Phase Stability Under Irradiation - A Review of Theory and Experiment

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Abstract

A critical review of the current state of the theories for phase stability under irradiation is presented. Starting from the primary damage structure a formalism is presented (following Nelson, Hudson and Mazey[10]) which is used to describe radiation enhanced diffusion, precipitate dissolution, metastable precipitate sequences and phase diagram modification under irradiation. In the case of precipitate dissolution it is shown that current theory has been misinterpreted and an alternative steady state analysis is presented which shows that that the particle distribution depends on the pre-existing distribution or on new nucleation.

Current theories for phase diagram modification under irradiation are critically reviewed and extended to cover the effect of radiation induced disordering and amorphization. For the former case a calculated phase diagram (Ti-Ru) is presented as modified by irradiation.

1. Introduction

Recent interest in radiation instability revives a topic which was a major issue in radiation damage for many years prior to the discovery of void formation. Several reviews exist of the situation as it appeared at that time.[1,2] More recent reviews have also been presented.[3,4,5] In this review a critical overview of the current state of the theory will be presented.

2. The Source of Radiation Structure Modification

The phenomenon of phase transformations under irradiation has been widely used to obtain information about cascade size and structure. Implicit in this are basic mechanisms for structural reorganization in cascades. Samples with long range order,for example,have long been known to progressively disorder under irradiation.[6,7] This has been used to provide information on cascade size and structure by electron microscopy using superlattice-dark-field imaging of the disordered cascade region.[7] The same displacement-mixing process causes solute precipitates to dissolve[8] when contained entirely by the cascade and this too has been used to measure cascade size.[9] This has been applied to larger precipitates by Nelson et al. [10] (NHM),in a model proposing both sputter dissolution and disordering dissolution.

In the case of precipitates (ordered or disordered) where a cascade overlaps the surface, one expects a mixing of the solute atoms to produce a region of enriched matrix; this is the dissolution model of NHM. The longer range scattering of high energy solute atoms from the surface by knock-on constitutes the sputtering process.

An even more dramatic mixing occurs in those systems where amorphous phase formation is possible.[11-17] A recent example[6] of this is in Zr_3Al which is rendered progressively amorphous by neutron irradiation after first disordering.

For individual displacement processes as in electron irradiation, all the phenomena referred to above may still continue if the temperature is low enough to freeze in the damage and allow it to accumulate.

The temperature effect controlling the rate of reversion toward equilibrium is of course quite general. We summarize the damage effects at a given temperature as a balance between irradiation disorder (with "disorder" used in the wide sense discussed above) and the reversion to thermal equilibrium. The latter is characterized by an irradiation enhanced diffusion coefficient D' due to the enhanced defect concentration. A good review of this enhancement is available[4] and the theory is well-developed.

The reversion to a lower energy state is therefore thermally activated since defect motion is required and irradiation enhanced. An equilibrium state is not necessarily the end product of the reversion process. The large energies involved in the production of defects by irradiation may be dissipated by processes which involve departure from thermodynamic equilibrium in other respects. The coupling of a defect flux to sinks with a solute flux to give solute segregation is an example of this and so are the changes in precipitate stability proposed by Russell[18] and Martin.[19]

3. Radiation Enhanced Approach to Equilibrium

When the radiation enhanced reversion is toward an equilibrium state, radiation enhanced diffusion is the clear explanation of phenomena in which equilibrium is attained under irradiation yet is normally unobtainable (or has not been attained in a given temperature range). These include radiation induced ordering (reviewed in ref. 20) and enhanced nucleation in sluggish systems.[21,22] This feature is clearly important for the Fe-Ni-Cr base alloys in which thermal transformation is notoriously sluggish.[23] Alloys which appear to have reached equilibrium as used, may in reality be far from it and under irradiation in the diffusion-enhanced temperature range, the true equilibrium structure may be attained under irradiation. The nomenclature of the subject will be confused if new precipitates forming by this process are referred to as "radiation induced". This phrase should be retained for true radiation induced phase changes which revert when the irradiation is removed. It is proposed that "radiation enhanced precipitation" be used for transformations in which equilibrium is attained at an enhanced rate. In experiments it is essential to make every effort to distinguish by testing for reversion either in the absence of irradiation or at a reduced dose rate.

In addition to increased diffusion, point defect fluxes can be effective in nucleation.[18] The view that vacancies are necessary for general homogeneous nucleation of incoherent precipitates[24] in order to relieve volume stress is in principle widely accepted. Whether it actually occurs is still debated[25] by those who argue for inhomogeneous nucleation on dislocation loops or solute clusters. The theory currently ignores interstitials,however,and requires simplifying assumptions about elastic constants of precipitate and matrix. Removal of such assumptions will permit more complex strain field interactions and may allow nuclei to behave as biased sinks.

Since the theory is used to predict a deviation from equilibrium further discussion follows in section 6.

4. Precipitate Dissolution

The models for precipitate dissolution due to Nelson, Hudson and Mazey[10] (NHM) take the scattering out of a precipitate particle caused by irradiation as

$$\frac{dV}{dt} = -4\pi r_o^2 y\ k \tag{1}$$

as described in section 2 above. V is the precipitate volume, r_o its radius, k the displacement rate and y a constant representing the number of atoms scattered from the precipitate surface unit area per d.p.a.

NHM present two models for y, the first being a "sputtering" from the precipitate surface when

$$y = \alpha/N\ . \tag{2}$$

N is the number of atoms per unit volume and α is the number of atoms ejected per displacement. NHM give a value of 10^{14} atoms/dpa/cm^2/sec for y.

