ANNEALING BEHAVIOR AND SELECTED APPLICATIONS
OF ION-IMPLANTED ALLOYS*

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ABSTRACT

Thermally activated processes cause ion-implanted metals to evolve from the initial state toward thermodynamic equilibrium. The degree of this equilibration is strongly dependent upon temperature, and is considered here for three temperature regimes which are distinguished by the varying mobilities of interstitial and substitutional atoms. In addition, perturbations resulting from the irradiation environment are discussed. Examples are given of the use of implanted and annealed alloys in studies of diffusion, phase diagrams, and solute trapping.

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Introduction

Ion implantation is an alloying tool whose unique properties lead both to increased complexity and to new metallurgical capabilities. The technique initially produces an intimate atomic mixture of controlled composition and depth distribution, essentially unmodified by thermally activated processes and independent of the equilibrium phase diagram. Its use also causes substantial lattice damage, typically > 1 displacement per atom for the doses used in metals work. These two characteristics have been exploited for basic, microscopic studies of metastable and equilibrium alloys, and also to achieve desired macroscopic properties, as detailed in these proceedings.

The present paper reviews the behavior of implanted inter-metallic systems when they are subjected to annealing. The thermally-activated evolution from the frequently metastable initial condition¹ toward thermodynamic equilibrium is considered in three temperature regimes, which are distinguished by the varying mobilities of interstitial and substitutional atoms. It will be shown that implanted and annealed alloys may be used advantageously for basic studies of alloying processes in metals. Examples will be taken from work on diffusion, phase diagrams, and solute trapping. The discussion will be primarily applicable to systems in which all of the constituents are metallic. In such cases defect-related effects are generally less dominant than in semiconductors and insulators.

Annealing Behavior

As the temperature of an implanted metal increases from absolute zero, the first species to achieve appreciable mobility are interstitials, followed by substitutional atoms at higher temperatures. Below the onset of interstitial migration, changes resulting from thermally activated processes are expected to be minimal. The state of the alloy is then determined primarily by nonthermal phenomena, including atomic collisions as the implanted atoms come to rest, spontaneous recovery of the lattice,² and atomic rearrangement during the transient elastic-collision spikes possibly associated with the damage cascades.³ (Elastic-collision spikes are frequently referred to as "thermal spikes". However, for purposes of clarity, a process will be termed "thermal" in the present discussion only if it is associated with a macroscopically defined temperature.) For most metals the temperature range of no interstitial mobility lies well below 100 K,⁴ and few studies providing microstructural information have been carried out for implanted systems in this regime. Nevertheless, the nonthermal processes enumerated above may reasonably be expected to produce certain effects that have been observed in metals implanted at room temperature. These include the frequent retention of crystallinity even for damage levels exceeding one displacement per atom,^{5,6} the preference of the implanted atoms for well-defined lattice sites,⁵⁻⁷ and the possible occurrence of a martensitic transformation to a more stable lattice structure.⁸ However, the formation of discrete, second-phase precipitates is

considered unlikely since this requires longer-range atomic transport.

In the intermediate temperature range where interstitials diffuse but substitutional atoms do not, the occurrence of precipitation in a supersaturated implanted mixture varies with conditions. Moreover, when present, second phases may either be metastable or correspond to thermodynamic equilibrium. Observed precipitation in this regime has thus far been limited to cases where the requisite composition could be achieved locally by the transport of an interstitial solute. For example, room-temperature implantation of oxygen into 20 pure metals produced oxide precipitates in 17 of the cases, with 16 of these yielding the phase expected from the phase diagram.⁹ Similarly, N implantation of Fe resulted in Fe nitrides,¹⁰ while C implantation into Fe followed by annealing at 603 K produced the metastable phase Fe_3C .⁸

When the temperature of an implanted intermetallic system is sufficiently high for all of its constituents to be mobile, and when the irradiation has ceased, phases expected from the equilibrium phase diagram usually form within the implanted layer. This has been observed consistently in the hosts Be, Al, Fe, and Cu.¹¹⁻¹⁷ While interfacial and grain boundary trapping of solute may occur as in any alloy, damage-related effects have not appeared to prevent the precipitation. Furthermore, in Be and Fe hosts, solute concentrations measured in the host phase immediately beneath the precipitated layer have agreed well with corresponding solid solubilities obtained by conventional

metallurgical procedures.^{14,17,18} These results indicate that, under the conditions specified above, a state of near-thermodynamic equilibrium develops within the implanted region. Although such equilibration has not been demonstrated to occur in all cases, exceptions have not thus far been reported. This behavior is central to the use of ion implantation in studying alloys composed of equilibrium phases, examples of which will be given below.

