage at a given energy in an amorphous target should be independent of the orientation of the target relative to the incident direction of the ion beam, i.e., no directional dependence of the radiation damage depth should be observed. However the experimental values of $\bar{x}_d^I$ and $\bar{x}_d^{II}$ exhibited a directional dependence which was attributed to the effect of channeling.

Visual inspection of a ball model of the ordered Ni_4Mo crystal showed that the atoms along the $[\bar{7}6\bar{2}]$ direction were situated in an almost random fashion, whereas along the $[\bar{1}\bar{2}2]$ direction the atoms were situated in a slightly more ordered fashion than along the $[\bar{7}6\bar{2}]$ direction. The $[100]$ direction was much more transparent than the other two directions. Figure 23 shows the position of the atoms along these directions. The critical half angle for channeling (ψ_c) in an $[hkl]$ direction, i.e., the angle between the incident direction of the ion beam and the normal to the (hkl) plane, beyond which ions will not channel, was calculated using the expression⁽⁹⁶⁾

$$\psi_c = (C^2 a_{TF}^2 Z_1 Z_2 e^2 / E d_{hkl}^3)^{1/4}, \quad (26)$$

where ψ_c is in, radians, the value of the constant C was taken to be 2.15,⁽⁹⁷⁾ a_{TF}^* is the Thomas-Fermi screening radius in $\text{\AA}$, e is the electronic charge ($e^2 = 14.4 \text{ eV} \cdot \text{\AA}$), E is the initial energy of the projectile in eV, and d_{hkl} is the atomic spacing along the $[hkl]$ direction in $\text{\AA}$. The

* The Thomas-Fermi screening radius is

$$a_{TF} = 0.8853 a_0 (Z_1^{1/2} + Z_2^{1/2})^{-2/3},$$
 where a_0 is the Bohr radius ($0.529 \text{ \AA}$).

Figure 23: Photographs of a ball model of an ordered Ni_4Mo crystal taken along the $[\bar{7}6\bar{2}]$, $[\bar{1}\bar{2}2]$ and $[100]$ crystallographic directions.

calculated values of ψ_c (in degrees) as a function of incident energy E for each of the $[\bar{7}6\bar{2}]$, $[\bar{1}\bar{2}2]$, and $[100]$ directions are shown in Fig. 24. These calculated values of ψ_c show that the acceptance angles for channeling, i.e., angles beyond which ions will not channel, were the largest for Ne^+ ions incident in the $[100]$ direction of the Ni_4Mo crystal, and that for each direction the ψ_c values were larger at the lower energies. A plot of the $\bar{x}_d^{\text{II}}$ values, which showed a greater directional dependence than the $\bar{x}_d^{\text{I}}$ values, versus the calculated values of ψ_c is shown in Fig. 25. From these observations the following was concluded:

1. ion channeling was observed in the $[100]$ direction;
2. channeling, to a lesser extent than in the $[100]$ direction, was also observed in the $[\bar{1}\bar{2}2]$ direction;
3. no channeling was observed in the $[\bar{7}6\bar{2}]$ direction; and
4. as the incident Ne^+ ion energy was increased the effect of channeling was reduced.

The energy loss mechanism⁽⁹⁸⁾ of an ion in a channeled direction is dominantly electronic. The energy dependence of the range of a low-energy ion projectile that loses its energy solely by inelastic processes in an amorphous target is proportional to $E^{1/2}$.⁽⁹⁶⁾ But since the values of the impact parameters in the collisions of channeled projectiles, with the atoms that constitute the channel, are greater than the impact parameters in the collisions of projectiles with atoms in an amorphous target, a channeled projectile loses less energy in a collision event than a projectile in an amorphous target loses in a collision event. Theoretical⁽⁹⁶⁾ and experimental⁽⁹⁹⁾ reports showed that the ratio of the stopping power for a channeled projectile to the electronic stopping power in an amorphous

Figure 24: Calculated critical half angles for channeling along the $[762]$ $[122]$ and $[100]$ crystallographic channels of ordered Ni_4Mo crystal as a function of incident Ne^+ ions energy.

Figure 25: The experimental $\bar{x}_d^{II}$ values for all the bombardment directions and energies employed versus the calculated values of ψ_c .

target was smaller than unity. These arguments, and the energy dependence of $\bar{x}_d^{II}$ shown in Table VII strengthen our conclusion that channeling was taking place in the [100] direction ($\bar{x}_d^{II} \propto E^{0.33 \pm 0.02}$), and less channeling was taking place in the $[\bar{1}\bar{2}2]$ direction ($\bar{x}_d^{II} \propto E^{0.50 \pm 0.05}$), and no channeling took place in the $[\bar{7}6\bar{2}]$ direction ($\bar{x}_d^{II} \propto E^{0.80 \pm 0.04}$). The last conclusion, i.e., no channeling, was also based on the agreement of the $\bar{x}_d^I \propto E^{0.67 \pm 0.04}$ relation with the theoretical predictions⁽¹⁵⁾ of transport of energetic ions in amorphous target. Therefore the experimental results for the $[\bar{7}6\bar{2}]$ direction will be compared with the theoretical predictions which were derived for amorphous solids.

Before comparing the experimental results to the theoretical predictions we will examine the effects of focused RCSs and sputtering on the experimental values of x_d . The passage of a focused RCS in an ordered alloy leaves a linear disordered trail of atoms in the crystal.⁽¹²⁾ Theoretical studies^(16,100) based on the energy lost in a single replacement in Cu, and the many-body computer simulation⁽¹⁰¹⁾ model of low-energy collisions, suggested a maximum chain length of about 60 Å. Experimental ranges that have been proposed, based on indirect measurements, differ greatly from a few tens to a several hundreds angstrom.^(102,103) But, as stated before, the effort to determine the exact positions of the atoms after an irradiation was unsuccessful. Therefore, we could not detect the RCSs events in the irradiated Ni₄Mo alloy, and thus could not determine their effect on the values of $\bar{x}_d$ that we measured.

The second effect that could have had an influence on our measurements was the sputtering of metal from the surface of the FIM specimen. In order to estimate the effect of sputtering, we calculated the sputtering yield

using the semi-empirical expression developed by Smith⁽¹⁰⁴⁾

$$G = \frac{20}{U_0} Z_1^2 Z_2^2 \frac{M_1}{M_2} \frac{E}{(E + 50 Z_1 Z_2)^2} \quad , \quad (27)$$

where G is in atoms per incident ion, E and the surface binding energy (U_0) are both in eV. The general shape of the energy dependent sputtering-yield curve was based on Sigmund's model,⁽¹⁰⁵⁾ whereas the magnitude was derived primarily from experimental data in the low-energy régime.⁽¹⁰⁴⁾ Using the cohesive energy of pure Ni of 5 eV as an approximation for U_0 , we obtained G values that varied between 0.2 atom/ion for 5000 eV Ne^+ ions and 0.7 atom/ion for 2500 eV Ne^+ ions. For a dose of $3 \cdot 10^{15}$ ion cm^{-2} , and a surface atomic density of $2 \cdot 10^{15}$ atom cm^{-2} for Ni_4Mo , we calculated that the sputtering amounted to less than one atomic layer even for the highest energy used. This can result in an error of less than 10% in the measured values of $\bar{x}_d$.

In order to compare the experimental results to the theoretical predictions, we used Winterbon's tabulations and the TRIM program to calculate the first four moments of the elastically deposited energy distribution. Winterbon's method, for estimating results for polyatomic targets from monoatomic target calculations, assumes a fictitious monoatomic target with Z and M equal to the averaged values for the real target material, for the Ni_4Mo $M = 66.15$ and $Z = 30.8$. In the TRIM program, the projectile collided at random, in a ratio of 1 to 4, with the Mo and the Ni atoms, respectively. Each range distribution was based on 2000 projectile histories, and a projectile history was terminated when its energy fell below one eV. The result of the Winterbon and the TRIM calculations of $\bar{x}_d$ and the straggling

Δx_d , i.e., the root mean square of the second moment of the distribution about the mean, of the elastically deposited energy distribution are summarized in Table VIII; the quantities $\bar{x}_d^I$ and $\bar{x}_d^{II}$ and σ for the radiation damage depths were measured for irradiations parallel to the $[\bar{7}6\bar{2}]$ direction. As was shown in Fig. 11 the value of the quantity $\bar{x}_d^I$ indicated a depth which was greater than the mode of the radiation damage profile, and the $\bar{x}_d^{II}$ value was even greater than the $\bar{x}_d^I$ value. This and the skewed nature of the radiation damage profiles in this energy régime⁽¹⁵⁾ indicated that the values of $\bar{x}_d^I$ were approximately the same as the mean depth of the radiation damage values. And indeed, the $\bar{x}_d^I$ values shown in Table VIII agree well with the theoretical values of the mean values of the elastically deposited energy profiles.

The experimental depth of radiation damage-energy relation, written in the LSS dimensionless reduced parameters, that we obtained for the irradiations parallel to the $[\bar{7}6\bar{2}]$ direction was

$$\rho_d^I = (0.42 \pm 0.04) \epsilon^{0.68 \pm 0.04} . \quad (28)$$

The path length-energy relation obtained by WSS using the LSS approach was

$$\rho_{p1} = 1.53 \epsilon^{2/3} . \quad (29)$$

Both, the experimental and the theoretical relations have the same energy dependence $\rho = g \epsilon^{2/3}$. The $2/3$ power of ϵ indicated that in the pertinent energy régime ($0.01 < \epsilon < 0.05$) the stopping mechanism was primarily nuclear.⁽¹⁵⁾ The different values of the constant g , i.e., 1.53 and 0.42, showed that the predicted x_{p1} was much greater than the experimental depth

Table VIII: Calculated Mean and straggling of the elastically deposited energy distribution, and the experimental mean and one standard deviation of the radiation damage depths $\bar{x}_d^I$ and $\bar{x}_d^{II}$ for the $[\bar{7}6\bar{2}]$ direction.

Energy (eV)	Calculated values of Mean and straggling (Å)		Experimental Mean and one standard deviation for the irradiation on parallel to the $[\bar{7}6\bar{2}]$ direction	
	TRIM	Winterbon	$\bar{x}_d^I$	$\bar{x}_d^{II}$
500	5.7 ± 4.3	6.5 ± 5.0	6.4 ± 1.3	6.9 ± 1.8
1000	9.5 ± 7.1	10.3 ± 8.0	10.3 ± 2.8	10.9 ± 3.1
1500	13.4 ± 9.8	13.6 ± 10.4	13.6 ± 4.2	16.6 ± 4.6
2000	16.5 ± 12.3	16.7 ± 12.7	18.7 ± 5.7	25.4 ± 6.5

of radiation damage. This was in agreement with theory^(15,4) which predicted large angle scattering events for $M_1 < M_2$ in this energy régime, thus causing the large difference between the theoretical $\bar{\rho}_{pl}$ values and the experimental $\bar{\rho}_d$ values. The trajectories of fifteen 1000 eV Ne^+ ions slowing down in an amorphous Ni_4Mo alloy were obtained employing the TRIM program and are shown in Fig. 26. The trajectories in this figure exhibit the large angle deflections suffered by the projectile ions in the collision events. The consequence of the large angle deflections was to shorten $\bar{x}_{pr}$ and $\bar{x}_d$ relative to the $\bar{x}_{pl}$ values. These range quantities were obtained as a function of the incident Ne^+ ions energy employing the TRIM program and are shown in Fig. 27.

The TRIM program also provided the total electronic energy loss of the projectiles in the target. An expression for the electronic energy loss, which was suggested by Oen and Robinson⁽²²⁾

$$\Delta E_e(E, r_0) = \frac{0.045kE^{1/2}}{ra_{TF}} \exp\left(-\frac{0.3r_0}{a_{TF}}\right) \text{ eV} , \quad (30)$$

where r_0 is the distance of closest approach of the projectile to a target atom in a collision event, was used in the TRIM program. This expression, rather than Lindhard-Scharff's expression ($kE^{1/2}$) was used in the TRIM program because it described better the electronic energy loss in the energy régime of interest.⁽¹⁰⁶⁾ Figure 28 shows the fraction of the total energy lost in electronic processes as a function of the incident Ne^+ ion energy obtained from the TRIM program. It was clear that the energy lost in electronic processes in this energy régime was less than 10% of the total energy. This confirmed our conclusion that in this régime,

Figure 26: Trajectories of twenty Na^+ ions, with incident energy of 1000 eV, slowing down in amorphous Ni_4Mo as obtained from the TRIM program.

Figure 27: Mean values of path length ($\bar{\rho}_{pl}$), projected range ($\bar{\rho}_{pr}$) and elastically deposited energy ($\bar{\rho}_d$) distributions versus incident Ne^+ ion energy E for amorphous Ni_4Mo as obtained from the TRIM program.

Figure 28: The fraction of energy lost in electronic processes by the Ne^+ ions penetrating an amorphous Ni_4Mo alloy as a function of incident Ne^+ ion energy.

nuclear stopping was the primary mechanism by which the projectiles lost their energy.

The effective stopping powers $(d\epsilon/d\bar{\rho}_d)_{\text{eff}}$, where $\bar{\rho}_d$ is the mean depth of the elastically deposited energy in the LSS dimensionless units, were derived from the calculated values, and were fitted by means of a least squares fit to an expression of the form

$$(d\epsilon/d\bar{\rho}_d)_{\text{eff}} = a\epsilon^D. \quad (31)$$

The term effective was used in connection with $(d\epsilon/d\bar{\rho}_d)_{\text{eff}}$ to distinguish between this stopping power, which is related to $\bar{\rho}_d$ and the theoretical stopping power $(d\epsilon/d\rho_{pl})$ which is related to ρ_{pl} . Since the observed radiation damage (disordered crystal) resulted from nuclear collisions only, a comparison between the experimental effective nuclear stopping power and the calculated predictions can be made. The effective stopping powers were:

$$(1) \text{ TRIM } (d\epsilon/d\bar{\rho}_d)_{\text{eff}} = 2.34\epsilon^{0.23}, \quad (32)$$

$$(2) \text{ Winterbon } (d\epsilon/d\bar{\rho}_d)_{\text{eff}} = 3.49\epsilon^{0.32}, \quad (33)$$

and

$$(3) \text{ Experimental } (d\epsilon/d\bar{\rho}_d^I)_{\text{eff}} = (3.50 \pm 0.4)\epsilon^{0.32 \pm 0.04}. \quad (34)$$

These relations, and the nuclear stopping powers of the WSS approach (eqn. 8), and the WHB approach (eqn. 10) with the constants $A = 0.56$, $B = 1.18$ and $C = 0.63$, and Kalbitzer et al. relation (eqn. 11) are shown in Fig. 29.