Alternatively, a mechanism is proposed in which cascades dissolve a volume of precipitate each time a cascade intersects the surface; then

$$y = \ell f \tag{3}$$

ℓ being the cascade size (~100 Å) and f the ratio of the dissolved volume to the cascade volume (~1).

The flow of solute out of the precipitate (assumed to be uniform), gives a supersaturated matrix and eventually a diffusional back flow is established into the precipitate. NHM use the theory of precipitation growth from supersaturated solution[26,27] from which they assert

$$\frac{dV}{dt} = 3\, D'(c_R - c_e) r_o / c_p \ . \tag{4}$$

D' is the radiation enhanced diffusion coefficient, c_R is the enhanced concentration in the matrix, c_e the equilibrium matrix concentration, c_p the solute concentration in the precipitates and r_o the precipitate radius. Applying the boundary condition for the total solute concentration

$$C = \frac{4}{3}\, c_p \pi r_o^3 n + c_R \tag{5}$$

and combining the diffusion flux with the dissolution flux they obtain finally,

$$\frac{dr_o}{dt} = \alpha k + \frac{3D'(c_R - c_e)}{4\pi r_o c_p} - D' r_o^2 n \tag{6}$$

which is plotted in Figure 1.

NHM interpret this graph to show that particles smaller than a critical size will grow (dr_o/dt positive) and those larger particles for which dr_o/dt is negative will shrink. This is misleading since equation (4) is obtained from a cellular model in which all precipitates are the same size. What fig. 1 in fact shows is that under irradiation the precipitation ceases for all precipitates at the size defined by the $dr_o/dt = 0$ line instead of the larger size defined by complete precipitation. This is the $dr_o/dt = 0$ line in the absence of irradiation (shown dashed in fig. 1). Equilibrium precipitates at this size would all shrink to the smaller size under irradiation because of the higher concentration then in solution due to dissolution. The precipitate size is defined by the size of a matrix cell containing the precipitate and that is fixed by the number of precipitate nuclei (n) which in this model is quite independent.

The model of NHM contains good physical insight and is attractively simple so that it can be usefully modified to allow a description of the solute concentration at steady-state under irradiation. A full treatment will be presented elsewhere but the uniform scattering from a precipitate is treated as a solute "creation" term in a steady state solution of the diffusion equation. The boundary conditions are that $(\partial c/\partial r)_R = 0$ at the cell boundary $(r = R)$ and that the equilibrium concentration occurs at the particle surface, $c(r_0) = c_e$. The solution is well-known,

$$c(r) = c_e + \frac{ykr_0^2}{2D(R^3 - r_0^3)} [\frac{2R^3(r - r_0)}{rr_0} - r^2 + r_0^2](c_p - c_e) \qquad (7)$$

and had the form shown in Figure 2a. The steady-state cell boundary concentration is just

$$c(R) = c_e + \frac{ykr_0^2}{2D(R^3 - r_0^3)} [\frac{2R^3}{r_0} - 3R^2 + r_0^2](c_p - c_e) \qquad (8)$$

shown in Figure 2b as a function of r. Note that this reverts correctly to c_e in the absence of irradiation.

The cell boundary concentration increases as we increase the precipitates size at first since the sputtering is greater for larger particles. At very large r_0 the diffusion flow becomes very great since the precipitate interface is very close to the whole outer shell of matrix.

Most alloy systems contain a small volume fraction of particles so that the size distribution lies well to the left of the peak in Figure 2b. In such distributions cells containing small particles will experience a net solute loss until steady-state is achieved with all particles at the same size, all having shrunk overall.

The fact that larger particles can have the same c_R as small ones suggests the possibility of stable bimodal distributions in which very large particles are in steady-state with much smaller ones. Such bimodal distributions have been observed experimentally.[28,29] Very large particles, almost filling their cells, are not well treated in these models, however, since internal sputtering must occur.

Note that Figure 2b plots the final steady-state radius. This is always smaller than the original size as one might expect in a sputtering model. Before a steady-state can be established the concentration c_R must build up to the steady-state value. For particles with a very large spacing this implies that total dissolution will occur before a steady-state diffusion flow can grow. This has often been observed.[30,31]

Note that in Figure 2b the total solute concentration is not fixed and must be maintained by an appropriate combination of cells all of the same size but not necessarily having the same size of particle within them. In fact the idea of a constant cell size is somewhat artificial and relaxing that requirement would permit any combination of cells that filled space, maintained the total solute concentration and all had the same boundary concentration.

The conclusion of this analysis is therefore that no steady-state constant size of precipitate will occur; instead the distribution will reflect the original distribution of the particles before irradiation which fixes the cell size. Further nucleation, however, may produce changes if it occurs.

The actual distribution of solute after sputtering or dissolution will not be uniform but peaked close to the precipitate. However, the steady-state concentration profile is established by the diffusion loss outwards from the local concentration maximum formed by sputtering (Fig.3). Most of the solute probably returns to the precipitate but a continuous solute loss to the matrix must occur down the outer concentration gradient. This continues until the c_R for steady-state is reached unless the particles dissolve before then. The uniform distribution is an approximation to this whole process.

An alternative possibility is that renucleation may occur in the solute enriched region adjacent to the precipitate (fig. 3) before a steady-state can be established. This is likely for low solubility solutes and under easy nucleation conditions. This was evidently the case in a recent study[32] of Ni-ThO_2 in which small particles of ThO_2 nucleated in haloes around the original large particles. This is a striking demonstration of the sputter phenomenon from precipitates. It also suggests how a bimodal distribution occurs initially.