Implantation studies are usually carried out in a host which is effectively semi-infinite. Consequently, complete equilibration implies a uniform and infinitesimal concentration of the implanted elements, a situation of little interest. However, at much shorter anneal times and before substantial diffusion of solute from the implanted layer, a local state of near-thermodynamic equilibrium develops within the surface region, encompassing the implanted layer and a portion of the underlying bulk. This condition arises because the diffusion-limited transport of solute between precipitates within the implanted layer is rapid compared to the loss to the underlying bulk. Thus, the precipitates are typically separated by 10 nm, with diffusion lengths of 10^3 nm being required to reduce the concentration of implanted elements below the threshold for precipitation. Hence, in this example, the characteristic time for local equilibration would be shorter than the time for precipitate dissolution by a factor of $\sim (10^3 \text{ nm} / 10 \text{ nm})^2 = 10^4$. This qualitative conclusion is borne out by detailed mathematical modeling of the evolution of the implanted alloy.¹⁷ Moreover, it has been found to hold even under rather unfavorable conditions, as when the implanted species

evaporates from the nearby surface during annealing.¹⁷ As a result, the time interval between local equilibration and precipitate dissolution is especially useful in studies of alloying, as will be discussed.

The composition-versus-depth profile under the above conditions generally exhibits a peak, corresponding to the precipitated layer, with a tail extending into the single-phase bulk. An example is given in Fig. 1, where the depth profile of Cu implanted into Be at room temperature is shown before and after 94 hours at 673 K.¹⁸ Depth is given in total atoms per unit area, which is the quantity obtained from the ion backscattering analysis, while the indicated 0.1 μm interval corresponds to the atomic density of pure Be. Prior to annealing, the system existed as a supersaturated mixture. The subsequent heat treatment, at a temperature where all the atomic species are mobile, then caused rapid precipitation of Be_3Cu , accompanied by a much slower diffusion of Cu into the bulk. As is generally observed, a discontinuity in slope occurs at the interface between the two- and one-phase regions. The concentration at this point is equal to the solid solubility at the annealing temperature, and the diffused tail has the functional form appropriate for the fixed-concentration boundary condition.

The phases existing in an implanted intermetallic system at temperatures where all atomic species are mobile, and after the irradiation has ceased, usually conform to the phase diagram as indicated earlier. However, microstructural details such as the precipitate size distribution may vary greatly with the

conditions of implantation. This is illustrated in Fig. 2, where AlSb precipitates in Sb-implanted Al are imaged by dark-field transmission electron microscopy for two samples.¹⁶ The implantations were carried out at 573 K, and all conditions were the same except for the ion dose rate (ion flux), which was smaller by a factor of 36 in the second micrograph. This single change is seen to have substantially increased the size and decreased the number of precipitates. Moreover, the difference occurs despite the absence of appreciable ripening by the conventional thermal mechanism, since further annealing of such precipitated samples at the implantation temperature produced no observable change. More generally, observed precipitate sizes in implanted alloys vary from the threshold of detection by transmission electron microscopy, ~ 1 nm, to about the thickness of the implanted layer. They tend to become larger and fewer with increasing implantation temperature or dose (ion fluence). The size distribution is controlled both by processes generally present in alloys and by effects unique to the implantation environment.¹⁹ Currently, a general theory for the size distribution capable of reliable prediction is not available, so that the microstructure should be determined experimentally when this information is important.