Figure 29: A summary of experimental and theoretical nuclear stopping powers as a function of incident Ne^+ ions energy.

Comparison between the effective nuclear stopping power and Kalbitzer et al. nuclear stopping power (eqn. 11), which was derived for the same energy régime, shows a large discrepancy. This discrepancy was due to the fact that in the present experiments, where the target-projectile mass ratio ($v = M_2/M_1$) was ~ 3 , the Ne^+ ions suffered large angle deflections in the collisions. Whereas, in the case of Kalbitzer et al., where the projectile ions were heavier than the target atoms, $v \sim 0.5$, the ions did not suffer large angle deflections and their trajectories were approximately straight. This shows that a universal nuclear stopping power for every energy régime has to be a function of v as well as ϵ . This point was included in the two calculational technique which were in a good agreement with our results.

III.5 Summary

1. A novel technique for measuring the depth of radiation damage in order-disorder alloys using the FIM has been described. The technique was based on the difference in the image contrast between an ordered and a disordered alloy as observed by the FIM.

2. The lower energy limit of this technique was determined by the ability of the incident ions to penetrate the target and to produce a measurable amount of radiation damage. The upper energy limit was determined by the requirement that the extent of radiation damage had to be smaller than the radius of the specimen. In the present experiment measurements were performed for ϵ values that varied from 0.01 to 0.05, using the ordered alloy Ni_3Mo as a target and 500 to 2500 eV Ne^+ ions as the projectiles.

3. Three sets of irradiation experiments were performed. In each set the bombardment direction was parallel to a different crystallographic

direction, i.e., [100], $[\bar{1}\bar{2}2]$, and $[\bar{7}6\bar{2}]$. A directional dependence of the depth of radiation damage $\bar{x}_d$ was observed and was attributed to the effect of channeled ions. The values of $\bar{x}_d$ indicated that the Ne^+ ions channeled in the [100] direction more than in the $[\bar{1}\bar{2}2]$ direction, and they did not channel in the $[\bar{7}6\bar{2}]$ direction. The ion channeling was most pronounced at the lower energies employed (500 and 1000 eV). As the ion's energy was increased (1500 to 2000 eV), no channeling was observed. These observations agreed with the theoretical prediction⁽⁹⁶⁾ for the critical half angle for channeling as a function of crystallographic directions and the incident ions energy. The energy dependence of $\bar{x}_d$, for the irradiations parallel to the [100] direction, in which the channeling effect was most pronounced, was proportional to $E^{0.33 \pm 0.02}$. This dependence agreed with the assumption⁽⁹⁸⁾ that the rate of electronic energy loss in a channeled direction was smaller than in an amorphous material.

4. The experimental effective stopping power for the non-channeling direction, i.e., the $[\bar{7}6\bar{2}]$ direction, was derived from the mean depth of radiation damage relation (eqn. 28)

$$\bar{p}_d = (0.42 \pm 0.04) \epsilon^{0.68 \pm 0.04},$$

was (eqn. 34)

$$(d\epsilon/d\bar{p}_d)_{\text{eff}} = (3.50 \pm 0.4) \epsilon^{0.32 \pm 0.04}.$$

These relations were in a good agreement with the theoretical predictions of Winterbon's tabulations⁽²⁶⁾ which were based on the WSS⁽¹⁵⁾ approach, and the results of the computer simulation of the transport of energetic

ions in an amorphous target coded TRIM⁽²⁵⁾ which were based on the WHB approach.⁽⁴⁾

IV. RADIATION DAMAGE PROFILES IN THE ORDER-DISORDER ALLOY Pt_3Co

The measurements of radiation damage profiles in the order-disorder alloy Pt_3Co were based on the change in the Bragg-Williams long-range order-parameter (S). Radiation damage profiles represent the total amount of disorder produced in the lattice, i.e., the number of displaced and replaced atoms, as a function of distance (x) from the irradiated surface of the specimen along the bombardment direction. Ordered Pt_3Co FIM specimens were irradiated in situ at 30 K with 500 to 2500 eV Ne^+ ions. The penetration of the projectile Ne^+ ions into the specimen disordered the Pt_3Co lattice and reduced the local value of S from its pre-irradiation value (S_0) to a value S_d which was a function of x . After an irradiation each specimen was dissected on an atom-by-atom basis employing the pulse field-evaporation technique, and the FIM images were photographed between each pulse. The number of misplaced atoms, i.e., displaced and replaced atoms, on each atomic plane was determined using all the micrographs in the evaporation sequence of that atomic plane, and S_d of each atomic plane was determined. The value of S_0 in the same atomic plane was also measured. This allowed us to determine the change in S , i.e., $\Delta S_d = S_0 - S_d$, as a function of x . The resulting ΔS_d versus x plot constituted a radiation damage profile.

The following sections describe the experimental method for determining ΔS_d as a function of x .

IV.1 Determination of S in the Order-Disorder Alloy Pt_3Co

The state of the long-range order in an alloy can be specified in terms of a system of interpenetrating sublattices and a set of occupational parameters describing the distribution of the atomic species on each

sublattice.⁽¹⁰⁷⁾ In an ideal ordered alloy each atomic species occupies completely one of the sublattices, thus the different atoms have a regular periodic arrangement with respect to one another. When part of a sublattice is occupied by a wrong atomic species the alloy is partially ordered. The FIM with its ability to resolve and to locate individual atoms on the surface of a crystal, is suitable for determining S.⁽⁹¹⁾ An atom-by-atom dissection of an FIM specimen, employing the pulse field-evaporation technique, allows a highly controlled examination of the local atomic arrangement in a specimen.

An ordered binary alloy has two types of atomic positions which are designated as α - and β -sites. In an ideal stoichiometric composition with perfect long-range order, the α -sites are all occupied by A-atoms and the β -sites by B-atoms. In general case, X_A and X_B represent the atomic fraction of atoms in a sample of the alloy; i.e., $X_A + X_B = 1$. If y_α and y_β are the respective fractions of the α -sites and the β -sites in the lattice, then $y_\alpha + y_\beta = 1$. In order to define S, Nix and Shockley⁽¹⁰⁷⁾ introduced the following four useful parameters:

r_α = fraction of α -sites occupied by the right atom (A),

w_α = fraction of α -sites occupied by the wrong atom (B),

r_β = fraction of β -sites occupied by the right atom (B),

w_β = fraction of β -sites occupied by the wrong atom (A).

Two relations between these parameters are obvious:

$$r_\alpha + w_\alpha = 1, \quad (35a)$$

and

$$r_{\beta} + w_{\beta} = 1 . \quad (35b)$$

The fraction of the sites occupied by A-atoms must be equal to the fraction of A-atoms, and the fraction of the sites occupied by B-atoms must be equal to the fraction of B-atoms in the sample. These two equivalent conditions are expressed by:

$$y_{\alpha} r_{\alpha} + y_{\beta} w_{\beta} = X_A , \quad (36a)$$

and

$$y_{\beta} r_{\beta} + y_{\alpha} w_{\alpha} = X_B . \quad (36b)$$

The value of S which is linearly proportional to $(r_{\alpha} + r_{\beta})$, is defined⁽¹⁰⁸⁾ as $S = 0$ for a completely random arrangement, and as $S = 1$ for a stoichiometric composition with $r_{\alpha} = r_{\beta} = 1$. Using the linear dependence of S on $(r_{\alpha} + r_{\beta})$ and eqns. (36), S is given by the expressions

$$S = \frac{r_{\alpha} - X_A}{y_{\beta}} = \frac{r_{\beta} - X_B}{y_{\alpha}} . \quad (37)$$

It was emphasized⁽¹⁰⁸⁾ that the value of S can reach unity only for stoichiometric composition, i.e., $X_A = y_{\alpha}$ and $X_B = y_{\beta}$; for a nonstoichiometric composition the value of S is always less than unity.

IV.1.1 Specimen Preparation

An ingot of the alloy Pt_3Co was prepared at Cornell University from 99.99 wt.% pure Pt (obtained from Engelhard Industries) and 99.999 wt.% pure Co (obtained from United Mineral Corp.) by arc melting in an argon atmosphere. After homogenizing in a vacuum of $5 \cdot 10^{-6}$ Torr for 100 hr at

1200°C the alloy was hot swaged into a 0.5 cm diameter rod, and was then drawn into 0.0127 cm diameter wire. The composition of the wire was determined by Ledoux and Company to be 76.06 at.% Pt and 23.93 at.% Co. The wire was cut into 10 cm long specimens which were sealed in evacuated quartz tubes at 10^{-5} Torr. In addition a small number of Pt_3Co wires, 0.0127 cm in diameter, where the composition was determined to be within few-tenths of an atomic percent of stoichiometry,⁽¹⁰⁹⁾ were kindly supplied by Professor T.-T. Tsong.⁽⁹¹⁾ Tsong's wires also were cut into 10 cm long specimens and were then sealed in evacuated quartz tubes at a pressure of 10^{-5} Torr. Following the ordering treatment given by Berg *et al.*,⁽¹⁰⁹⁾ Tsong's specimens were recrystallized at 1000°C for two hours and given the following annealing treatment to obtain $S = 1$: Annealed at 685°C for 4 days; 660°C for 7 days; 620°C for 3 days; 600°C for 2 days; 575°C for 2 days; 550°C for 3 days, 525°C for 3 days; 500°C for 3 days; 475°C for 4 days; 450°C for 2 days; 425°C for 3 days; and 400°C for 3 days. Berg and Cohen⁽¹¹⁰⁾ noted that the critical temperature (T_C) for ordering Pt_3Co may vary sharply with composition near stoichiometry, and the fact that Menzinger and Paoletti⁽¹¹¹⁾ reported that "no ordering was detected in a sample with a composition of 24.88 at.% Co that had been annealed for over 100 hours at 650°C while practically complete order $S = 0.9 \pm 0.1$ was achieved by annealing the samples at 600°C for a time between 100 and 600 hours." Since the alloy that was prepared at Cornell University was slightly rich in Pt, the ordering treatment of Berg *et al.*⁽¹⁰⁹⁾ was modified. The specimens were annealed for longer periods of time at the higher temperatures, i.e., 7 days instead of 2 days at each of the following temperatures: 620°C; 600°C; and 575°C.

FIM specimens were prepared by electropolishing one cm long wires in a molten salt solution of four parts NaNO_3 and one part NaCl (by weight) maintained at 350°C .⁽⁹¹⁾ A potential of 3 Vdc was applied, with Pt as the cathode and the specimen as the anode. To ensure that the hot molten solution did not affect the state of order in the specimen, the temperature of the specimen was measured, using a Chromel-Alumel thermocouple, 0.0076 cm in diameter. The thermocouple was connected to the specimen by dipping the tip of the thermocouple together with the specimen wire in the molten solution and rapidly removing it from the solution. A small amount of the salt solution solidified around the specimen and the tip of the thermocouple, joining the two 6 mm above the bottom end of the specimen. The temperature was measured when 5 mm of the specimen was inside the molten salt, leaving the thermocouple 1 mm above the solution. The temperature rose to 280°C when the specimen was held in the solution for one minute. Immersion for 2-5 minutes did not increase the temperature any further. Also, when a current of 100 mA was applied to the specimen for half a minute, the temperature did not rise above 250°C . An FIM specimen was prepared by dipping the Pt_3Co wire in the molten salt solution for a short period of time, about one sec each period, which would not affect the degree of long-range order in the specimen. When examined at a magnification of 400x, employing an optical microscope, a good specimen had the appearance of a well-sharpened pencil. The electropolishing procedure was continued until a satisfactory degree of sharpness was achieved. The specimen was transferred to the FIM, which was then evacuated to a pressure of $6 \cdot 10^{-6}$ Torr. Next the specimen was field-evaporated continuously at ~ 70 K, and then pulse field-evaporated at 20 K to a final end-form at a background pressure of $1 \cdot 10^{-4}$ Torr He.

IV.1.2 Data Recording Procedures

When first observed, the FIM images of the ordered Pt_3Co specimens were not atomically regular. Many atom layers were removed by the pulse field-evaporation technique before the images were regular on an atomic scale. Then the specimens were carefully pulse field-evaporated and FIM images were photographed between each pulse. The pulse height and duration were adjusted so that on the average, three atoms were removed for each pulse from the $\{220\}$ atomic planes. A total of 6000 frames of film were photographed for each measurement. The developed ciné film was analyzed with the aid of the Vanguard motion analyzer. The number of misplaced atoms on each plane was determined using all the micrographs in an evaporation sequence.

IV.1.3 Analysis Procedure

The Pt_3Co alloy has a face-centered cubic (fcc) structure. In the ordered phase the Pt_3Co has the structure of an Ll_2 type lattice.⁽¹¹²⁾ In this lattice the Co atoms occupy the sites at the cube corners, and the Pt atoms occupy the face-centered positions. The unit cubic cell is shown in Fig. 30. Three types of planes exist in the ordered Pt_3Co alloy. Planes of the first type, with Miller indices* of mixed parity consisting of half Co and half Pt atoms, were designated as H-planes. Planes of the second type, with Miller indices of mixed parity, interleaving the H-planes, and consisting of all Pt atoms, were designated as P-planes. Planes of the third type, with Miller indices of unmixed parity, consisting of one Co atom to three Pt atoms, were designated as U-planes. Figures 31, 32, and

* The Miller indices given in this chapter refer to the fcc unit cell.

Figure 30: A cubic unit cell showing the location of the Co and Pt atoms in the ordered alloy Pt_3Co .

33 exhibit photographs of ball models, which were built on a (200) plane, of a (220) H-plane, a (220) P-plane, and a (111) U-plane, respectively.