A further suggestion in NHM[10] was that for precipitates smaller than ~100 Å the entire precipitate could dissolve in a single cascade. Experiments sensitive to the presence of very small particles show that they do indeed disappear. Piercy[9] demonstrated by magnetic measurement of Co particles in copper that the critical size was 12 Å diameter which suggests that only when a particle is in the core of the cascade does total dissolution occur.

There is disagreement over whether or not a steady-state size of precipitates is approached in the γ' nickel base alloys.[10,29,33] However, a general tendency to observe shrinkage as predicted is evident not only in the nickel base alloys but also in the other systems referred to above.[28-33] The sputtering model explains this without resort to the more sophisticated coarsening theory.[34]

Finally it should be noted that solute segregation when it occurs will drastically modify these effects. Regions depleted in solute precipitates will naturally dissolve while corresponding nucleation will occur in enriched regions.[29]

5. Metastable Precipitate Sequences

Under irradiation of supersaturated solutions the defect concentrations accelerate the nucleation and growth of the intermediate coherent precipitate sequences just as quenched-in vacancies do. One might predict that the interstitials could affect clustering processes for oversize atoms (which are limited under quenching conditions) and be promoted by solute interstitial doping.[35] No theoretical approach has yet been developed for this or for any appearance of new precipitates in the sequence under irradiation.

A further irradiation modification in the precipitation sequence is the rapid onset of incoherency by precipitate/dislocation loop interactions. The theory for this has been developed[36,37] and confirmed by experiments.[36-40]

6. Radiation Induced Phase Changes

There are many examples of phases forming on irradiation which are either not found on the equilibrium phase diagram or else are formed in composition ranges where they are out of place. It is suspected that many of these can be explained by solute partitioning due to solute-defect flux interactions.[40,41] The solute segregation will cause precipitates to form in what were originally single phase alloys (e.g., Ni-Si[19]).

However, there remain examples of wrong phase formation which cannot be explained in this way. Examples are found in steels[42] and strikingly, in the W-Re system.[43] This has stimulated the development of theories to explain phase diagram modification.[18,19,44,45]

The theory of Maydet and Russell[19] has already been noted as a description of incoherent nucleation under irradiation. The authors propose that (for a solute with larger atomic volume in the precipitate, i.e., "oversized"), volume strains caused by excess vacancy over interstitial annihilation at the incoherent interface, are relieved by solute absorption. Conversely for "undersize" solutes, precipitate dissolution is proposed to occur driven by the excess vacancy flux.

While this is convincing for nucleation from a supersaturated matrix it seems doubtful that it could modify established precipitates already in equilibrium with the matrix. An alternative event is prismatic dislocation loop punching which is commonly observed around incoherent precipitates and requires little energy expenditure compared with forced solubility changes.[36]

Martin[44] has proposed that the defects introduced by irradiation act like a ternary component. It was shown that in the case of strong defect/defect interaction a spinodal type solute/vacancy concentration fluctuation could be produced. The principal objection to such an approach is the small defect concentrations even under favorable conditions. It seems unlikely that "ternary" additions of 10^{-4} maximum could induce spinodals unless the binary alloy was already close to this.*

The same objection can be made to the proposal[45] that an irradiated phase is shifted to higher free energy because of its steady-state defect concentration. Only for enormous defect enthalpies could this produce the several hundred J/mol. required to modify the phase diagrams significantly.[46] Also high formation enthalpies are associated with low motion enthalpies. This implies easy defect motion and corresponding lower steady-state concentrations.

A recent modification of this idea[47] considers the anti-structure defects in strongly-ordering intermetallic compounds. It was observed in section 2 that the damage process induces disorder by its "stirring" action. This is balanced by an enhanced re-ordering due to defect motion. Both processes have been demonstrated experimentally (re-ordering[48-52] and disordering[6,7,53-56]).

At temperatures low enough to freeze out defect motion, re-ordering ceases and the mixing process can be observed. At these temperatures the ordered phases will disorder cumulatively even at low damage rates. If the ordered phase exists in equilibrium with its disordered version as in first order transitions,[20] it will then eventually disappear. At higher temperatures higher damage rates are required to achieve this due to the enhanced thermal re-ordering. At the highest temperature ranges where thermal diffusion dominates, radiation disorder will be lost in the

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*Note that this is distinct from the coupling of annihilation to solute fluctuations proposed by Martin in this conference.

thermal disordering and little departure from the equilibrium condition will be observed. The radiation modified phase diagram produced in such cases is shown in Figure 4a, b.

These general principles also apply to the amorphization induced by radiation damage which is also thoroughly experimentally substantiated.[6,15-17,57] The formalism of section 2 applies here also. An amorphization rate is obtained from the damage rate and balanced by the enhanced recrystallization rate. At low temperatures the amorphous (liquid) phase is frozen in. As the temperature increases higher rates of irradiation are required to produce the amorphous phase. The radiation modified phase diagram will then be as shown in Figure 4c, d. The amorphous phase is most easily produced for systems in which non-metallic bonding is important or in which substantial size differences exist, or both.

It is interesting to note that the mixing induced by radiation is analogous to that produced by temperature at equilibrium. Yet the radiation mixing is most evident at <u>low</u> temperatures. The diagram in Figure 4c shows a solid solution but the effect is general; the amorphous phase would subsume <u>any</u> other type of phase at high enough damage rates and low enough temperatures. However in a particular case this may not be practically attainable. The true liquid or amorphous state represents the ultimate in radiation damage since no displacements can make a random array more random.