While an implantation is in progress, local thermodynamic equilibrium may be absent even though all atomic species are mobile, due to transient effects caused by the irradiation damage. For example, the ion beam continuously produces mobile vacancies and interstitials, which then undergo mutual recombination, or

form clusters, or migrate to fixed sinks such as extended defects and the surface. One result is an enhancement of atomic diffusion by the vacancy and interstitial mechanisms,^{20,21} which simply accelerates the evolution of the alloy. In addition, however, if the mobile point defects interact differently with solute and host atoms, the flow of defects to sinks may induce solute enrichment or depletion in the vicinity of these sinks, which persists until the irradiation is stopped.^{22,23} An example is given in Fig. 3, which shows the Si depth distribution in a bulk Ni-1 at.% Si alloy after irradiation with 3.5 MeV Ni⁺ at three temperatures.²³ In this alloy the flow of mobile defects to the surface sink has resulted in substantial Si enrichment within the near-surface region, whereas in annealed but unimplanted samples the Si distribution remained uniform. Such results illustrate the importance of the post-implantation annealing stipulated above for the attainment of local thermodynamic equilibrium.

Application to Metallurgical Studies

Ion-implanted metals have proved very useful for studies of alloying phenomena in the higher-temperature regime where most of the phases are in thermodynamic equilibrium. Thus, when the initial atomic mixture is formed nonthermally by implantation, the extent of thermally activated processes such as diffusion and precipitation are subject to a unique degree of control. Moreover, the atomic intimacy of the starting alloy speeds the approach to equilibrium and often removes the necessity of following an extended path on the phase diagram to the point of interest.

Interfacial problems, such as reduced interdiffusion due to oxide barriers, are bypassed. In addition, the close control over the depth distribution facilitates the formation of multilayered systems, and this allows subsequent solute interactions to be followed through the composition-versus-depth profile. Another important property is that the entire alloyed system is microscopic in one dimension, greatly reducing the time required for diffusion-limited processes. Furthermore, in at least some cases, the evolution can be further accelerated through enhanced diffusion under ion irradiation. The above features are particularly useful at temperatures where the atomic diffusivities are small, and also in separating phenomena which occur in more complex, multicomponent systems. Indeed, in a number of cases, the use of implantation has permitted the acquisition of previously unavailable metallurgical information. Examples of such applications will now be taken from work on diffusion, phase diagrams, and solute trapping by precipitates.

Diffusion rates and solid solubilities may be determined by ion implanting the solute into the near-surface region of the metal host, and then monitoring the concentration-versus-depth profile as a function of annealing. This procedure is illustrated by the depth profiles for Cu in Be shown in Fig. 1, which were obtained by ion backscattering analysis.¹⁸ The diffused tail in the single-phase region after annealing has the functional form

$$C(d,t) = C_0 \operatorname{erfc} \left(\frac{d - d_0}{N_H \sqrt{4Dt}} \right), \quad (1)$$

where C_0 is the solid solubility, D is the diffusion coefficient, d is the depth in units of atoms per unit area, d_0 is the particular depth at the intersection of the tail with the peak, t is the accumulated anneal time, and N_H is the atomic density of the host phase. From Eq. 1, it is seen that the time evolution of the diffused tail yields the diffusion coefficient, while the solubility is equal to the concentration at which the tail intersects the precipitated layer.

Diffusion²⁴ and solubility¹⁸ results from these and similar measurements are given in Figs. 4 and 5, where open symbols represent the ion beam data and the solid points are taken from conventional measurements reported in the literature.²⁵⁻²⁷ The two sets of diffusion coefficients shown in Fig. 4 correspond to the inequivalent directions of diffusion in the hcp Be lattice. Because of the microscopic scale of these implantation experiments, it was possible to extend both the diffusion and solubility data to substantially lower temperatures than achieved previously. Moreover, the solubility measurements were extended still further by using enhanced diffusion under ion irradiation. These lower-temperature diffusion-rate and solubility data are important for the control of precipitation when Cu is used for solution strengthening of Be.²⁸

The above approach can be extended to multicomponent alloys, and also to boundaries on the phase diagram other than those which border the host phase. An example of this is provided by studies of the Be-rich region of the Be-Al-Fe phase diagram,¹⁴ for which

a schematic isotherm is shown in Fig. 6. Here the portion of the diagram containing the α -Be phase is expanded by a factor of $\sim 10^3$ relative to that about the δ phase. Compositions listed for the phases on the figure are only nominal, since in several cases there is an appreciable range of composition. The points labeled 1-6 were investigated in the experiment. Ion implantation was used for alloy formation, with depth profiling by ion back-scattering analysis and identification of the precipitated phases by transmission electron microscopy.