In a positive black and white print a Pt atom was imaged as a solid white spot, whereas a Co atom did not produce an observable contrast effect.^(74,113) When a Pt atom occupied a Co-site its image appeared as a white spot on that Co-site, and when a Co atom occupied a Pt-site, the image of the Pt atom was missing in the FIM micrographs. Figures 34(a) and 35(a) show the atomic configuration of Pt and Co atoms in a (220) H-plane and a (220) P-plane, respectively. Figures 34(b) and 35(b) show the same configurations as in (a) where the invisible Co atoms have been removed, i.e., showing only the Pt atoms which were observable in the FIM micrographs. As shown in these figures misplaced Pt atoms, i.e., Pt atoms on Co-sites and Pt atoms in interstitial sites, imaged as bright spots in between the normal Pt rows. Cobalt atoms on Pt-sites did not produce an observable contrast effect (λ -contrast effect[†]). In addition, a distinction between Pt atoms on Co-sites (replacement events) and Pt atoms in interstitial sites (displacement event) was made (see numbers 3 and 4 in Fig. 34), whenever it was possible. In the ordered alloy most of the images of the misplaced Pt atoms appeared on Co-sites, i.e., replacement events. Thus we were able to detect replacement events between the Pt and the Co sublattices, but only displacement events produced in the Pt sublattice were detected.

The atomic arrangement for each atomic plane was obtained from the micrographs that were taken during the dissection of the specimen. The

[†] λ -contrast effect can result from: (1) a vacant lattice site; or (2) from Co atom in a Pt-site. The observed contrast effect in a positive print, is a dark spot.

Figure 31: A photograph of a ball model of a (220) H-plane. The white balls represent platinum atoms and the black balls represent cobalt atoms. An H-plane consists of equal number of platinum and cobalt atoms.

Figure 32: A photograph of a ball model of a (220) P-plane. The white balls represent platinum atoms and the black balls represent cobalt atoms. A P-plane consists of all platinum atoms.

Figure 33: A photograph of a ball model of a (111) U-plane. The white balls represent platinum atoms and the black balls represent cobalt atoms. A U-plane consists of one cobalt atom to three platinum atoms.

- Figure 34: (a) The atomic positions of platinum and cobalt atoms in a (220) H-plane;
- (b) The same (220) H-plane as observed in the FIM images, i.e., only the Pt atoms which were observed in the FIM images are shown. The number (1) indicates a cobalt atom sitting in a Pt-site, the number (2) indicates a vacant platinum lattice site, the number (3) indicates a platinum atom sitting in an interstitial site, and the number (4) indicates a platinum atom sitting in a cobalt site.

(a)

(b)

● Pt

○ Co

Figure 35: (a) The atomic positions of platinum and cobalt atoms in a (220) P-plane; (b) The same (220) P-plane as observed in the FIM images. The number (1) indicates a cobalt atom sitting in a Pt-site, the number (2) indicates a vacant Pt lattice site, and the number (3) indicates a platinum atom sitting in an interstitial site.

● Pt
○ Co

number of misplaced atoms, λ -contrast effects, and Pt-sites were counted for each plane. These numbers were then used in eqn. (37) to obtain the values of S.

IV.1.4 Experimental Results

Planes with a large number (~ 80) of atoms and good atomic resolution were needed for measuring S. Figure 36 shows an FIM micrograph of an image of the ordered Pt_3Co alloy, and Fig. 37 shows the crystallographic map of this image; the local tip radii are indicated along the main zone axes. The planes with the most number of atoms per plane were the $\{111\}$, $\{002\}$ and $\{220\}$ planes. The $\{111\}$ and $\{002\}$ planes did not have good atomic resolution, whereas the $\{220\}$ planes had good atomic resolution. Therefore The $\{220\}$ planes were examined in detail, and the value of S was determined for these planes. In addition, the value of S was also measured in nine planes which were contained within the $1\bar{1}1$ -002-111 triangle.

A typical evaporation sequence of a $\{220\}$ H-plane is shown in Fig. 38(a-e), and the atomic arrangement determined from this sequence is shown in Fig. 38(f). Figure 39(a-e) shows a typical evaporation sequence for a $\{220\}$ P-plane, and its atomic arrangement is shown in Fig. 39(f). Each of the H- and P-planes was reconstructed as shown in figures 38(f) and 39(f), and the number of replaced and displaced atoms, and the number of λ -contrast effects was registered for each plane. In a P-plane, the resolution was good enough to detect λ -contrast effects only when the plane was greatly reduced by the pulse field-evaporation technique relative to its original size; therefore only a small portion of a P-plane was examined for λ -contrast effects.

The $\{220\}$ H-planes examined had, on the average, about 100 Pt atoms, and therefore an average of 100 Co-sites. The $\{220\}$ P-planes were examined

Figure 36: An helium FIM micrograph of an ordered Pt₃Co recorded at a specimen temperature of 30 K.

Figure 37: The crystallographic map of the FIM micrograph shown in Fig. 36. The numbers between the zone poles are the values of the local tip radii along the zone axes which are indicated by the arrows.

Figure 38: Five FIM micrographs showing the atom-by-atom dissection of a (220) H-plane of the Pt_3Co alloy, recorded at a specimen temperature of 30 K. The position of the Pt atoms are illustrated below each micrograph. The arrangement of the Pt atom in that entire H-plane is given in (f).

Figure 39: Five FIM micrographs showing the atom-by-atom dissection of a (220) P-plane of the Pt₃Co alloy, recorded at a specimen temperature of 30 K. The position of the Pt atoms are illustrated below each micrograph. The arrangement of the Pt atoms in that entire P-plane is given in (f).

when the number of Pt atoms was reduced to about 50 Pt atoms. The value of S was calculated for two consecutive H- and P-planes at a time. When the number of λ -contrast effects on both planes was greater than the number of misplaced Pt atoms on both planes, it was used to calculate the value of S; otherwise the number of misplaced Pt atoms were used. Equation (37) with the appropriate subscripts was used to calculate the values of S. When the number of misplaced Pt atoms, i.e., extra bright spots in between the normal Pt rows, was used eqn. (37) became

$$S = \frac{r_{Co} - X_{Co}}{y_{Pt}} . \quad (38)$$

For example, for a Cornell specimen with $X_{Co} = 0.239$ and $X_{Pt} = 0.761$, and P-plane with 50 Pt sites and H-plane with 100 Pt-sites, let the number of misplaced Pt atoms on two consecutive H- and P-planes be 4, then

$$r_{Co} = \frac{100-4}{100} , \text{ and } S = \frac{0.960-0.239}{0.761} = 0.947 .$$

When the number of λ -contrast effects was greater than the number of misplaced Pt atoms, the value of S was calculated using

$$S = \frac{r_{Pt} - X_{Pt}}{y_{Co}} . \quad (39)$$

For example, let the number of λ -contrast effects on the above two consecutive H- and P-planes be 3; then

$$r_{Pt} = \frac{150-3}{150} , \text{ and } S = \frac{0.973-0.761}{0.239} = 0.887 .$$

The results for the specimens obtained from Tsong and for the specimens prepared at Cornell University are summarized in Tables IX and X, respectively. The values of S were calculated for the planes (202), (204), (206), (406), (113), ($\bar{1}\bar{1}3$), (115), (224), and ($2\bar{2}4$) contained within the $\bar{1}\bar{1}1$ -002-111 triangle. The smallest and largest values of S , and the number of Pt- and Co-sites detected in each plane are also given in these Tables. The weighted average values of S were 0.96 ± 0.05 and 0.95 ± 0.05 for the Tsong and Cornell specimens, respectively. In addition, the values of S were measured, for both specimens, along the normal to the (202) planes to a depth (n) of about $270 \text{ \AA}$. The examination of 5000 and 3498 Co-sites resulted in an average value of S of 0.94 ± 0.04 for both specimens. An error of 10% in the number of Pt-sites resulted in an error of less than 1% in the values of S . The values of S as a function of depth (n) in the crystal for the Tsong and Cornell specimens are given in Figs. 40 and 41, respectively.

IV.1.5 Discussion

The average values of S obtained for the region contained within the $\bar{1}\bar{1}1$ -002-111 triangle were 0.96 ± 0.05 and 0.95 ± 0.05 for the Tsong and Cornell specimens, respectively. These values of S were somewhat greater than the S values ($S = 0.94 \pm 0.03$) for the (202) planes in these specimens. On most of the planes in this region it was difficult to identify all the misplaced atoms. In some of the planes the atomic resolution was not good enough, and in others there was irregular behavior in the evaporation of atoms during the pulse field-evaporation sequence which made it difficult to accurately detect all of the misplaced atoms. This irregular field-evaporation behavior did not occur in the (220) planes, where the atoms

Table IX: The experimental values of S measured for different atomic planes in the specimen supplied by Tsong.

Plane	Number of H- planes examined	Number of P- planes examined	Number of U- planes examined	Pt-sites on P-planes	Pt-sites on H-planes	Pt-sites on U-planes	Lowest S value	Highest S value	Total Pt- sites	Total Co- sites	Average S value
(202)	50	50		55	100		0.89	0.97	7,750	5,000	0.94 ± 0.03
(204)	24	24		40	52		0.86	1.00	2,208	1,248	0.98 ± 0.05
(206)	24	24		96	48		0.92	1.00	3,456	1,152	0.98 ± 0.05
(406)	20	20		56	18		0.89	1.00	1,480	360	0.91 ± 0.05
(224)	10	10		70	56		0.93	1.00	1,260	560	0.94 ± 0.05
(2 $\bar{2}$ 4)	10	10		80	64		0.93	1.00	1,440	640	0.93 ± 0.05
(113)			27			47	0.89	1.00	1,269	423	0.96 ± 0.05
(1 $\bar{1}$ 3)			27			47	0.89	1.00	1,269	423	0.94 ± 0.05
(115)			40			26	0.85	1.00	1,040	347	0.98 ± 0.05
(1 $\bar{1}$ 5)			40			26	0.85	1.00	1,040	347	0.98 ± 0.05
Total									22,212	10,500	0.96 ± 0.05

Table X: The experimental values of S measured for different atomic planes in the specimens prepared at Cornell.

Plane	Number of H-planes examined	Number of P-planes examined	Number of U-planes examined	Pt-sites on P-planes	Pt-sites on H-planes	Pt-sites on U-plane	Lowest S value	Highest S value	Total Pt- sites	Total Co- sites	Average S value
(202)	53	53		72	66		0.88	1.00	7,314	3,498	0.94 $\pm$ 0.03
(204)	35	35		30	47		0.85	1.00	2,695	1,645	0.91 $\pm$ 0.05
(206)	38	38		40	33		0.92	0.96	2,774	1,254	0.95 $\pm$ 0.03
(406)	40	40		14	12		0.89	1.00	1,040	480	0.98 $\pm$ 0.03
(224)	15	15		80	66		0.92	1.00	2,190	990	0.96 $\pm$ 0.05
($\bar{2}\bar{2}4$)	15	15		80	66		0.92	1.00	2,190	990	0.96 $\pm$ 0.05
113			38			30	0.87	1.00	1,140	380	0.96 $\pm$ 0.05
($\bar{1}\bar{1}3$)			38			30	0.87	1.00	1,140	380	0.98 $\pm$ 0.05
(115)			56			22	0.82	1.00	1,232	410	0.97 $\pm$ 0.05
($\bar{1}\bar{1}5$)			56			22	0.82	1.00	1,232	410	0.98 $\pm$ 0.05
Total									22,947	10,442	0.95 $\pm$ 0.05

Figure 40: The values of S , determined from the (220) planes for Tsong's specimen, as a function of depth η in the crystal.

Figure 41: The values of S , determined from the (220) planes for a Cornell specimen, as a function of depth η in the crystal.

were well resolved, and a large number of atoms were examined in a given plane.

The examination of 5000 and 3500 Co-sites on the (220) planes in the Tsong and Cornell specimens, respectively, yielded an S value of 0.93 ± 0.03 for both specimens. For a specimen from the same batch and with the same heat treatment as Tsong's specimen, Berg et al.⁽⁹¹⁾ obtained an average S value of 0.93 ± 0.15 from the examination of 3500 Co-sites in the (220) planes. This showed that the FIM procedure for measuring S is an accurate and reproducible one. In a partially ordered specimen, i.e., $S = 0.68$, Berg et al. found that the values of S oscillated, when S was measured as a function of depth in the crystal, with a typical wavelength of about 80-160 Å. The detailed S values versus depth, which were measured along the normal to (202) planes in both specimens, shown in Figs. 40 and 41 indicated no periodic oscillations in S ; the oscillations in S were small and random. Therefore, because the {220} planes had: (1) a good atomic resolution; (2) a large number of atoms; (3) no periodic oscillations in the S versus depth measurements; and (4) small oscillations in the S values, they were chosen for the measurements of the changes in the S values in the irradiation experiments performed to measure the radiation damage profiles.

IV.2 Radiation Damage Profiles

IV.2.1 Description of the Experiment

The specimens prepared at Cornell were used to measure the radiation damage profiles. After achieving an atomically regular FIM image, each specimen was carefully pulse field-evaporated, and the FIM images were photographed between each pulse. The dissection of the specimen was

continued until about $100 \text{ \AA}$ of material along the normal to a $\{220\}$ plane was removed. The data from these photographs was used to determine the average value S_0 for those $\{220\}$ planes. Next each specimen was bombarded in situ at 30 K, at a constant pressure of $1.2 \cdot 10^{-3}$ Torr neon, with a specially constructed ion gun. Each specimen was bombarded with low-energy (500-2500 eV) Ne^+ ions to a constant Φ_d of $3 \cdot 10^{15} \text{ ion cm}^{-2}$ at a constant ϕ of $5 \cdot 10^{12} \text{ ion cm}^{-2} \text{ sec}^{-1}$; in the case of the 500 eV Ne^+ ion bombardments the ϕ value was $2.5 \cdot 10^{12} \text{ ion cm}^{-2} \text{ sec}^{-1}$, and the bombardment time was 20 minutes. At these values of ϕ and background pressure, i.e., $6 \cdot 10^{-10}$ Torr, and temperature (30 K), the time required to form an adsorbed monolayer was greater than the irradiation period.