Radiation does not affect all phases equally and the radiation induced disordering is particularly selective. In cases where a high ordering energy exists the radiation induced disorder at steady-state makes a large change in the ordered phase's free energy.[47] In the latter reference it is shown that this leads to the disappearance of such phases at lower temperatures and the establishment of a new equilibrium between the disordered phases on either side.

Figure 5 illustrates this for the Ti-Ru diagram in which the strongly ordered compound TiRu disappears at lower temperatures under irradiation rates typical of both heavy ion and neutron irradiations. A new two phase region then appears between the adjacent bcc Ti phase (β) and the hcp Ru (ε').

It is clear that this theory can produce quantitative predictions of phase diagram modification and more diagrams are currently being calculated. Experimental testing of the predictions is next required. The type of radiation phase diagram presented here is obviously useful as a point where theory and experiment meet.

7. Acknowledgements

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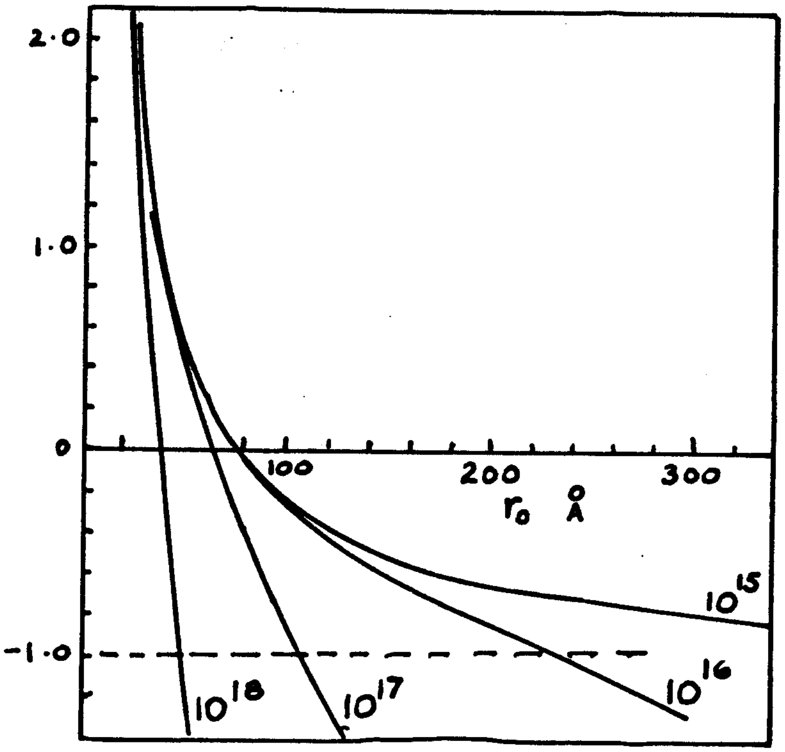


Fig. 1 Nelson et al.'s results[10] for precipitate growth rate versus precipitate size for different values of precipitate density n.

