УДК 539.438

Fe-Ni INVAR ALLOY: AN EXAMPLE OF FRACTAL DECOMPOSITION?

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The important experimental results of irradiated and unirradiated Ni-Fe alloys and some approaches to explain the various contradictory behaviours are discussed. We outline the known results of the scattering experiments and suggest possible directions for theoretical investigations to explain the decomposition kinetics of the FeNi Invar alloy.

INTRODUCTION

The purpose of this report is to examine possible theoretical approaches to some of the unexplained experimental results on decomposition kinetic of the FeNi Invar alloy. The subject has received considerable attention. There exists in the literature a critical review of the experimental status on various properties related to the decomposition kinetics of FeNi alloys at various compositions as also at the compositions of the Invar alloy [Russell and Garner 1988, and Russell and Garner 1989]. On the basis of several measured properties, these authors suggest that there is a miscibility gap at the Invar alloy composition which is very narrow. Most recent experiments of Wiedenmann et al. [1989, 1990, 1992, 1994] appear to be the most reliable evidence for the decomposition of the alloy even though one should expect to see the signature of the decomposition in some direct experiments which one does not (TEM, FIM etc.) . Even so, we outline the known results of the scattering experiments and suggest possible directions for theoretical investigations.

CRITICAL DISCUSSION OF THE EXPERIMENTAL RESULTS ABOUT A FE-NI DECOMPOSITION

In this section we briefly summarize experimental results on Fe Ni which has been critically reviewed by Russell and Garner [1988]. Some relevant conclusions of this study will be recalled below.

The information on the decomposition tendency of Invar alloys comes from two sources. These alloys exhibit a near zero thermal expansion coefficient over a wide temperature range. For this reason they have been used in reactors as well. Under these conditions they exhibit remarkable resistance to dimensional changes arising due to void swelling under neutron as well as heavy ion irradiation. Therefore, some information about these alloys is available from such studies. Additional information regarding certain properties can also be obtained from metallic meteorites, which have the approximate composition of Fe-35 at.%Ni. In the critical review by Russell and Garner [1988], the authors examine evidence relevant to high temperature miscibility gap from variety of properties such as magnetic properties, thermal expansion coefficient, lattice parameter, electrical resistivity, interdiffusion coefficient and micro structural tools. Some of the conclusions they arrive at are important for the discussion attempted here.

Invar alloys exhibit low thermal expansion coefficient below Curie temperature. This gradual transition gives the volume contraction which approximately cancels out the volume expansion due to thermal expansion. Asano [1969] and Crangle and Hallan [1963] had attributed this gradual transition from ferromagnetic to paramagnetic phase to the development of microscopic compositional inhomogeneities. Further it is known that the Curie temperature of FeNi alloys increases with Ni contents. Due to this, lower Ni regions transforms at lower temperature compared to the regions of higher Ni contents. Asano suggest domain size of 60 atoms.

In the context of studies of thermal expansion Morita et al [1984] using splat quenching conclude that the results are consistent with decomposition into high and low Ni regions.

Lattice parameter measurements also indicate anomalous behaviour in the composition region of 25 to 45 % of Ni. Kacho and Asano [1969] find broad diffraction peaks which are indicative of decomposition into regions of different lattice parameter. The mean lattice parameter is considered as the mean of paramagnetic higher density lower Ni content regions with ferromagnetic lower density higher Ni content. Elastic constant $C_L = C_{11} + C_{12} + C_{44})/2$ also shows anomalous behaviour up to 200 K above the Curie temperature T_c according Hausch and Warlimont [1973], while one should have expected this anomalous behaviour for temperatures below T_c. They proposed that the alloy contained very small precipitates of Fe_3Ni and FeNi. Jago and Rossiter [1982] also found some extra diffraction spots.

Kondorsky and Sedov [1960] found anomalously large residual resistivity for alloys in 30…50%Ni which could be caused by some kind of fine scale heterogeneities. Kachi and Asano [1969] on the basis of Mossbauer spectra measurements came to the conclusion that this alloy consists of ferromagnetic and antiferromagnetic domains. The electron scattering was attributed to the scattering from the interface. Thermoelectric potential of paramagnetic FeNi measurements carried out by Tanji et al. [1978-1979] also show a pronounced low temperature hump around the same composition where

other properties also show anomalies. They attribute this anomaly to fluctuations involving large number of small Ni rich and Fe rich clusters. They note that the anomaly persist up to 1073 K.

There is some additional evidence from Mossbauer spectroscopy suggesting the possibility of an instability in this composition regime [Becker 1990].