After an irradiation each specimen was carefully pulse field-evaporated, and the FIM image was photographed between each pulse. The pulse height and duration were adjusted so that on the average one $\{202\}$ plane was field evaporated per 50 pulses. The controlled dissection of the specimen was continued until the damaged (disordered) region had been removed from the specimen. A total of 1200, 2000, 2500, and 3000 frames of film were shot for each of the 500, 1000, 2000, and 2500 eV bombardments, respectively. The recorded and developed ciné film was analyzed with the aid of the Vanguard motion analyzer. The number of misplaced atoms and the number of λ -contrast effects on each plane was determined, using all the micrographs in an evaporation sequence of each plane and the values of S_d for every two consecutive $\{220\}$ planes were calculated.

IV.2.2 Procedure for Analyzing Radiation Damage Profiles

The ion beam was perpendicular to the axis of the FIM specimen. As a result of an irradiation the bombarded side, i.e., the side exposed to

the ion beam, became disordered. Figure 42 is a schematic representation of an FIM specimen after an irradiation (initial surface) and after the removal of $n(h_1 k_1 l_1)$ atomic planes to expose the $(h_2 k_2 l_2)$ ordered plane contained in the final surface. The interplanar distance d_{hkl} for the ordered alloy Pt_3Co was calculated employing the expression

$$d_{hkl} = \frac{a}{Q(h^2 + k^2 + l^2)^{1/2}} \quad (40a)$$

where

$$\begin{aligned} Q &= 1 \quad \text{if } h, k \text{ and } l \text{ are all odd, and} \\ Q &= 2 \quad \text{if } h, k \text{ and } l \text{ are of mixed parity} \end{aligned} \quad (40b)$$

An FIM specimen is not an ideal hemispherical cap, but has a complicated end form which is a result of the field-evaporation process.⁽⁹⁰⁾ The measurement of the local tip radii (r_i) along the main zone axes is known to give a good description of the shape of the end form.⁽⁸⁹⁾ Such measurements were performed on each of the specimens before an irradiation. The measured values of r_i showed that the shape of the end form could be best approximated by a hemisphere. A typical example of an FIM micrograph recorded during the pulse field-evaporation dissection of a specimen before an irradiation is shown in Fig. 36. Figure 37 exhibits a crystallographic map of the image shown in Fig. 36, with the local tip radii along the $[0\bar{1}1]$, $[110]$, $[10\bar{1}]$, $[100]$, $[10\bar{1}]$, $[110]$, $[0\bar{1}1]$, and $[0\bar{1}0]$ zone axes indicated. The values of r_i in Fig. 37 showed that the shape of the end form could be best approximated by a hemisphere with an average radius $\bar{R}$ where

Figure 42: Schematic representation of an FIM specimen after an irradiation (initial surface) and after removing $n(h_1 k_1 \ell_1)$ atomic planes to obtain a final surface which contained the $(h_2 k_2 \ell_2)$ atomic plane.

$$\bar{R} = \frac{1}{m} \sum_{i=1}^m r_i . \quad (41)$$

In the present example (Fig. 37) $\bar{R} = 331 \pm 26 \text{ \AA}$. We also checked to see if the shape of the end form was changed as a result of the field-evaporation process. To perform this check we recorded the number of atomic planes removed from each plane during the controlled pulse field-evaporation process. These numbers and the knowledge of the crystallographic angles between the planes showed that the amount of material that was removed from each of the planes was the same, and showed that the shape of the end form had not changed. Thus the shape of the end form was taken to be a hemisphere.

The general expression for the depth (x) derived in Chapter III was

$$x = [R_1^2 - (R_2 \cos \beta - n d_{h_1 k_1 l_1})^2]^{1/2} - R_1 \sin \beta , \quad (42)$$

where R_1 and R_2 were the average tip radii before the irradiation and after the removal of $n(h_1 k_1 l_1)$ central planes, respectively. The local tip radii were remeasured after the irradiated metal had been removed; usually the removal of about $80 \text{ \AA}$ of metal was sufficient to remove all the damaged region. The values of r_i and hence $\bar{R}$ were found to be the same before and after the removal of the damaged region. Therefore the value of R_2 used in eqn. (42) was the same as R_1 . Under these conditions eqn. (42) became

$$x = [R_1^2 - (R_1 \cos \beta - n d_{h_1 k_1 l_1})^2]^{1/2} - R_1 \sin \beta . \quad (43)$$

In order to determine the radiation damage profile the long-range order-parameter (S_d^i) of every ($h_2k_2l_2$) plane dissected during the controlled pulse field-evaporation of the irradiated specimen, was measured. The value of S was also measured in that ($h_2k_2l_2$) plane before the irradiation and the average S_o value for many of these ($h_2k_2l_2$) atomic planes was calculated. The values of S_d^i and x_i were calculated for each of the dissected ($h_2k_2l_2$) atomic planes and the radiation damage profile was constructed by plotting $\Delta S_d^i = (S_o - S_d^i)$ versus x_i .

To illustrate how a radiation damage profile was constructed we refer the reader to Fig. 43. As an example a triangular-shaped radiation damage profile was chosen and is illustrated in detail in Fig. 43(a). The quantities $x_o \dots x_4$ are the depths of the center of the ($h_2k_2l_2$) atomic planes from the surface of the initial surface parallel to the beam direction. The plot of the quantity ($S_o - S_d^i$) as a function of x_i illustrated in Fig. 43(b), represents the measured radiation damage profile. The depth resolution of this profile was $\pm 2 \text{ \AA}$.

IV.2.3 Experimental Results

IV.2.3.1 Control Experiments

The isochronal recovery experiments, which are described in Chapter V, showed that there was no long-range migration of SIAs between 6 and 30 K. In order to minimize the adsorption of residual gases during the irradiation period, and also to prevent any changes in the radiation damage profiles as a result of the migration of SIAs, the temperature of the experiment was chosen to be 30 K. Control experiments were performed in order to determine the effect of the adsorption of residual gases on the surface of the specimen during the irradiation period. The experiments

were performed for a period of ten minutes, which was the time required to achieve a Φ_d of $3 \cdot 10^{15}$ ion cm^{-2} , under the same temperature and vacuum conditions as the irradiation experiments, i.e., 30 K and $1.2 \cdot 10^{-3}$ Torr neon. The ion gun was turned on but the potentials on the Einzel lens and the plasma chamber were maintained at zero voltage. Some adsorption was observed; although, pulse field evaporation of less than one monolayer of atoms was required to detect the perfectly ordered crystal. Therefore, it was concluded that the surface adsorption of residual gases during the irradiation periods did not affect our measurements.

Exploratory runs, in which a specimen was bombarded to different Φ_d values, were performed in order to optimize the detection of the radiation damage profiles. When the specimen was bombarded with a dose of less than $5 \cdot 10^{14}$ ion cm^{-2} the radiation damage was not extensive enough, and therefore ΔS_d^i was not distinct enough to obtain the radiation damage profile. When the dose was increased above $4 \cdot 10^{15}$ ion cm^{-2} the radiation damage was so extensive that the S_d^i values could not be measured. The smallest S value we could measure was about 0.7; for S less than this value, the positions of the atoms in the planes could not be determined and therefore S_d^i could not be measured. Experiments performed with Φ_d values between $5 \cdot 10^{14}$ and $4 \cdot 10^{15}$ ion cm^{-2} showed that at a dose of $3 \cdot 10^{15}$ ion cm^{-2} the extent of the radiation damage was optimal for measuring the radiation damage profiles.

IV.2.3.2 Radiation Damage Profiles Measurements

The ion beam, in each of the bombardment experiments, was perpendicular to the axis of the FIM specimen. The crystallographic orientation of each specimen was different and therefore the ion beam direction relative

to the crystallographic directions of each specimen was different. The incident ion beam directions were parallel to the $[1\ 1\ 0]$, $[\bar{2}\ \bar{4}\ 1]$, $[2\ 1\ \bar{5}]$, $[\bar{4}\ 4\ 1]$, $[4\ \bar{9}\ 1]$, $[7\ 6\ \bar{2}\bar{5}]$ or the $[1\ \bar{10}\ 43]$ crystallographic directions. For each of the above crystallographic directions the irradiations yielded a radiation damage profile for at least one, and usually more than one, of the 500, 1000, 2000, and 2500 eV projectiles energies. Thus for each of the above energies at least three radiation damage profiles were measured. The S values for determining the radiation damage profiles, in all the specimens, were measured on the $\{220\}$ planes. As an example the detailed experimental data for one experiment is given in Table XI. This specimen was irradiated with 500 eV Ne^+ ions parallel to the $[2\ 1\ \bar{5}]$ crystallographic direction. The average number of Pt-sites in each of the H- and P-planes were 85 and 70, respectively, and the S_0 value was 0.93 ± 0.02 . The number of the misplaced atoms and the x_i values of each plane are also given in Table XI. The values of S_d^i were calculated for each two consecutive H- and P-planes, and were tabulated with the values of ΔS_d^i . The values of ΔS_d^i were then plotted as a function of x_i to obtain the radiation damage profile which is shown in Fig. 44; the indicated curve was fitted by eye to the data points.

The mean of the radiation damage profile was calculated employing the expression

$$\bar{x}_d = \sum_{i=1}^n x_i P_i, \quad (44)$$

where the probability of occurrence (P_i) was

Table XI: Experimental data for a specimen irradiated with 500.eV Ne⁺ ions parallel to the [215] crystallographic direction.

Plane type	x_{10} in Å	Number of misplaced Pt atoms	Number of λ -contrast effects	S_d^{i*}	ΔS_d^i
P	1.3	12	2	0.81	0.12
H	2.6		3		
P	4.2	13	3	0.79	0.14
H	5.8		3		
P	6.9	11	4	0.82	0.11
H	7.0		3		
P	8.2	10	2	0.84	0.09
H	9.4		3		
P	10.7	10	1	0.84	0.09
H	12.0		3		
P	13.2	8	2	0.87	0.06
H	14.4		2		
P	16.5	7	3	0.86	0.05
H	18.7		0		
P	20.0	6	1	0.90	0.03
H	21.2		1		
P	24.0	5	3	0.92	0.01
H	26.7		0		
P	28.6	4	1	0.93	0.00
H	30.5		0		
Quantity Value		$(\bar{x}_d)$ in Å 9.94±1.75	(Δx_d) in Å 6.31±1.24	(Sk) (Ku) 0.70 2.77	$(x_{0.95})$ in Å 21.5±1.0

* The values of S_d^i were calculated for two consecutive H- and P-planes at a time.

Figure 44: Experimental radiation damage profile for 500 eV Ne^+ ions incident parallel to the $[2\bar{1}5]$ crystallographic direction of the ordered Pt_3Co alloy.

$$P_i = \frac{(S_o - S_d^i)}{\sum_{i=1}^n (S_o - S_d^i)} = \frac{\Delta S_d^i}{\sum_{i=1}^n \Delta S_d^i} \quad (45)$$

The values of ΔS_d^i were taken from the curve of the radiation damage profile in intervals of $0.5 \text{ \AA}^0$; thus determining the value of n in the summations. The k^{th} moment of each radiation damage profile about the mean (μ_k) was calculated employing the expression^(92,114)

$$\mu_k = \sum_{i=1}^n (x_i - \bar{x}_d)^k P_i \quad (46)$$

The square root of μ_2 is commonly called the straggling (Δx_d); it is a measure of the breath of the radiation damage profile parallel to the direction of the incident beam. The third and fourth moments of a radiation damage profile about the mean describe the shape of a profile.⁽¹¹⁴⁾ The skewness (Sk) which is a parameter describing the departure of the distribution from a Gaussian symmetry was calculated by employing the expression

$$Sk = \frac{\mu_3}{(\Delta x_d)^3} \quad (47)$$

The kurtosis (Ku) which is a measure of the peakedness of the profile was

* Many interval values were examined before the $0.5 \text{ \AA}^0$ was chosen as the interval value. We started with an interval value of $2.5 \text{ \AA}^0$. For each interval value we calculated the $\bar{x}_d$ value; as the interval values decreased the percentage difference between the $\bar{x}_d$ values decreased. The $0.5 \text{ \AA}^0$ interval was chosen as the difference between the $\bar{x}_d$ value for this interval and the $0.3 \text{ \AA}^0$ interval was smaller than 0.2%.

calculated by employing the expression

$$Ku = \frac{v_4}{(\Delta x_d)^4} \quad (48)$$

These values were calculated for the radiation damage profile shown in Fig. 44 and are summarized in Table XI. The experimental standard deviation in the mean (S_m) was calculated using the expression⁽⁹²⁾

$$S_m \approx \frac{\Delta x_d}{m^{1/2}} \quad (49)$$

where m is the number of data points in the radiation damage profile. The experimental standard deviation in the straggling (S_p) was calculated using the expression⁽⁹²⁾

$$S_p \approx \frac{\Delta x_d}{(2m)^{1/2}} \quad (50)$$

The approximate symbol ($\approx$) was used to indicate that these values, which were derived⁽⁹²⁾ for a Gaussian distribution, did not represent the exact values of the standard deviations, as the experimental profiles were skewed. These last two standard deviation values were indicated as $\pm$ values in Table XI. Another quantity that is indicated in Fig. 44 and is presented in Table XI is the $x_{0.95}$ value; this is the depth to which 95% of the total amount of damage extended.

Using the same procedures as in the example above, the radiation damage profiles were constructed for each of the irradiations and the parameters defining these radiation damage profiles are summarized in Table

XII. Also, a composite radiation damage profile for each of the bombardment energies was constructed, using all the experimental data except the data for the irradiation which was parallel to the [110] direction which is shown in Fig. 45. For the [110] direction it was found (see Table XII) that the $\bar{x}_d$, Δx_d and $x_{0.95}$ values were greater than for the bombardments parallel to the high index crystallographic direction. This result was taken to be indicative of channeling in the [110] direction. The composite radiation damage profiles for the bombardments parallel to the high index crystallographic directions are shown in Figs. 46 to 49 for each of the bombardment energies 500, 1000, 2000 and 2500 eV. The parameters defining these radiation damage profiles are summarized in Table XIII.