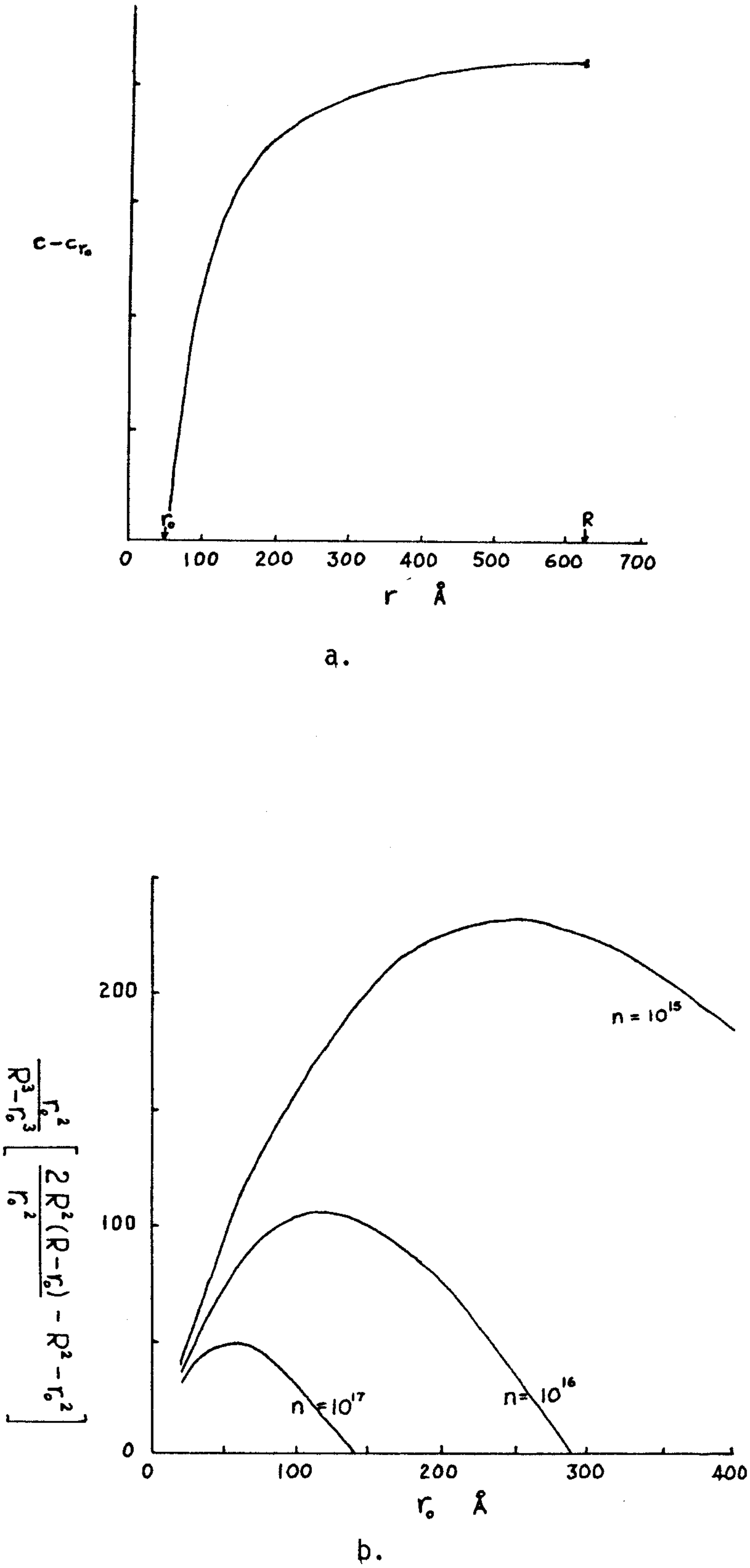


Fig. 2 a) Steady state concentration across the precipitate cell according to Eq. (7).
b) The cell boundary concentration for fixed cell size and sputtering rate. (Eq. (8)).

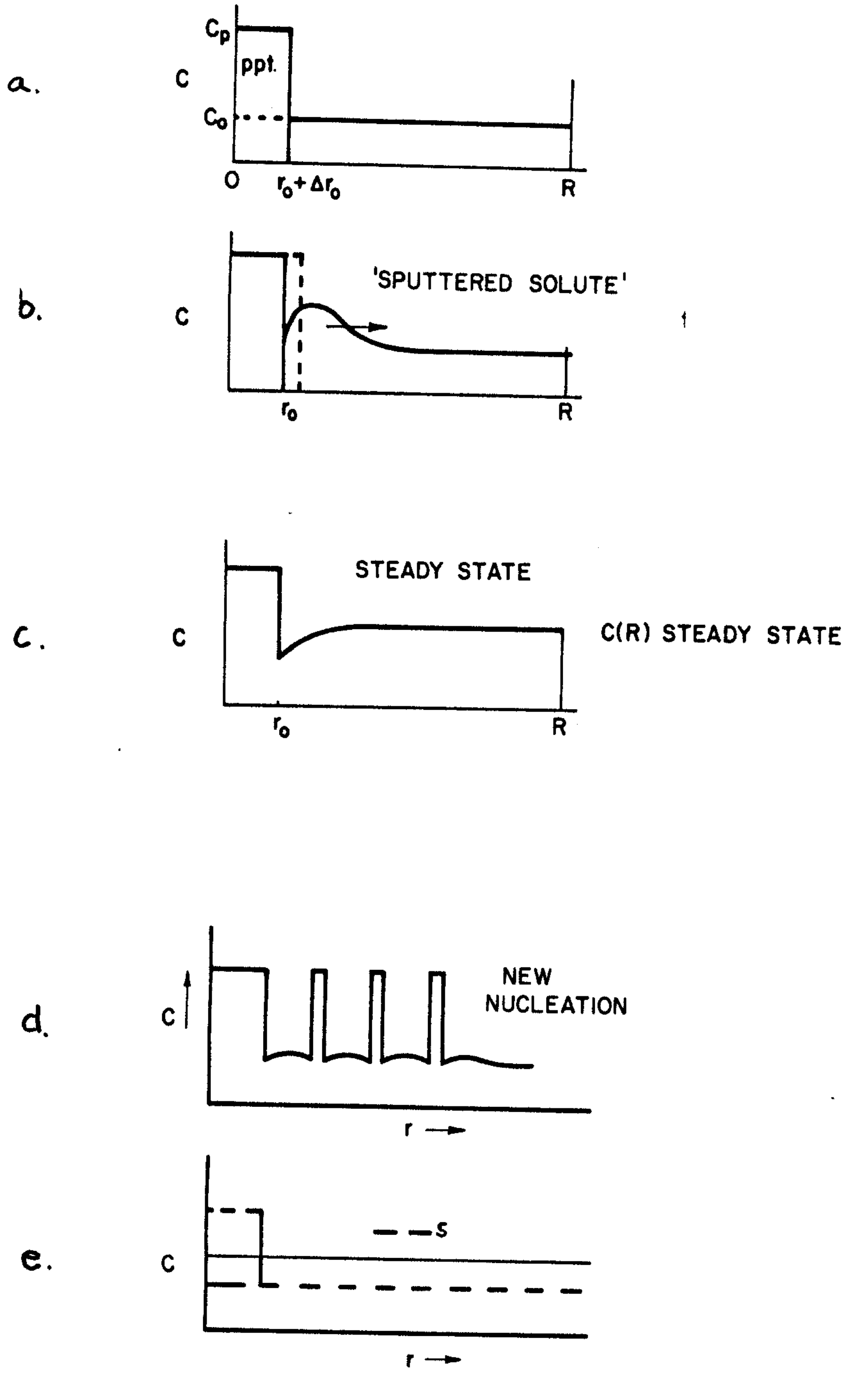


Fig. 3 Sputtering from a precipitate's surface giving rise to a steady state distribution (a-c) or (d) to new nucleation, or (e) to dissolution. In (e) the dashed lines represent the supersaturation necessary for nucleation.