Points number 1 and 2 in Fig. 6 correspond respectively to the binary solubilities of Fe and Al in Be, and were obtained as described above for Cu in Be. In the case of points number 3 and 4, the coordinates are the Al and Fe solubilities associated with a three-phase equilibrium. Their determination is illustrated by the procedure used for point number 3. In this case the Be host was implanted with Fe and with a sufficient excess of Al to precipitate both the β and δ phases during subsequent annealing, as confirmed by transmission electron microscopy. The solubilities of Al and Fe were then taken from the respective diffused tails, as in the binary measurements.

Points number 5 and 6 in Fig. 6 represent the limiting compositions of the ternary δ phase when it coexists with α -Be, and the Al-to-Fe ratio was measured for these extremes. This was accomplished by implanting the two solutes at different depths initially, with a three-fold excess of one species. During subsequent annealing, their reaction to form the very stable

δ phase was followed in the composition-versus-depth profile. When the reaction saturated, leaving unreacted a portion of the excess solute, an extreme composition was assumed to exist. Depth profiles for the Al-rich case at 873 K are shown in Fig. 7, where the initial and saturated conditions are given. The implanted Al and Fe are initially well separated, but after annealing a portion of the Al has migrated to form a new peak which coincides with the Fe peak. The Al-to-Fe atomic ratio in this region is found from Fig. 7 to be 1.4, which is believed to characterize the composition of the δ phase in its Al-rich extreme.

The results for the Be-Al-Fe phase diagram at one temperature, 1073 K, are listed in Fig. 7. Previously, no quantitative information was available in the Be-rich, ternary region of this diagram, despite its importance for the control of Al and Fe impurity effects in commercial grades of Be.²⁹ One notable feature of the data is the strong influence of Al on the Fe solubility, which decreases by more than an order of magnitude from point number 1 to point number 3. This is a consequence of the high stability of the ternary δ phase.

Solute atoms may be immobilized by trapping mechanisms which do not involve incorporation into a precipitated thermodynamic phase, and this is frequently important in determining the effects of dilute impurities on alloy properties. In characterizing such phenomena, the use of ion implantation complements more conventional metallurgical procedures. For example, implantation and

annealing may be employed to form a dense array of the trapping entities within a limited depth interval, so that the flow of solute to and from traps can be observed in the composition-versus-depth profile. In addition, implantation may be used to introduce the solute at a desired depth. This approach is exemplified by studies of the trapping of the embrittling element Sb in Fe-Ti-C alloys.³⁰ In these experiments, Fe containing ~ 0.01 at.% C as a bulk impurity was ion-implanted with Sb and Ti at room temperature, and then annealed isothermally at 873 or 973 K. Depth profiling was accomplished by ion backscattering analysis, and the microstructure was determined using transmission electron microscopy.

Representative concentration profiles before and after annealing are shown in Fig. 8 for the case where the Sb and Ti were initially introduced at different depths. The Ti distribution could be determined only in the region of its peak because of the similarity of the Ti and Fe nuclear masses. This profile was essentially unchanged by the 0.5 hour anneal at 873 K, and is given by the dashed line in the figure, scaled down by about a factor of 4 in concentration to facilitate comparison with the Sb. In contrast to the Ti, the Sb is seen to have undergone substantial redistribution during the anneal, and to have formed a new peak which conforms to the Ti profile. This behavior did not occur in Fe samples implanted only with Sb; instead, the evolution during annealing was qualitatively similar to that

shown in Fig. 1.¹⁷ Specifically, a diffused tail emerged from the initial peak at a concentration equal to the binary solubility of Sb in Fe, which is indicated in Fig. 8. These results are taken as evidence that the Sb is attracted to the Ti-rich region of the Fe.