Figure 50 shows the experimental values of $\bar{x}_d$ versus E which were derived from the composite radiation damage profiles; the error bars on the experimental data indicate the S_m values. The experimental $\bar{x}_d$ values versus E curve in this figure were fitted, by the method of least squares, to a power-law expression of the type

$$A = fE^c. \quad (51)$$

The expression obtained for $\bar{x}_d$ as a function of E was

$$\bar{x}_d = (0.15 \pm 0.02) E^{0.67 \pm 0.02} \frac{^\circ}{A}, \quad (52)$$

where the incident ion energy E is in eV. The effective stopping power $(dE/d\bar{x}_d)_{\text{eff}}$ derived from the above expression was

$$(dE/d\bar{x}_d)_{\text{eff}} = (10.2 \pm 1.3) E^{0.33 \pm 0.02} \text{ eV } \frac{^\circ}{A^{-1}}. \quad (53)$$

Table XII: The distribution parameters describing each of the experimental radiation damage profiles.

He ⁺ ion energy (eV)	Irradiation direction	S_0	Number of E-planes (m)	Mean ($\bar{x}_d$) (Å)	Straggling (Δx_d) (Å)	Skewness (Sk)	Kurtosis (Ku)	($x_{0.95}$) (Å)
500	[1 $\bar{1}0$ 43]	0.97 ± 0.03	8	8.9 ± 2.6	5.5 ± 1.5	0.70	2.7	19.5 ± 1.0
500	[2 1 $\bar{3}$]	0.94 ± 0.02	9	9.4 ± 1.7	5.6 ± 1.2	0.60	2.6	19.5 ± 1.0
500	[4 $\bar{3}$ 1]	0.95 ± 0.02	13	9.0 ± 1.6	5.6 ± 1.1	0.72	2.8	19.5 ± 1.0
500	[1 $\bar{1}0$ 43]	0.97 ± 0.03	10	9.4 ± 2.0	5.7 ± 1.4	0.63	2.7	19.5 ± 1.0
500	[2 1 $\bar{3}$]	0.94 ± 0.02	10	9.9 ± 1.7	6.3 ± 1.2	0.70	2.8	21.5 ± 1.0
1000	[2 1 $\bar{3}$]	0.94 ± 0.02	11	16.4 ± 3.0	9.1 ± 2.1	0.36	2.4	33.5 ± 1.0
1000	[7 6 $\bar{2}5$]	0.92 ± 0.02	17	14.8 ± 2.0	8.0 ± 1.4	0.56	2.7	29.5 ± 1.0
1000	[$\bar{5}$ 4 1]	0.92 ± 0.02	17	14.7 ± 2.0	7.9 ± 1.4	0.46	2.6	28.5 ± 1.0
1000	[2 1 $\bar{3}$]	0.94 ± 0.02	16	14.9 ± 2.0	8.0 ± 1.4	0.51	2.6	29.5 ± 1.0
2000	[2 1 $\bar{3}$]	0.94 ± 0.02	16	22.8 ± 3.9	13.7 ± 2.8	0.66	2.7	48.5 ± 1.0
2000	[$\bar{2}$ $\bar{5}$ 1]	0.94 ± 0.02	22	21.3 ± 2.6	12.3 ± 1.9	0.63	2.8	44.5 ± 1.0
2000	[2 1 $\bar{3}$]	0.94 ± 0.02	25	21.5 ± 2.9	13.5 ± 2.0	0.71	2.8	47.5 ± 1.0
2500	[2 1 $\bar{3}$]	0.94 ± 0.02	21	29.0 ± 4.2	18.4 ± 3.0	0.98	2.5	63.5 ± 1.0
2500	[1 $\bar{1}0$ 43]	0.97 ± 0.02	30	26.4 ± 3.3	17.1 ± 2.3	0.67	2.7	58.5 ± 1.0
2500	[7 6 $\bar{2}5$]	0.92 ± 0.02	32	28.1 ± 3.1	18.2 ± 2.2	0.66	2.7	59.5 ± 1.0
1000	[1 1 0]	0.94 ± 0.03	19	37.0 ± 4.4	19.2 ± 3.1	0.25	2.3	73.5 ± 1.0

Figure 45: Experimental radiation damage profile for 1000 eV Ne^+ ions incident parallel to the $[110]$ direction of the ordered alloy Pt_3Co .

Figure 46: A composite radiation damage profile for 500 eV Ne^+ ions incident parallel to the $[1 \ 10 \ 43]$, $[2 \ 1 \ 5]$, and the $[4 \ 9 \ 1]$ crystallographic direction of the ordered Pt_3Co alloy.

Figure 47: A composite radiation damage profile for 1000 eV Ne^+ ions incident parallel to the $[4\ 4\ 1]$, $[7\ 6\ \bar{2}5]$, and the $[2\ 1\ \bar{5}]$ crystallographic direction of the ordered Pt_3Co alloy.

Figure 48: A composite radiation damage profile for 2000 eV Ne^+ ions incident parallel to the $[2\ 1\ \bar{5}]$ and $[2\ \bar{4}\ 1]$ crystallographic direction of the ordered Pt_3Co alloy.

Figure 49: A composite radiation damage profile for 2500 eV Ne^+ ions incident parallel to the $[2 \ 1 \ \bar{5}]$, $[1 \ \bar{10} \ 43]$, and the $[7 \ 6 \ \bar{25}]$ crystallographic direction of the ordered Pt_3Co alloy.

Table XIII: The distribution parameters obtained at each bombardment energy for the composite radiation damage profiles.

Ne ⁺ ion energy (eV)	Mean ($\bar{x}_d$) (Å)	Straggling (Δx_d) (Å)	Skewness (Sk)	Kurtosis (Ku)	($x_{0.95}$) (Å)
500	9.3 ± 0.8	5.7 ± 0.6	0.67 ± 0.05	2.7 ± 0.1	19.5 ± 1.0
1000	14.7 ± 1.0	8.2 ± 0.7	0.53 ± 0.09	2.7 ± 0.3	30.5 ± 1.0
2000	22.6 ± 1.7	13.2 ± 1.2	0.67 ± 0.04	2.8 ± 0.2	48.5 ± 1.0
2500	27.8 ± 2.5	17.9 ± 1.8	0.66 ± 0.05	2.7 ± 0.2	61.0 ± 1.0

Figure 50: Mean depth of radiation damage ($\bar{x}_d$) versus incident Ne^+ ions energy E (Also shown is $\bar{p}_d$ versus ϵ) for Pt_3Co alloy.

Figure 51 shows the experimental values of Δx_d which were derived from the composite radiation damage profiles. The Δx_d versus E curve, which was fitted by the method of least squares to a power-law expression of the type of eqn. (51) was

$$\Delta x_d = (0.08 \pm 0.02) E^{0.69 \pm 0.03} \text{ \AA} . \quad (54)$$

Figure 52 shows the experimental values of $x_{0.95}$ for each of the irradiations except the one parallel to the [110] direction. The expression for $x_{0.95}$ as a function of E was obtained by fitting, employing the method of least squares, the data to a power-law expression of the type of eqn. (51) which gave

$$x_{0.95} = (0.29 \pm 0.05) E^{0.67 \pm 0.02} \text{ \AA} . \quad (55)$$

In the irradiated specimens the distinction between replacement and displacement event was more difficult than in the unirradiated specimens. The difficulties arose from the distortion of the Pt rows in the disordered (220) planes, thus causing difficulties in the exact mapping of the atoms in each plane. Table XIV summarizes the results obtained for some of the irradiated specimens. The quantity of λ -contrast effects represents the sum of the number of vacant lattice sites and the number of Co atoms on the Pt sublattice. The quantity n_d is the number of Pt atoms found in interstitial positions (displacements), and the quantity n_r is the number of Pt atoms found on Co-sites (replacements); the numbers in the parenthesis are the uncertainties in the measurements. The ratio of the number of replacements to the number of displacement events (n_r/n_d) is given in the last column.

Figure 51: Straggling (Δx_d) versus incident Ne⁺ ions energy E for Pt₃Co alloy.

Figure 52: The x_{0.95} values for the different irradiation directions versus Ne⁺ ions energy E for Pt₃Co alloy.

Table XIV: The experimental values of (n_r/n_d) measured on (220) planes of the irradiated specimens.

Ne ⁺ ion energy (eV)	Irradiation direction	Number of λ -contrast effects	n_d	n_r	(n_r/n_d)
500	$[1 \bar{1}0 \ 43]$	80	23	52(11)	2.26
500	$[1 \bar{1}0 \ 43]$	67	22	42(14)	1.92
500	$[21\bar{5}]$	55	19	35(14)	1.84
1000	$[7 \ 6 \ \bar{2}5]$	142	50	74(28)	1.48
1000	$[21\bar{5}]$	129	35	50(15)	1.43
1000	$[21\bar{5}]$	149	36	56(20)	1.56
2000	$[21\bar{5}]$	168	58	96(20)	1.66
2000	$[\bar{2}\bar{4}1]$	185	56	112(10)	2.00
2000	$[21\bar{5}]$	192	45	93(20)	2.07

IV.2.4 Discussion

The irradiation of single crystals may lead to radiation damage profiles that are affected by the lattice structure of the crystals, i.e., channeling and focused RCSs. Visual inspection of a ball model of the ordered Pt_3Co crystal showed that the atoms along the irradiation directions, except the $[110]$ direction, were situated in a random-like array. The $[110]$ direction is the most transparent direction in the Pt_3Co crystal, therefore ions can penetrate deeper in this direction than in the less transparent directions. Figure 53 shows the position of the atoms along the experimental bombardment direction except the $[110]$ which is shown in Fig. 54. Figure 55 shows the stereographic projection of a fcc structure with the critical half angles for channeling (ψ_c) around the major low index poles for 1000 eV Ne^+ ions incident on the Pt_3Co crystal; the values of ψ_c were calculated employing eqn. (26). The values of ψ_c for 1000 eV Ne^+ ions, for the experimental directions, increased linearly with decreasing hkl values, from 0.35 degrees for the $[1\ 10\ 43]$ direction to 10.5 degrees for the $[241]$ direction. This figure demonstrated that none of the experimental bombardment directions, except the $[110]$ direction, were close to a channeling direction. In addition, a comparison between $\bar{x}_d$ values as a function of ψ_c for 1000 eV Ne^+ ions penetrating along different crystallographic direction indicated (see Fig. 56) that the $\bar{x}_d$ value for the irradiation parallel to the $[110]$ direction was more than double the $\bar{x}_d$ values for the irradiations parallel to the $[2\ 1\ \bar{5}]$, $[7\ 6\ \bar{25}]$ or $[\bar{4}\ 4\ 1]$ directions. This demonstrated that when ions did channel, as in the case of $[110]$ direction, the present technique was capable of detecting this effect. Furthermore, the experimental $x_{0.95}$ values for the different

Figure 53: Photographs of a ball model of an ordered Pt_3Co crystal taken along different bombardment directions.

Figure 54: Photograph of a ball model of an ordered Pt₃Co crystal taken along the [110] direction.

Figure 55: Stereographic projection of a fcc structure showing the critical angles for channeling (ψ_c) around the major low index poles for 1000 eV Ne^+ ions incident on Pt_3Co alloy.

Figure 56: Experimental mean depth of damage $\bar{x}_d$, for 1000 eV Ne^+ ions incident parallel to the $[21\bar{5}]$, $[7\bar{6}2\bar{5}]$, $[\bar{4}41]$ and $[110]$ directions in the Pt_3Co alloy. Also shown is $\bar{x}_d$ versus the critical half angle for channeling (ψ_c).

bombardment directions, except the [110] direction, for each of the bombardment energies, were comparable, (within the experimental accuracy) to each other (see Fig. 52). The similarity in the $x_{0.95}$ values, which were more sensitive to channeling than the $\bar{x}_d$ values, indicated that the majority of the ions did not channel in the experimental bombardment directions, except the [110] direction, in spite of the different ϕ_c values. Therefore we conclude that the experimental results for all, except the [110], experimental bombardment directions, could be compared with the theoretical prediction⁽²⁵⁻²⁶⁾ of radiation damage profiles which were derived for amorphous targets. It was found that for all the crystallographic directions employed, with the exception of the [110] direction, the radiation damage profiles at a given E were essentially identical, therefore a composite radiation damage profile for each E employed was constructed and its results were compared with the theoretical predictions.

The n_r/n_d values reported in the literature are from experiments performed with various alloy systems, measurement techniques, irradiation species and energies, all different from ours. These experiments gave indirect results for the replacement events, and have been interpreted as indicating n_r/n_d values of 2.2 ± 2.2 ,⁽⁵³⁾ 10,⁽⁴⁹⁾ 50,⁽¹¹⁵⁾ and 132.⁽⁵⁴⁾ Moreover computer modeling of low-energy radiation damage^(52,116) showed that this ratio depended very strongly on the energy and direction of the incident projectiles. The n_r/n_d values shown in Table XIV did not show a dependence on the direction of the incident projectiles which were parallel to different crystallographic directions, and did not show an energy dependence. In addition, the experimental bombardment directions were far from any close-packed crystallographic direction for which the

theory⁽¹⁶⁾ predicted short RCSs and a small n_r/n_d value. Therefore the experimental results are a direct evidence that replacement events did take place, although the 1.8 ± 0.3 n_r/n_d value obtained is not conclusive.

In order to compare the experimental results to the theoretical predictions, we used Winterbon's⁽²⁶⁾ tabulations and the computer code called TRIM,⁽²⁵⁾ to calculate the first four moments of the elastically deposited energy distribution. Winterbon's method for estimating results for polyatomic targets from monoatomic target calculations, assumes a fictitious monoatomic target with Z and M equal to the average values for the real target materials. In the case of Pt_3Co $M = 161.05$ and $Z = 65.25$. In the TRIM program, the projectile ion collided at random, in a ratio of 1 to 3, with the Co and Pt atoms, respectively. Each distribution was based on 2000 projectile histories, and a projectile history was terminated when its energy fell below 1 eV. The four first moments of the elastically deposited energy profiles obtained from the Winterbon and the TRIM calculations are summarized in Tables XV and XVI, respectively.