Even though mixing enthalpies of FeNi alloys are negative indicating ordering rather than phase separation (Kubaschewski et al. [1977]), there is a pronounced hump around 25% Ni. This might be considered as evidence for phase separation in these alloys. On the basis of some of these studies Tanji and coworkers [1978-1979], suggest a coherent miscibility gap and coherent spinodal in the free energy. They also estimate the peak of coherent miscibility gap at 1100 K. Russell and Garner [1989] suggest that the coherent miscibility gap and the coherent spinodal region span a narrow composition range and these are depressed well below the chemical miscibility gap.

Additional important evidence for the existence of spinodal decomposition comes from the measurement of interdiffusion coefficient D. The measurement of interdiffusion coefficient D for Fe-Ni alloys for the entire range 0…100 %Ni at temperatures 1123…1373 K has been carried out by Nakagawa et al. [1979]. The results show a deep minima in D in the Invar region suggesting the proximity to the spinodal.

It is also known that a disordered Fe_3Ni phase with fcc structure and an ordered FeNi is found in meteorites of these compositions. The application of energetic particle irradiation (electrons, neutrons and ions) in order to accelerate the diffusion has clearly shown a phase separation in NiFe alloy of various compositions [Chamberod et al. 1979, Garner et al. 1986]. Very long wave lengths were observed under neutron irradiations [Garner et al. 1986], which could be explained if the dominant wavelength $\lambda_{max} = 2p/q_{max}$ in a spinodal decomposition is shifted due to irradiation-induced mixing [Abromeit &Krishan 1986, Abromeit & Martin 1999]. However, under proton irradiation such a wavelength were not observed. Instead a Porod law $d\sigma(q)/d\Omega \sim q^{-4}$ was found, which would be in agreement with the nucleation and growth in a system with radiation enhanced diffusion [Wiedenmann 1990].

On the basis of several of these results Russell and Garner [1988] postulate the existence of miscibility gap and the spinodal boundary which is narrow in the composition range having the peak around 1200 K.

The above discussion suggests that there is sufficient evidence that the Invar alloy is undergoing decomposition and the structure of the decomposition changes with increasing annealing time.

SUMMARY OF RESULTS OF SANS EXPERIMENTS

In this section we briefly recapitulate the main results of the experiments by Wiedenmann et al. [1989,1990,1992,1997] and the interpretation by the authors. Some effort has been made by these authors to provide a phenomenological basis for the experimentally measured decomposition kinetics of Ni-Fe alloys in the composition interval $0.26 < x < 0.45$. The main results to be considered here are taken from the Small Angle Neutron Scattering (SANS) differential cross-sections $d\sigma/d\Omega$ obtained from the Fe-34 at. %Ni alloy after subtraction of the magnetic scattering contribution. They are [Li et al. 1997] (Fig. 1 and Fig. 2 are examples taken from Li et al. 1997):

1) The scattering intensities show no pronounced maximum in the investigated interval of the scattering vector interval $\xi_{min} = 0.03\ nm^{-1} < q < 1\ nm^{-1} = \xi_{max}$.

2) The scattering intensity follows a potential law $q^{-\alpha}$ with $1.1 < \alpha < 4.0$.

3) The lower value ξ_{min} and the exponent α increases with the aging time t.

4) Both parameters can be scaled according to homologous time given by $t_h = t \exp(2.3eV/k_B(1/898 - 1/T))$.

5) The value of $\alpha(t_h)$ approaches 3 - 4, and $\xi_{min}(t_h) = \alpha(t_h)\ b$ with $b = 0.2$.

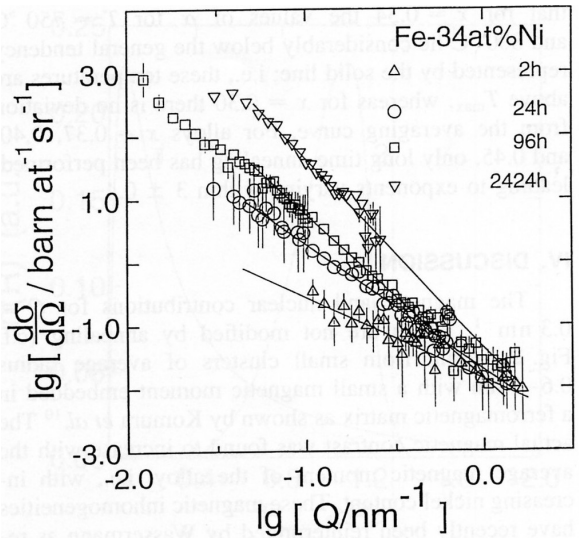

Fig. 1: The excess scattering intensity for x=0.34 after annealing at 675°C following power laws (solid line) with exponents increasing with increasing time (according Fig 6 of Li et al. 1997)