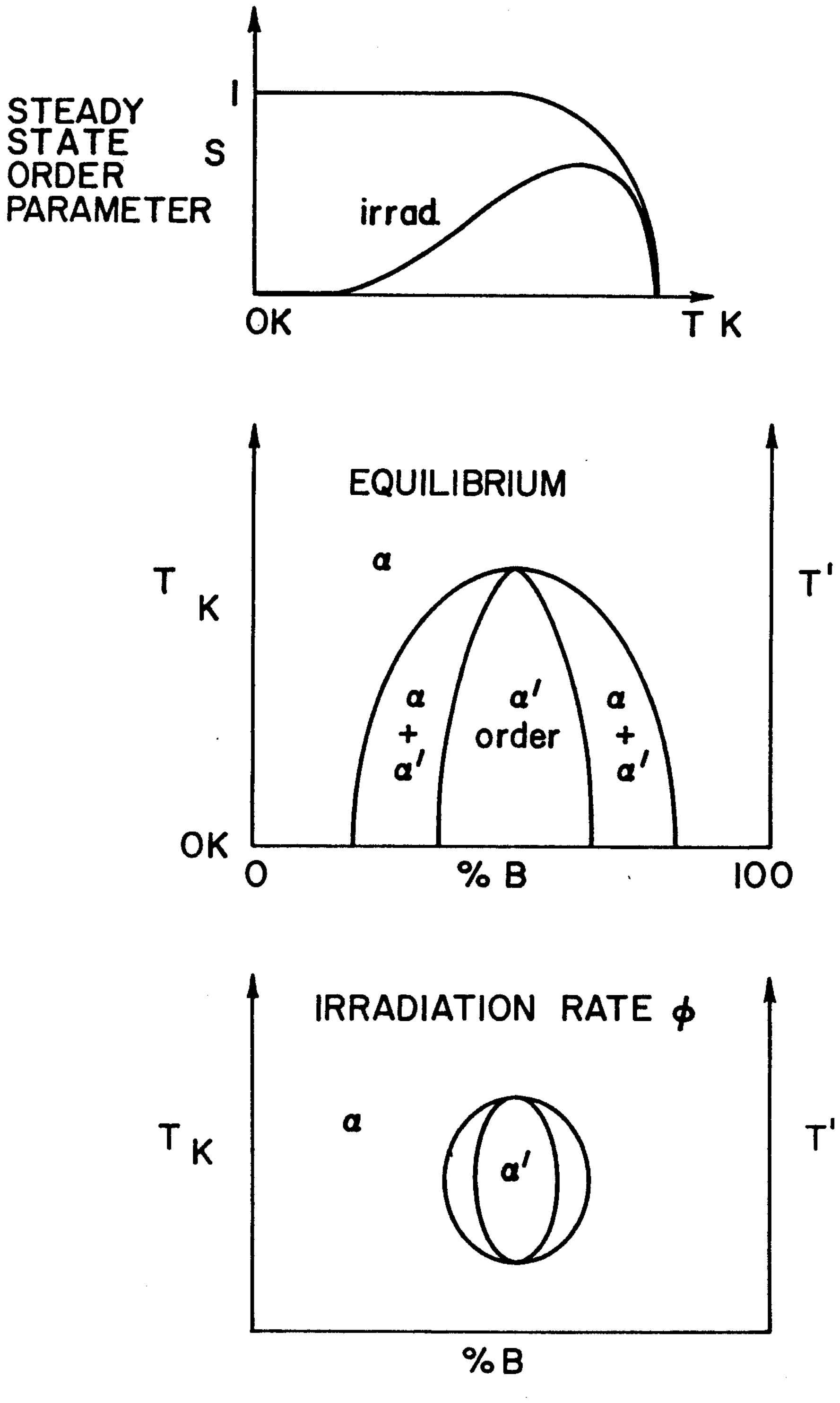


Fig. 4a Upper diagram - the change in degree of long range order with and without irradiation. Note the disordering effect (S decreasing) under irradiation. Center - an equilibrium phase diagram (no irradiation) for an ordering system. Lower - the effect of irradiation, creating disordering at low temperatures.