A comparison between the experimental expression obtained for $\bar{x}_d$, written in the LSS dimensionless reduced parameters, i.e., $\beta_d = (0.29 \pm 0.04)\epsilon^{0.67 \pm 0.02}$, and the path length-energy relation of the WSS approach, i.e., $p_{pl} = 1.53\epsilon^{2/3}$, showed that both expressions had the same energy dependence ($\epsilon^{2/3}$). The $2/3$ power of ϵ indicates that in the energy régime of interest, i.e., $0.005 < \epsilon < 0.025$, the stopping mechanism was primarily nuclear.⁽¹⁵⁾ The different values of the constant f , i.e., 1.53 and 0.29, showed that the predicted path length was much greater than the experimental β_d . This indicated that in the discussed energy régime the

Table XV: The first four moments of the elastically deposited energy distribution calculated from Winterbon's tabulation.

Ne ⁺ ion energy (eV)	Mean ($\bar{x}_d$) A	Straggling (Δx_d) A	Skewness (Sk)	Kurtosis (Ku)
500	7.2	7.0	.54	4.2
1000	11.3	10.7	.54	4.2
2000	18.2	17.1	.53	4.1
2500	21.4	19.9	.53	4.1

Table XVI: The parameters describing the elastically deposited energy distribution calculated with the TRIM program.

Ne ⁺ ion energy (eV)	Mean ($\bar{x}_d$) in Å	Stragglng (Δx_d) in Å	Skewness (Sk)	Kurtosis (Ku)
500	7.5	5.9	1.3	5.0
1000	11.5	8.9	1.3	4.9
2000	19.0	14.7	1.3	4.5
2500	21.6	17.1	1.3	4.2

deflection angles in the binary collisions were large. This agrees with the theoretical approach which predicted large angle scattering events for $M_1 < M_2$ in this energy régime and thus causing the large difference between the theoretical ρ_{pl} and the experimental $\bar{\rho}_d$ values. This effect can be seen in the trajectories of fifteen 1000 eV Ne^+ ions slowing down in amorphous Pt_3Co which was obtained employing the TRIM program; these trajectories are shown in Fig. 57. The consequence of these large angle deflections was to shorten the mean projected ranges and the mean depth of elastically deposited energy relative to the average total path length of the trajectory ions. These range quantities were obtained as a function of the incident Ne^+ ions energy employing the TRIM program and are shown in Fig. 58. The TRIM program, using the expression suggested by Oen and Robinson⁽²²⁾ for the electronic energy loss (eqn. 30), gave the total electronic loss of the projectiles in the target. Figure 59 shows the fraction of the total energy lost in electronic processes as a function of the incident Ne^+ ion energy. The data in this figure indicated that the energy lost in electronic processes varied from about 7 to 14% of the total energy lost. This confirmed our conclusion that in this energy régime the dominant energy loss mechanism was due to elastic collisions.

The term radiation damage, as stated earlier, was used to include both displacement and replacement events. The experimental observed effect was the change in the S value (ΔS_d) of the specimen before and after an irradiation. This quantity ΔS_d is an absolute measurement of all the radiation damage produced if the energy required to produce a displacement event in the Co sublattice is the same as that for the Pt sublattice. If the Co atoms were more easily displaced from the Co sublattice than the Pt atoms

Figure 57: Trajectories of fifteen 1000 eV Ne^+ ions slowing down in amorphous Pt_3Co as obtained from the TRIM program.

Figure 58: Mean values of the path length ($\bar{p}_{pl}$), the projected range ($\bar{p}_{pr}$), and the elastically deposited energy distribution ($\bar{p}_d$), versus incident Ne^+ ions energy E for Pt_3Co alloy obtained from the TRIM program.

Figure 59: The fraction of energy lost in electronic processes by the Ne⁺ ions penetrating an amorphous Pt₃Co alloy as a function of incident Ne⁺ ion energy obtained from the TRIM program.

were displaced from the Pt sublattice then the ΔS_d underestimated the total amount of radiation damage. On the other hand, if the Pt atoms were more easily displaced from the Pt sublattice than the Co atoms were displaced from the Co sublattice then the ΔS_d value overestimated the total amount of radiation damage. No experimental data on the separate displacement energies of either the Pt or the Co atoms is available. Since the observed radiation damage (disordered) resulted mainly from nuclear collisions, we can compare the experimental radiation damage profiles to the theoretical predictions for the elastically deposited energy distribution.

All the radiation damage profiles exhibited a departure from a Gaussian symmetry. Therefore the first two moments of the profiles were only location parameters, and they did not describe the shape of the profiles. The experimental and the theoretical expression for $\bar{x}_d$ and Δx_d as a function of the incident Ne^+ ions energy were obtained by the method of least squares fit of the data points to the power-law expressions

$$\bar{\rho}_d = p\varepsilon^D, \quad (56)$$

and

$$\Delta\rho_d = q\varepsilon^G, \quad (57)$$

where $\bar{\rho}_d$ and $\Delta\rho_d$ are the mean and the straggling of the profiles in LSS-reduced units and ε is the LSS-reduced energy. For the $\text{Ne-Pt}_3\text{Co}$ system, 1000 eV and 1 Å corresponds to $9.7 \cdot 10^{-3}$ and $9.11 \cdot 10^{-4}$ dimensionless LSS-reduced units, respectively. The fitted constants for $\bar{\rho}_d$ and $\Delta\rho_d$ for the experimental and the theoretical data are presented in Table XVII. The experimental expressions for $\bar{\rho}_d$ and $\Delta\rho_d$ were:

Table XVII: The values of the constants obtained from fitting the data, by the method of least squares, to the expressions $\bar{\beta}_d = p\epsilon^D$ and $\Delta\beta_d = q\epsilon^G$.

Constants	Mean ($\bar{\beta}_d$)			Constants	Straggling ($\Delta\beta_d$)		
	Experimental Winterbon TRIM				Experimental Winterbon TRIM		
p	0.29 ± 0.04	0.24	0.24	q	0.19 ± 0.04	0.20	0.18
D	0.67 ± 0.02	0.68	0.67	G	0.69 ± 0.03	0.65	0.67

$$\bar{\sigma}_d = (0.29 \pm 0.04) \epsilon^{0.67 \pm 0.02} , \quad (58)$$

and

$$\Delta \rho_d = (0.19 \pm 0.04) \epsilon^{0.69 \pm 0.03} , \quad (59)$$

respectively. A comparison between the experimental and the theoretical expressions for $\bar{\rho}_d$ showed a good agreement in the power of the energy dependence, i.e., the exponent of ϵ , whereas the experimental prefactor of ϵ was somewhat greater ($\sim 20\%$) than the theoretical one. In general, the theoretical $\Delta \rho_d$ values were greater ($\sim 10\%$) than the experimental $\Delta \rho_d$ values.

The skewness describes the deviation of the mean from the mode (most probable depth) of a distribution⁽¹¹⁴⁾

$$Sk = \frac{\bar{x}_d - \text{mode}}{(\Delta x_d)^3} . \quad (60)$$

A positive Sk corresponds to the mean being greater than, i.e., to the right of, the mode. Figure 60 shows the Sk as a function of E for the experimental and the theoretical profiles. The average experimental Sk value (~ 0.66) was close to the Sk values of the Winterbon calculations (~ 0.54), however both of these values were smaller than the Sk values predicted by the TRIM program (~ 1.3). The experimental Ku value was approximately 2.7, much smaller than the 4.2 value predicted by both theoretical calculations. For a normal distribution the Ku has a value of 3, for a sharply cut-off distribution the Ku value is less than 3. Since the quantity Ku involved the fourth moment of a profile, its value is very sensitive to the tail of the profile. The discrepancy between the

Figure 60: Experimental and theoretical skewness values versus incident Ne^+ ions energy E for Pt_3Co alloy.

theoretical and the experimental Ku values can be attributed to the fact that we could not determine the shape of each profile tail in detail. The AS_d values in the tails of the radiation damage profiles were of the same order of magnitude as the variations in the S_0 values (see Fig. 41). Thus, the detailed shape of the tails could not be determined and therefore the Ku values could not be determined accurately.

The elastically deposited energy profiles for the WSS approach was obtained employing the first four moments given by Winterbon's tabulation in an Edgeworth expansion series. This expansion series was recommended by Baroody⁽¹¹⁷⁾ and WSS for constructing skewed Gaussian distributions. The elastically deposited energy distribution function ($F(x_i)$) was written as⁽¹¹⁷⁾

$$F(x_i) = \phi_0(z) - (\gamma_1/6)\phi_3(z) + (\gamma_2/24)\phi_4(z) + (\gamma_1^2/72)\phi_6(z) + \dots \quad (61)$$

where $\phi_0 = (2\pi)^{-1/2} \exp(-z^2/2)$,

$$z = (x_i - \bar{x})/\mu^{1/2} ,$$

$$\gamma_1 = \mu_3/\mu_2^{3/2} ,$$

$$\gamma_2 = (\mu_4/\mu_2^2) - 3 ,$$

and ϕ_j was the j^{th} derivative of the normalized Gaussian. The $\bar{x}$ value was obtained from

$$\bar{x} = \int_{-\infty}^{\infty} x_i F(x_i) dx , \quad (62)$$

and

$$\nu_j = \langle (x_i - \bar{x})^j \rangle$$

Employing the first four moments, given by Winterbon's tabulation, in eqn. (61), the elastically deposited energy profiles were constructed. The elastically deposited energy profiles were obtained directly from the TRIM program. The experimental and calculated profiles, for each of the bombarding energies are shown in Figs. 61 to 64. The experimental profile for each of these energies falls between the theoretical profiles.

The effective stopping powers $(d\epsilon/d\bar{p}_d)_{\text{eff}}$, where $\bar{p}_d$ is the dimensionless mean depth of damage and the mean depth of the elastically deposited energy, were derived from the $\bar{p}_d = p\epsilon^D$ expressions for the different cases (Table XVI). Their values were:

$$(1) \text{ TRIM} - (d\epsilon/d\bar{p}_d)_{\text{eff}} = 6.22\epsilon^{0.33}, \quad (63)$$

$$(2) \text{ Winterbon} - (d\epsilon/d\bar{p}_d)_{\text{eff}} = 6.13\epsilon^{0.32}, \quad (64)$$

$$(3) \text{ Experimental} - (d\epsilon/d\bar{p}_d)_{\text{eff}} = (5.15 \pm 0.5)\epsilon^{0.33 \pm 0.02}, \quad (65)$$

These relations and the nuclear stopping powers of the WSS approach (eqn. 8), and the WHB approach (eqn. 10) with $A = 0.56$, $B = 1.18$ and $C = 0.63$, and Kalbitzer et al. (eqn. 11), are shown in Fig. 65. Since the observed radiation damage (i.e. disorder) resulted from nuclear collisions only, we can refer to the effective stopping power derived from the experimental data as an effective nuclear stopping power. Comparison between the effective nuclear stopping power and Kalbitzer et al. experimental nuclear stopping power (eqn. 11), which was derived for the same energy régime,

Figure 61: The radiation damage profiles obtained from (1) the TRIM program, (2) the experimental results and (3) the Edgeworth expansion series with Winterbon's moments, for 500 eV Ne⁺ ions incident on Pt₃Co alloy.

Figure 62: The radiation damage profiles obtained from (1) the TRIM program, (2) the experimental results and (3) the Edgeworth expansion series with Winterbon's moments, for 1000 eV Ne⁺ ions incident on Pt₃Co alloy.

Figure 63: The radiation damage profiles obtained from (1) the TRIM program, (2) the experimental results and (3) the Edgeworth expansion series with Winterbon's moments, for 2000 eV Ne^+ ions incident on Pt_3Co alloy.

Figure 64: The radiation damage profiles obtained from (1) the TRIM program, (2) the experimental results and (3) the Edgeworth expansion series with Winterbon's moments, for 2500 eV Ne⁺ ions incident on Pt₃Co alloy.

Figure 65: Experimental and theoretical nuclear stopping powers versus incident Ne^+ ions energy in LSS-units.

shows a large discrepancy. This large discrepancy is due to the fact that in the present experiment the Ne^+ ions suffered large angle deflections in the collisions, whereas in the case of Kalbitzer et al., where the projectile ions were heavier than the target atoms, the ions did not suffer large angle deflections and therefore their trajectories were approximately straight. Thus, their total path length was very close to their projected range, which is confirmed by the closeness of the Kalbitzer et al. curve to the WHB and the WSS curves, which were derived for the path length. This shows that a universal nuclear stopping power has to be a function of the mass ratio $v = M_2/M_1$ as well as ϵ . This last point was included in the two calculational techniques, i.e., the TRIM program and the WSS approach, which were in a good agreement with our results.

Moreover, a general expression for the effective nuclear stopping power was derived from the experimental data of the Ne^+ ions incident on Ni_4Mo and Pt_3Co alloys. Using the experimental values of $\bar{\beta}_d^I$ for the case of Ne^+ ions incident parallel to the $[\bar{7}6\bar{2}]$ direction of the Ni_4Mo crystal, as the approximate mean depth of the radiation damage distribution, and the present values of $\bar{\beta}_d$, the effective nuclear stopping power was expressed as a function of ϵ and v . The v values for Ne^+ ions incident on Ni_4Mo and Pt_3Co were 3.28 and 7.98, respectively. The effective nuclear stopping power expressions for the Ne^+ ions irradiations of Ni_4Mo and Pt_3Co were

$$(\text{d}\epsilon/\text{d}\bar{\beta}_d^I)_{\text{eff}} = (3.5 \pm 0.4)\epsilon^{0.32 \pm 0.04},$$

and

$$(\text{d}\epsilon/\text{d}\bar{\beta}_d)_{\text{eff}} = (5.15 \pm 0.5)\epsilon^{0.33 \pm 0.02},$$

respectively. These values were fitted to the expression

$$(d\epsilon/d\bar{p}_d)_{\text{eff}} = A v^n \epsilon^{1/3} \quad (66)$$

and the general approximate expression derived from the experimental expressions was

$$(d\epsilon/d\bar{p}_d)_{\text{eff}} = 3/2 v^{2/3} \epsilon^{1/3} \quad (67)$$

This effective nuclear stopping power also approaches the nuclear stopping curve of Kalbitzer et al. for this energy régime.