With continued annealing, a lower-amplitude Sb tail eventually appeared from the Ti-rich region toward greater depths. However, the maximum concentration in the tail was smaller than the binary solubility of Sb in Fe by an order of magnitude. Moreover, this concentration decreased with increasing anneal time, in direct proportion to the amount of Sb still associated with the Ti. The latter behavior is shown in Fig. 9 for an isothermal series of measurements at 973 K, where the accumulated anneal time is given at each data point. Such a variation is inconsistent with the existence of the Sb within a bulk thermodynamic phase, but it is expected for occupation of traps when the occupancy is small compared to one. Consequently, the observed affinity of the Sb for the Ti-rich region is considered to result from the presence of trap sites.

The trapping entities in these experiments are believed to be small (~ 2 nm) TiC precipitates. Transmission electron microscopy and nuclear reaction analysis³¹ of the near-surface region for C have shown that these particles form within the Ti-implanted layer during annealing, in a time short compared to the duration of the experiment. The highly mobile interstitial C is getterred

from the underlying bulk of the sample and possibly from the environment. This trapping mechanism is not surprising in retrospect, since the large Sb atom has a known affinity for lattice boundaries in Fe.³² Its discovery has potentially important implications for the control of the grain-boundary embrittlement of steels by Sb impurities,³² because such trapping would inhibit the Sb migration to grain boundaries. Indeed, this effect may contribute to the observed but previously unexplained reduction of grain-boundary embrittlement in a steel containing 0.1 wt.% Ti and dilute Sb impurities.³³

Summary

The evolution of an intermetallic system following ion implantation depends strongly upon temperature: when all atomic species are immobile, the effects of thermally activated processes are minimal; in the intermediate temperature range where interstitials are mobile but substitutional atoms are not, the extent of equilibration depends upon the specific conditions, such as whether precipitation can result from interstitial transport alone; and at temperatures sufficiently high for all atoms to be mobile, a local state of near-thermodynamic equilibrium is usually achieved within the implanted region. In the highest of these three temperature regimes, the control and versatility of ion implantation make it a powerful tool for the study of alloying phenomena in metals, as exemplified by studies of diffusion, phase diagrams, and solute trapping.

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Figure Captions

- 1) Concentration profiles of Cu ion-implanted into Be, a) before and b) after annealing at 673 K.¹⁸
- 2) AlSb precipitates formed by Sb implantation into Al at 573 K for two dose rates.¹⁶
- 3) Silicon redistribution in a Ni - 1 at.% Si alloy caused by 3.5 MeV Ni⁺ bombardment.²³
- 4) Diffusion coefficients of Cu in Be for the a and c crystallographic directions. The open symbols are ion beam data,²⁴ while the solid points are taken from the literature.²⁵
- 5) Solid solubility of Cu in Be. The open symbols are ion beam data,¹⁸ while the solid points are taken from the literature.^{26,27}
- 6) Schematic isotherm of the Be-rich region of the Be-Al-Fe phase diagram. The points labeled 1 - 6 were investigated using ion implantation, and the results at 1073 K are listed.¹⁴
- 7) Concentration profiles of Al and Fe in Be a) before and b) after annealing at 873 K. These data were used to determine the maximum Al-to-Fe ratio in the ternary δ phase.¹⁴
- 8) Concentration profiles of Sb and Ti in Fe before and after annealing at 873 K, showing the Sb trapping in the Ti-rich region.³⁰ The Ti profile is scaled down by a factor of 4 in concentration to facilitate comparison with the Sb.
- 9) Relative variations of trapped Sb and the concentration of Sb in solution, as a function of isothermal annealing at 973 K.³⁰

FLUX DEPENDENCE

Al (50 KeV Sb, 5×10^{15} Sb/cm², 300°C)

2.25×10^{13} Sb/cm², s

6.25×10^{11} Sb/cm², sec

(2f)

1073K

- ① [Fe] = 0.11 AT. %
- ② [Al] < 0.007 AT. %
- ③ [Al] < 0.02 [Fe] < 0.01
- ④ [Al] < 0.02 [Fe] = 0.08
- ⑤ [Al] / [Fe] = 1.4
- ⑥ [Al] / [Fe] = 0.9

α = HCP-Be

β = FCC-Al

γ = HCP-FeBe₁₁

δ = FCC-Al FeBe₄