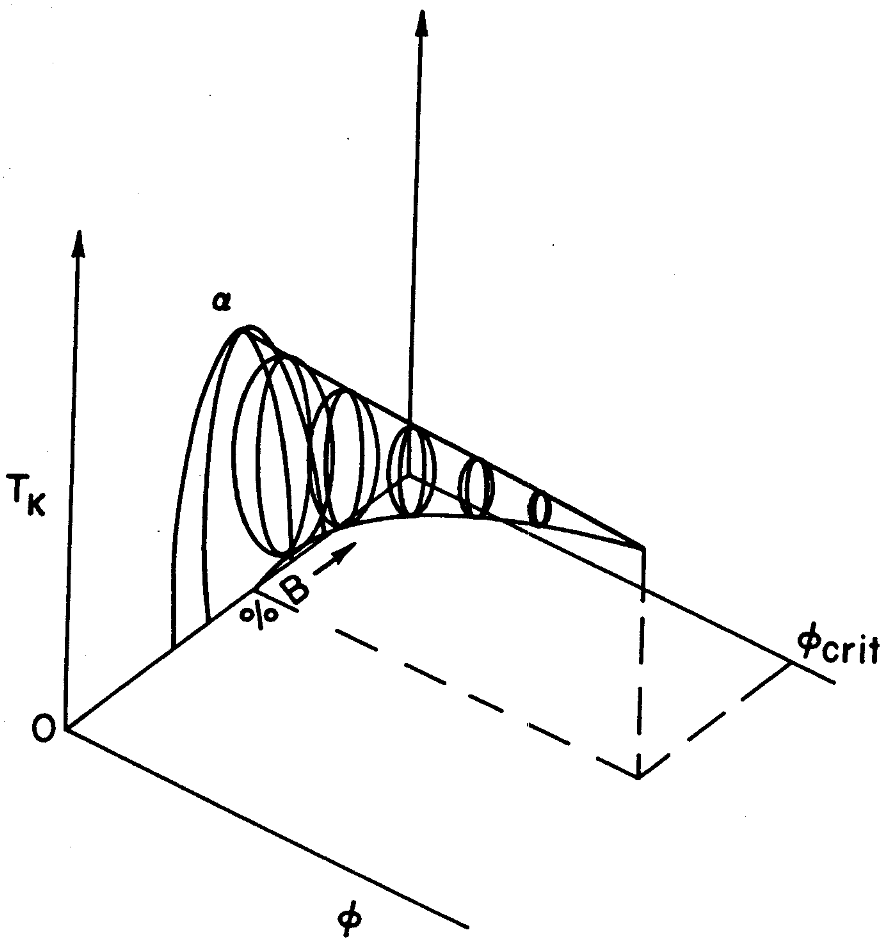


Fig. 4b The phase diagram with irradiation rate as a variable.

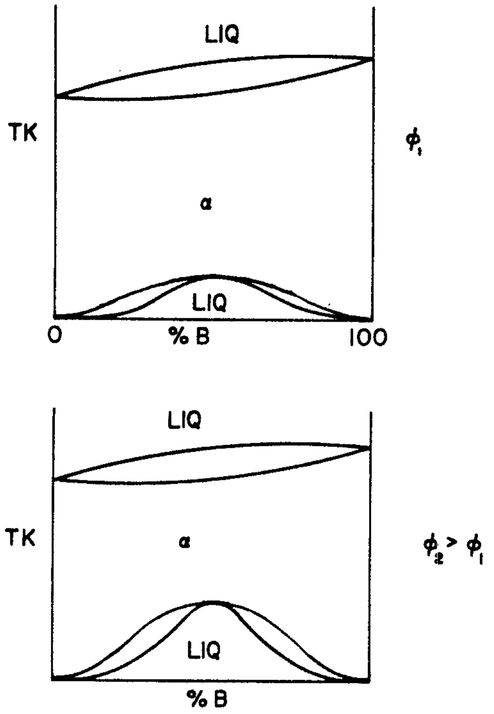


Fig. 4c The radiation modification of a phase diagram in which radiation forms the amorphous phase at low temperature.

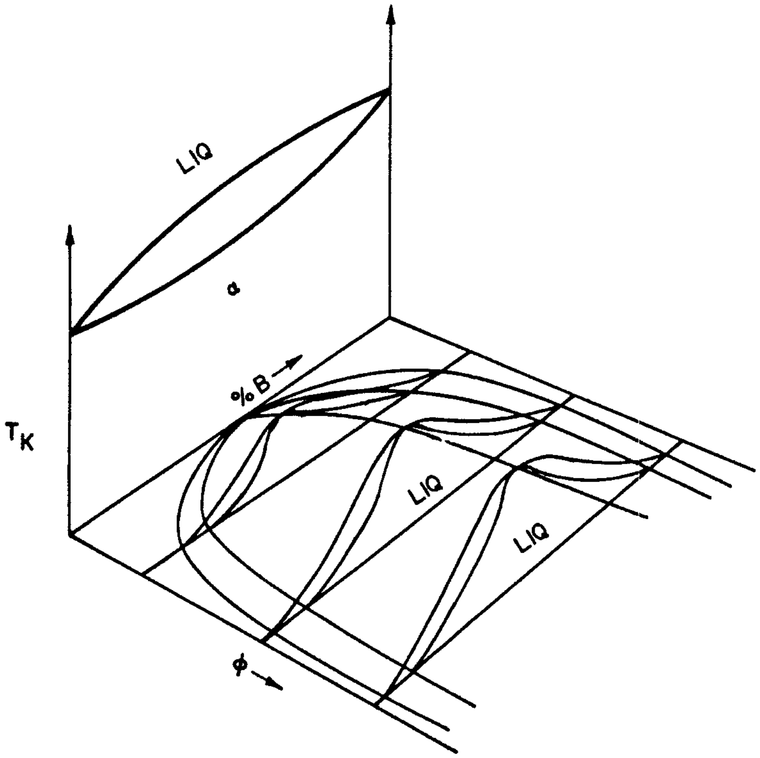


Fig. 4d The amorphous phase on a phase diagram with radiation rate as a variable.