IV.2.5. Summary

1. We demonstrated that the exact determination of the local value of the Bragg-Williams long-range order-parameter (S) by the FIM technique can be used to measure radiation damage profiles. The values of S can be measured on a routine basis and very accurately when the imaging atoms constituted the majority of the atoms in the alloy.

2. A technique for measuring radiation damage profiles in order-disorder alloys using the FIM has been described. This technique was based on the change (ΔS_d) in the S values in an irradiated specimen relative to the unirradiated specimen. The resulted ΔS_d versus distance (x) from the irradiated surface plot constituted a radiation damage profile.

3. Single crystals of ordered Pt_3Co alloy were irradiated with 500 to 2500 eV Ne^+ ions, which corresponded to the LSS reduced energy (ϵ) values that varied from 0.005 to 0.025. The irradiations were parallel to different crystallographic directions of the ordered Pt_3Co alloy. For the $[110]$ direction, which is the most transparent direction, the radiation

damage profile extended to a greater depth than for the bombardments parallel to the high index crystallographic directions. This result was taken to be indicative of channeling in the [110] direction.

4. Detailed radiation damage profiles were obtained for each of the irradiations. A composite radiation damage profile was constructed for each incident Ne^+ ions energy, for the nonchanneling directions. These profiles were compared to the theoretical predictions of the depth distributions of the elastically deposited energy.

5. The experimental effective stopping power was derived from the mean depth of radiation damage profiles versus energy relation (eqn. 58)

$$\bar{p}_d = (0.29 \pm 0.04) e^{0.67 \pm 0.02},$$

was (eqn. 65)

$$(d\varepsilon/d\bar{x}_d)_{\text{eff}} = (5.15 \pm 0.5) e^{0.33 \pm 0.02}.$$

These relations were in a good agreement with the theoretical predictions of Winterbon's tabulations⁽²⁶⁾ which were based on the WSS⁽¹⁵⁾ approach, and the results of the computer simulation of the transport of energetic ions in an amorphous target called TRIM⁽²⁵⁾, which was based on the WHB approach.⁽⁴⁾

6. The experimental relation for the straggling was (eqn. 59)

$$\Delta p_d = (0.19 \pm 0.04) e^{0.69 \pm 0.03}.$$

The theoretical straggling values, in general, were greater than the experimental straggling values. Also, the average experimental skewness

value (~ 0.66) was close to the average skewness value of Winterbon (~ 0.54); both of these were smaller than the average skewness value predicted by the TRIM program (~ 1.3).

7. Combining the results obtained for the effective stopping powers for Pt_3Co and Ni_4Mo alloys a general expression for the effective stopping power was derived. This approximate relation expresses the effective stopping power as a function of the mass ratio $v = M_2/M_1$ as well as ϵ , that is (equ. 66)

$$(d\epsilon/d\bar{p}_d)_{\text{eff}} = \frac{3}{2} v^{2/3} \epsilon^{1/3} .$$

V. POST IRRADIATION ISOCHRONAL WARMING EXPERIMENTS

V.1. Introduction

Isochronal warming experiments of the type described by Seidman^(32,75) were performed on the ordered Ni_4Mo and Pt_3Co alloys. In this experiment the FIM specimen was irradiated in situ, in UHV, in the absence of an electric field. This procedure removed the question of the role played by the imaging electric field (E_i) during the point-defect production-stage. After the specimen was irradiated at a given temperature (T_i) the specimen was re-imaged and then warmed isochronally at a prescribed rate (δ), and the FIM images of the surface were simultaneously recorded. The surface of an FIM specimen served as an excellent sink for mobile point defects and the arrival of an SIA was detected as an extra bright-spot(s) which appeared on the surface of an FIM specimen.

During the isochronal warming stage the value of E_i was about 4.4 VA^{-1} .⁽¹¹⁸⁾ The effect of E_i was to subject the tip to a negative hydrostatic pressure (p) which was approximately given by the expression $E_i^2/8\pi$.⁽¹¹⁹⁾ This p dilated the lattice and changed the enthalpy of migration (Δh_{li}^m) of an SIA by an amount of $p\Delta v_{li}^m$, where Δv_{li}^m was the volume change of migration of an SIA. The quantity Δh_{li}^m is related to the internal energy change of migration (Δu_{li}^m) through the expression⁽⁶⁶⁾

$$\Delta h_{li}^m = \Delta u_{li}^m - |p|\Delta v_{li}^m = \Delta u_{li}^m - E_i^2 \Delta v_{li}^m / 8\pi \quad . \quad (68)$$

Therefore, the effect of E_i was to reduce the value of Δh_{li}^m by an amount of $E_i^2 \Delta v_{li}^m / 8\pi$, which implied that the diffusivity (D_{li}) of an SIA in the stressed state was enhanced and made the SIA mobile at a temperature where

in the absence of E_i it would be immobile. The effect of E_i on the value of D_{1i} for tungsten⁽¹²⁰⁾ and platinum⁽¹²¹⁾ SIAs has been studied in detail in our laboratory. These studies showed that the upper limit of Av_{1i}^m , in both cases, was of the order of $(2-3) \cdot 10^{-2}$ atomic volume (Ω_a); thus the value of Au_{1i}^m was reduced by less than 0.02 eV. In the present work we did not study the effect of E_i on D_{1i} , but in view of the above results we assumed that the migration processes of the SIAs were affected very little by E_i . Although a downward shift of our data by ~ 7 K as a result of the pAv_{1i}^m effect was possible.⁽³²⁾

It is emphasized that the FIM isochronal warming experiment can only detect the SIAs which undergo long-range migration and cannot detect SIAs which recover as a result of close-pair recombination. In addition, because of the process of image formation of ordered alloys, only one migrating atomic species can be detected, i.e., Pt in Pt_3Co and Mo in Ni_4Mo . But this does not necessarily mean that the recovery spectrum obtained was a result of the migration of only one atomic species. The diffusion process occurred via an interstitial mechanism in which both species may have been involved. Thus, even though only one atomic species can be detected, its arrival at the surface of the FIM specimen may have been a result of the migration of both species.

There are six geometrically possible stable interstitial configurations in the fcc lattice.⁽¹²²⁾ Theoretical calculations⁽¹²³⁾ of the formation energy (U_{1i}^f) of a single SIA in pure Cu(fcc) indicated that the most stable configuration was the [100] split interstitial. There are three configurations in which a single atom is distinguished as the interstitial: the octahedral, crowdion and tetrahedral configurations. In the other three configurations, the split interstitials, the extra

atom is accommodated by sharing a site with another atom. These split configurations are sometimes referred to as [100], [110] and [111] dumbbells.

The situation in ordered alloys is more complicated than in pure metals because each of the different atoms which constitute the alloy can become an interstitial. There are two sublattices, one for each species, in a binary ordered alloy; an atom from either sublattice can be displaced to an interstitial position. This doubles the number of geometrically possible configurations as compared with a pure metal. Thus, there are twelve geometrically possible configurations for the SIA in binary ordered alloys. Of the possible split interstitial configurations, three may be mixed split interstitials, i.e., two atoms with different chemistry share one atomic site. The mixed [100] dumbbell was recently discovered in dilute Al alloys.⁽¹²⁴⁾ Interstitialcy migration of most of the interstitial configurations involves the migration of both atomic species which constitute the alloy. At present⁽¹²⁵⁾ there are no calculations of the relative values of the migration and formation energies of interstitials in ordered alloys. Because interstitialcy migration in ordered alloys has not experimentally been demonstrated we will compare the recovery behavior of the ordered alloys to the recovery behavior of the component elements of the alloys.

V.2 Experimental Details

The Pt_3Co and Ni_4Mo specimens used in these experiments were the same specimens described in the previous chapters. The experiments were performed in the FIM that was attached to the Hill-Nelson sputtered metal ion source.⁽⁶⁶⁾ The irradiations were performed in situ at a pressure of $1 \cdot 10^{-9}$ Torr in the FIM. The total fluence employed was about

$(2-10) \cdot 10^{12}$ ions cm^{-2} of either 30 or 60 keV Kr^+ or Xe^+ ions and the flux of ions at the specimen position was about $3 \cdot 10^{10}$ ions $\text{cm}^{-2} \text{sec}^{-1}$.

Since the main reason for performing these experiments was to determine the temperature at which the radiation damage distribution would not change as a result of the migration of SIAs, we irradiated the specimens at the lowest temperature that could be achieved in the FIM, i.e., $T_i \leq 6$ K. At this temperature the only imaging gas that could be used without extensive condensation was helium. Therefore we used helium as the imaging gas even though at high temperatures (70 K for Pt_3Co and 90 K for Ni_4Mo) the FIM images disappeared. The imaging voltage was maintained at a value which was 10% below the BIV characteristic of T_i to prevent continuous field-evaporation of the specimens at high temperatures. After an irradiation, the temperature of the FIM specimen was raised at a δ of $\approx 2 \text{ K min}^{-1}$ while the surface was photographed continuously at a rate of 40 frames min^{-1} which corresponded to 20 frames K^{-1} . The cine film obtained in the above manner was then examined for the SIA contrast effect, i.e., extra bright spot which we have denoted a defect.

V.3 Experimental Results

V.3.1. Control Experiments

Prior to each irradiation a control experiment was performed. The experimental conditions, i.e., T_i , δ , background pressure and the time the specimen was exposed to the ion source were identical to the experimental conditions for an isochronal warming experiment, except that the ion beam was stopped before it hit the specimen. An average of three artifact SIA contrast effects per control isochronal warming experiment were detected. Since the average number of SIA contrast effects (defects)

detected in the isochronal warming experiments was about 30, the background artifact concentration was about 10% of the total number of defects in the irradiated specimens.

V.3.2. Isochronal Warming Experiments of Pt_3Co Alloy

The presence of a defect in the FIM images was detected by an extra bright-spot contrast effect,⁽⁷⁵⁾ which went from no-contrast to full-contrast within one frame of ciné film. Two examples of the appearance of extra bright-spot contrast effects are shown in Fig. 66. Each pair of micrographs, recorded before and after the appearance of the defect (see arrows), was taken 1.5 sec apart; the latter corresponded to a temperature interval of 0.05 K. The experimental conditions, the number and the concentration of the defects observed in each isochronal warming experiment are summarized in Table XVIII. The atomic concentration of defects (f) listed in the last column of Table XVIII was calculated from the equation⁽¹²⁶⁾

$$f = \frac{N\Omega_a}{V} = \frac{3N\Omega_a}{2\pi R^3(1-\cos\psi)} \quad , \quad (69)$$

where N is the number of defects observed in each isochronal warming experiment and V is the volume element from which the defects originated. The quantity V was calculated for a cone cut out of a sphere of radius R , where the cone's apex coincided with the center of the sphere and its half-angle ψ was 54.7° . The quantity Ω_a was equal to $a_0^3/4$ where a_0 was the lattice parameter of ordered Pt_3Co .

The composite isochronal warming spectrum for the three experiments is shown in Fig. 67; the ordinate of the spectrum is the fraction of the total number of defects per 5 K interval and the abscissa is the tip

Figure 66: Two examples of SIAs arriving at the surface of an FIM specimen during the isochronal recovery experiment. In (A) the extra bright-spot appeared on the (111) plane at 35 K. In (B) the extra bright-spot appeared on the (224) plane at 45 K. Micrographs (1) and (2) were taken 1.5 sec apart, in both A and B, which correspond to a temperature interval of 0.05 K.

Table XVIII: Data for the isochronal recovery experiments of irradiated Pt_3Co .

Specimen Number	Irradiation dose (10^{12} ion cm^{-2})	Xe ⁺ Ion Energy E_0 (keV)	Tip Radius ($\text{\AA}$)	Number of Defects (N)	Concentration of Defects (f) (10^{-6} at. fr.)
1	7	30	300	20	9.8
2	4	60	310	23	11.3
3	9	60	320	29	14.3

Figure 67: A composite isochronal recovery spectrum for Pt_3Co irradiated at 6 K with 30 or 60 keV Xe^+ ions and warmed to 70 K employing a 2 K min^{-1} warming rate.

temperature. An emergence plot, i.e., the points of emergence of the defects on the surface of the specimen is shown in Fig. 68. The five concentric circles shown in this figure divided the area of the hemispherical tip into five equal areas. The angular dependence as a function of θ and ϕ (see inserted drawing in Fig. 69) for the emergence distribution presented in Fig. 68 is shown in Fig. 69. The θ dependence was measured from the [001] direction out to 54.7° and the ϕ dependence was measured from the [011] direction toward the [101] direction. Visually, Figs. 69a and 69b demonstrated that the emergence distribution had no angular dependence.

V.3.3. Isochronal Warming Experiments of Ni_4Mo Alloy

The contrast effect associated with the arrival of an SIA at the surface was identical to that in Pt_3Co , i.e., no-contrast to full-contrast within one frame of ciné film. Two examples of the appearance of extra bright-spot contrast effects are shown in Fig. 70. The experimental conditions, the number and the concentration of the defects observed in each isochronal warming experiment are summarized in Table XIX. The value of f was calculated using eqn. (69) with $\psi = 46.6^\circ$ and Ω_a was equal to $a_o^2 c_o / 10$ where a_o and c_o were the lattice parameters of the ordered bct Ni_4Mo .

The composite isochronal warming spectrum for the experiments is shown in Fig. 71. An emergence plot is shown in Fig. 72, and the angular dependence of the emergence distribution as a function of θ and ϕ is shown in Fig. 73. The observed emergence distribution showed no angular dependence.

Figure 68: An emergence plot representing the positions of the appearance of the extra bright-spots at the surface of an FIM specimen during the isochronal recovery experiments. The five concentric circles in this figure divided the area of the hemispherical tip into five equal area.

Figure 69: The θ and ϕ dependence for the emergence distribution of defects shown in Fig. 68. In (a) the θ angles $23^\circ 50'$, $33^\circ 54'$, $41^\circ 43'$, $48^\circ 33'$ and $54^\circ 42'$ divided the area observed on the specimens' surfaces into five regions of 0.53 sr each. In (b) the observed area was also divided into five equal regions.

Figure 70: Two examples of SIAs arriving at the surface of an FIM specimen during the isochronal recovery experiment. In (A) the extra bright-spot appeared on the (110) plane at 35 K. In (B) the extra bright-spot appeared on the (150) plane at 50 K. Micrographs (1) and (2) were taken 1.5 sec apart, in both A and B, which corresponded to a temperature interval of 0.05 K.

Table XIX: Data for the isochronal recovery experiments of irradiated Ni_3Mo .

Specimen Number	Irradiation dose (10^{12} ion cm^{-2})	Kr^+ Ion Energy E_0 (keV)	Tip Radius ($\text{\AA}$)	Number of Defects (N)	Concentration of Defects (f) (10^{-6} at. fr.)
1	5	30	390	29	3.6
2	10	30	400	33	4.1
3	10	30	410	33	4.1

Figure 71: A composite isochromal annealing spectrum for Ni_4Mo irradiated at 6 K with 30 KeV Kr^+ ions, and warmed to 100 K at rate of 2 K min^{-1} .

Figure 72: An emergence plot showing the positions of the appearance of the extra bright-spots at the surface of an FIM specimen during the isochronal recovery experiments. The five concentric circles in the figure divided the area of the hemispherical tip into five equal areas.

Figure 73: The θ and ϕ dependence for the emergence distribution of defects shown in Fig. 72. In (a) the θ angles $20^\circ 22'$, $28^\circ 58'$, $35^\circ 41'$, $41^\circ 23'$ and $46^\circ 35'$ divided the area observed on the specimens' surfaces into five regions of 0.39 sr each. In (b) the observed area was also divided into five equal regions.

V.4. Discussion

In the analysis of annealing behavior it is important to pay attention to the state of damage in the irradiated specimen. In a pure metal irradiated with 30 or 60 keV Kr^+ or Xe^+ ions, it has been shown⁽⁴⁶⁾ that depleted zones (DZs) were produced at a depth 20 to 100 Å below the irradiated surface. These DZs consisted of a vacancy-rich core (~ 10 at.%) surrounded by a mantle of SIAs (~ 1 at.%), and a number of SIAs which were separated from the DZs by a distance of 50-100 Å produced by the focused RCS mechanism. In irradiated ordered Cu_3Au Jenkins et al.⁽⁴⁸⁾ reported on the creation of zones of disorder within the volumes of the displacement cascades. In summary, the state of damage in an ordered alloy has the characteristic of an irradiated pure metal with the addition of the various disordering effects.

The fact that a binary ordered alloy has two different atoms which accommodate two different sublattices (A atoms on α -sites and B atoms on β -sites) causes the nature of the point defects to be more complex than in pure metals. In an ordered alloy both the α -sites and the β -sites can be vacant, and the A and the B atoms can both be in interstitial positions. The atomic configuration of each of these defects will determine the mobility of these defects. Also, the atomic size of the different species and the ordering energy can effect the mobility of these defects. All of the above characteristics of the point defects in ordered alloys cause the recovery behavior to be more complex than in pure metals. Nevertheless, to obtain a first order estimate of the physical quantities relevant to the recovery behavior of point defects the results were analyzed under the assumption of a single thermally-activated diffusion model developed by

Scanlan et al.⁽⁶⁶⁾ for the case of an FIM specimen. The results are also compared with the annealing results for the component elements of the ordered alloys.

The diffusion model⁽⁶⁶⁾ was based on the following assumptions: (1) the FIM tip was approximated by a sphere of radius R; (2) the initial distribution of SIAs was uniform; (3) the specimen's surface was the dominant sink for SIAs; (4) there were no SIA clusters or SIA-impurity atom clusters formed during the recovery; (5) the diffusion of SIAs occurred via a single thermally-activated process; and (6) the concentration gradient had no angular dependence. The expression⁽⁶⁶⁾ for the flux of SIAs (J_{li}) crossing the surface of the FIM tip as a function of the tip temperature (T) was given by

$$J_{li} = \gamma \exp(-1/y) \sum_{n=1}^{\infty} \exp[-n^2 \Gamma y^2 \exp(-1/y)] \quad , \quad (70a)$$

where

$$y = kT/\Delta h_{li}^m \quad , \quad (70b)$$

$$\gamma = f_r 8\pi R c_{li}^0 D_{li}^0 \quad , \quad (70c)$$

and

$$\Gamma = \pi^2 D_{li}^0 \Delta h_{li}^m / R^2 6k \quad . \quad (70d)$$

The quantity D_{li}^0 is the pre-exponential factor of the SIA diffusion coefficient, c_{li}^0 is the initial concentration of SIAs (cm^{-3}), f_r is the fraction of the total area of the sphere which was observed and k is Boltzmann's constant. This model predicted the Δh_{li}^m value to be:

$$\Delta h_{1i}^m = kT_{\max}/y_{\max} \quad , \quad (71)$$

where $T_{\max}$ was obtained from the observed isochronal spectrum and $y_{\max}$ was given by: (126)

$$y_{\max} = 1.25 \cdot 10^{-3} + 0.937(\ln \Gamma - 2 \ln \ln \Gamma)^{-1} \quad . \quad (72)$$

The results of this analysis give a Δh_{1i}^m value which is probably accurate to $\leq 10\%$, while the value of D_{1i}^0 is most likely uncertain by a factor of ~ 10 . (121)

In order to obtain the best values of Δh_{1i}^m and D_{1i}^0 using this analysis we had to determine which Γ value was the appropriate one to use; this was done employing eqn. (70d). For each case the values of R and δ were known, and were inserted in eqn. (70d). An approximate value of Δh_{1i}^m was obtained employing eqn. (70b) where $y_{\max}$ was calculated employing eqn. (72) with different values of Γ ($10^{10} < \Gamma < 10^{17}$); because of the $\ln \ln \Gamma$ dependence the $y_{\max}$ value did not vary much. The D_{1i}^0 value was obtained from (127)

$$D_{1i}^0 = g a_o^2 v_o \exp(\Delta s_{1i}^m/k) \quad (73)$$

where g is a geometric factor which depends on the configuration of the SIA (~ 0.3), a_o is the lattice constant, v_o is the attempt frequency and Δs_{1i}^m is the entropy change of migration. The attempt frequency (v_o) was estimated employing the expression (128)

$$v_o = \left(\frac{2\Delta h_{1i}^m}{ma_o^2} \right)^{1/2} \quad (74)$$

where m is the mass of the SIA. This ν_0 was defined⁽¹²⁸⁾ as the frequency of the small-amplitude oscillations of an atom in the minima of a potential energy profile.

V.4.1. Annealing Behavior of Pt₃Co Alloy

The isochronal warming experiments were performed between 6 and 70 K. The annealing spectrum shown in Fig. 67 showed a broad peak centered around 37.5 K which extended to ~ 65 K. No long-range migration (extra bright-spot contrast effects) was observed below 29 K. Employing $\Delta h_{1i}^m = 0.1$ eV and $m = 161 \cdot 1.67 \cdot 10^{-24}$ gr we obtained $\nu_0 = 10^{12} \text{ sec}^{-1}$ and $D_{1i}^0 = 10^{-3} \text{ cm}^2 \text{ sec}^{-1}$, with these values eqn. (70d) yielded $\Gamma = 10^{14}$. The exact end of the first thermally activated long-range migration peak could not be determined precisely, therefore in order to calculate Δh_{1i}^m the experimental data was terminated at 45 K. The calculated peak shown in Fig. 74 superimposed best on the experimental spectrum for $\Gamma = 10^{14}$. The fit was made such that the areas under the calculated curve and under the experimental spectrum, which was terminated at 45 K, were equal. The calculated spectrum peaked at $T_m = 38.0$ K and the Δh_{1i}^m value obtained for $\Gamma = 10^{14}$ was 0.084 eV. Table XX shows the calculated Δh_{1i}^m and D_{1i}^0 values obtained for different Γ values. These values indicated that the value of D_{1i}^0 was quite sensitive to the value of Γ whereas the Δh_{1i}^m value was not; a change by a factor of 10 in Γ , i.e., from 10^{14} to 10^{15} , yielded a $\sim 9\%$ increase in the Δh_{1i}^m value, whereas D_{1i}^0 increased by a factor of ~ 10 .

The isochronal annealing spectrum of the alloy Pt₃Co was compared mainly to the annealing spectrum of pure Pt for the following reasons:

- (1) no annealing data for Pt₃Co alloy was available in the literature;
- (2) the majority of the atoms (75%) constituted the Pt₃Co alloy were Pt atoms;
- (3) both the alloy Pt₃Co and the metal platinum have the same

Figure 74: A calculated isochronal annealing spectrum for a single thermally activated process superimposed on the experimental spectrum for Pt_3Co irradiated with Xe^+ ions.

Table XX: The calculated values of Δh_{li}^m and D_{li}^o obtained for different r values.

r	Δh_{li}^m (eV)	D_{li}^o ($\text{cm}^2 \text{sec}^{-1}$)
10^{12}	0.070	$3.2 \cdot 10^{-5}$
10^{13}	0.077	$2.9 \cdot 10^{-4}$
10^{14}	0.084	$2.6 \cdot 10^{-3}$
10^{15}	0.091	$2.4 \cdot 10^{-2}$
10^{16}	0.098	$2.3 \cdot 10^{-1}$

crystal structure, i.e., fcc; and (4) the metal cobalt has an hcp structure. Standard resistivity measurements of the annealing of pure cobalt showed that the long-range migration stage occurred around 55 K.⁽¹²⁹⁾ For pure platinum, standard resistivity measurements determined the activation energy for migration Δu_{li}^m to be 0.063 eV at $T = 26.5$ K.⁽¹³⁰⁾ An FIM isochronal warming experiment on pure Pt, performed by Petroff and Seidman⁽¹²¹⁾, was characterized by $T_m = 15$ K and $\Delta h_{li}^m = 0.042$ eV. The difference in the values of Δu_{li}^m and Δh_{li}^m was attributed⁽¹²¹⁾ to the effect of E_i on the mobility of the SIA and enabled these authors to place a limiting value on the quantity Δv_{li}^m of about $(2-3)10^{-2}\Omega_a$. In comparison with the FIM isochronal warming experiment on pure platinum the annealing spectrum of Pt₃Co alloy was broader and the peak shifted to higher temperatures, and the Δh_{li}^m value was higher. These observations agreed with the general annealing behavior of point defects in other order-disorder alloy systems.^(115, 131)

V.4.2. Annealing Behavior of Ni₄Mo Alloy

The annealing spectrum shown in Fig. 71 showed two broad peaks, one peak around 37.5 K and one around 77.5 K. No long-range migration was observed below 29 K. The appropriate Γ value was calculated to be $\sim 10^{14}$. Thus, the calculated peaks superimposed on the experimental spectrum shown in Fig. 75 were fitted with $\Gamma = 10^{14}$. The fit was made such that the area under the first calculated peak was equal to the area under the experimental spectrum which was terminated at 45 K. The area under the second calculated peak was equal to the area under the experimental spectrum of the second peak. The first calculated spectrum peaked at $T_m = 37$ K with $\Delta h_{li}^m = 0.083$ eV and $D_{li}^0 = 4.14 \cdot 10^{-3} \text{ cm}^2 \text{ sec}^{-1}$, the second

Figure 75: The calculated spectrum for two single activated processes superimposed on the experimental spectrum obtain for Ni_4Mo irradiated with Kr^+ ions.

peaked at $T_m = 75.5$ K with $\Delta h_{11}^m = 0.174$ eV and $D_{11}^0 = 2.0 \cdot 10^{-3} \text{ cm}^2 \text{ sec}^{-1}$.

No isochronal recovery data, for irradiated Ni_4Mo , was found in the literature to compare with the present annealing spectrum. An FIM isochronal warming experiment on pure Mo performed in our laboratory⁽¹³²⁾ showed a major peak centered at 32 K and a minor peak at 45 K which extended to 120 K. Neutron⁽¹³³⁾ and electron⁽⁵³⁾ irradiation of pure Ni showed a long-range migration peak at 57 K with activation energy of about 0.15 eV. A comparison between the alloy and the pure metals data is difficult in this case.

V.5. Summary

Results of the isochronal recovery behavior of ion-irradiated order-disorder Ni_4Mo and Pt_3Co alloys between 6 and ~ 90 K were reported. The FIM specimens were irradiated in situ under ultra-high vacuum conditions at 6 K, with either 30 or 60 keV Kr^+ or Xe^+ ions, to a total fluence of $(2-10) \cdot 10^{12} \text{ ions cm}^{-2}$. The experimental results showed that in both cases there was no long-range migration of SIAs between 6 and 30 K. The isochronal annealing spectrum for Pt_3Co showed a broad peak centered around 37.5 K which extended to ~ 65 K. The experimental results were analyzed on the basis of a single thermally activated diffusion mechanism. The analysis of the experimental spectrum gave an enthalpy change of migration (Δh_{11}^m) of 0.084 eV and a pre-exponential factor of the SIA diffusion coefficient (D_{11}^0) of $\sim 2.6 \cdot 10^{-3} \text{ cm}^2 \text{ sec}^{-1}$. The isochronal annealing spectrum for Ni_4Mo showed two broad peaks, the first one was centered around 37.5 K and the second one around 77.5 K. The analysis of the experimental results on the basis of a single thermally activated diffusion mechanism gave a Δh_{11}^m value of 0.084 eV and D_{11}^0 value of $\sim 4.14 \cdot 10^{-3}$

$\text{cm}^2 \text{sec}^{-1}$ for the first peak, and a Δh_{11}^m value of 0.174 eV and D_{11}^0 value of $\sim 2.0 \cdot 10^{-3} \text{cm}^2 \text{sec}^{-1}$ for the second peak.

The isochronal annealing spectrum of irradiated Pt_3Co alloy was broader and slightly shifted to higher temperatures when compared to the annealing spectrum of electron irradiated pure Pt. These effects agreed with the general annealing behavior of point defects in other order-disorder alloy systems as described in the literature.

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