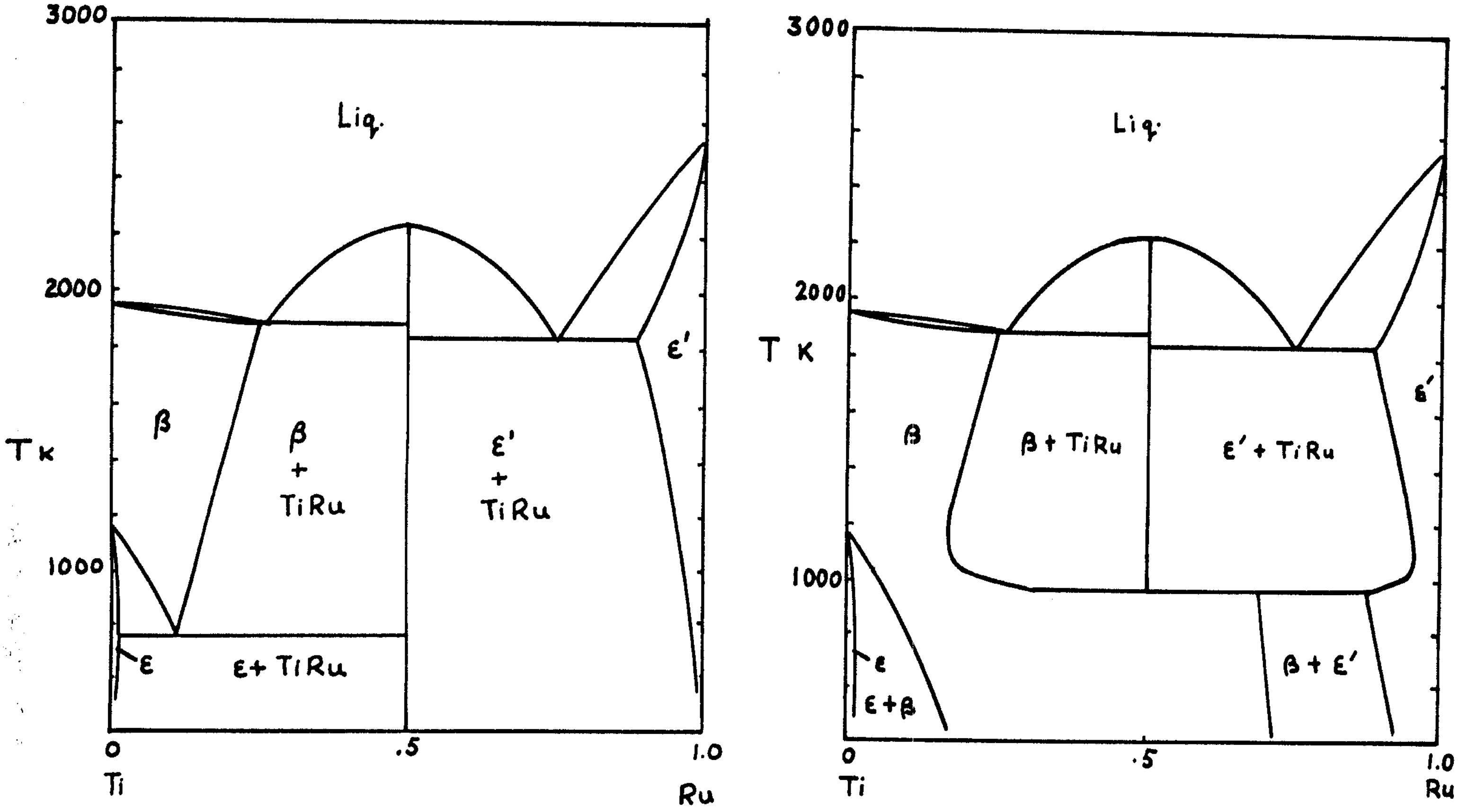
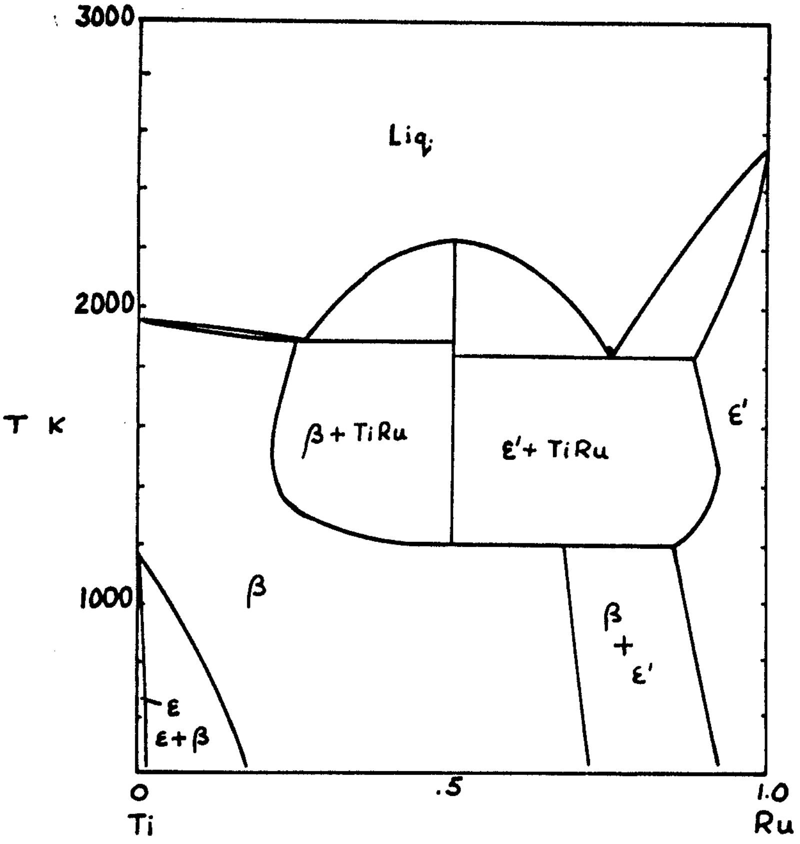


Fig. 5 The Ti-Ru system, a) as calculated in Ref. 48 at equilibrium (following Kaufman[58]). This is very close to the experimental diagram. b) As modified by a dose rate of 10^{-3} dpa/sec. c) For a dose rate of 10^{-7} dpa/sec.b) and c) are calculated using a dislocation density of 10^{11} lines/cm^2 as the only defect sinks.



5.c.