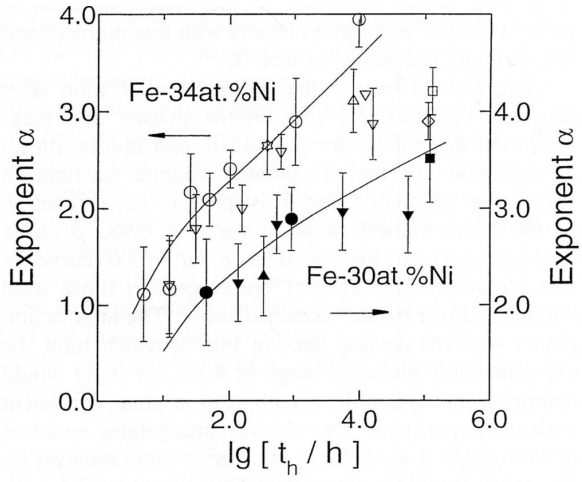

Fig. 2. The exponents $\alpha(t_h)$ as a function of a homologous annealing time t_h for x=0.34 (open symbols) and x=0.30 (full symbols). Different symbols characterize different time-temperature combinations (according Fig 7 of Li et al. 1997)

It is stated by these authors that these results can be explained only with the assumption of a fractal geometry of the decomposed material, with a maximum concentration fluctuation of Δc = 0.5…4 at%.

However, these results should be compared with the observations of three other experimental investigations performed on similar material. A Small Angle X-ray Scattering (SAXS) experiment by Simon et al. [Simon 1992] could not confirm a decomposition after a long time annealing, which may be due to a low sensitivity of SAXS for this special alloy system [Wiedenmann, 1994]. A first FIM-AP investigation of 34 at. %Ni alloy also failed as a martensitic transformation in the FIM tip was occurring [Li 1992]. However, using a highly sophisticated data analysis (repeated smoothing procedure RSP) Rьsing et al. [Rьsing 1998] could show in a Fe-37 at.%Ni Invar alloy that at 898 K a concentration fluctuation develops with increasing amplitude for increasing annealing time and a size larger than 2 nm. Decomposition could not be detected by Transmission Electron Microscopy (TEM) using the same samples that were used in SANS experiments [Wanderka, 1992]. It must be stated here that the scattering power of Fe and Ni are nearly the same for X-rays, electrons and neutrons. This might add to the complication.

CRITICAL DISCUSSION OF THE SANS RESULTS

As for the quantitative aspects, we examine some of the results of Wiedenmann and coworkers critically to see what results of the experiments should be taken seriously. We present below some discussion on points 2 - 5 of the last section.

α< 2: The low exponent value α is only seen for two scattering cross sections [Li 1992, Li 1997]. The scattering data corresponding to the lowest value of α ~ 1.08 has not been shown in these papers. In addition, the scattering vector interval is limited to ξ_{min}= 0.1 $nm^{-1} < q < 1\ nm^{-1} = \xi_{max}$ and shows significant error bars. A fit by a slightly curved function e.g., a broad distribution of frequencies would also be possible. Therefore, there is no compelling reason to explain values α < 2. Further, if the basic mechanism is triggered by decomposition of the alloy, values less than 2 are improbable since this would amount to the restriction of the concentration fluctuations (profiles) to two dimensions. However, scattering is not anisotropic. Instead, it is probable that the scattering could be from a surface fractal. But an analysis along similar lines would show that α > 2.

2 < α < 3: This range appears to be the most reliable region in the estimation of the value of α(t). The interval of the scattering vector for the scaling regime is ξ_{min} = 0.03 $nm^{-1} < q < 1\ nm^{-1} = \xi_{max}$. Any theoretical attempt should explain the value of $\alpha(t)$ and $\xi_{min}(t)$.

3 < α < 4: Only data in the scattering vector interval ξ_{min} = 0.03 $nm^{-1} < q < 0.1\ nm^{-1} = \xi_{max}$ is shown. They show a possible maximum at ξ_{min} for α ≈ 3. The value of α ≈ 4 is shown in the same way as the thermal data in their figure but this actually corresponds to proton irradiated results and does not fit in the homologous time. Since this dependence corresponds to proton irradiated situation, it also increases the pace of the kinetics by increasing the effective diffusion in the sample. This might perhaps explain the higher value of α = 4.

The scaling according to an homologous time should be taken as a general guideline but the activation energy 2.3 eV should not be taken too seriously.

It must be pointed out that the above emerging picture is the sum total of the evidence gathered on the basis of various properties and must be taken with some caution. A careful reading of the literature will show that there is considerable scope for interpreting these results differently and this can lead to conflicting evidences.

CLASSICAL APPROACHES FOR THE DECOMPOSITION OF ALLOYS

The main features of the scattering intensity profiles will have to examined critically in view of the fact that these results are not consistent with the principal feature of the decomposition of other metallic alloys. Before doing this we will recapitulate some basic facts about decomposition of alloys.

The conventional approach to modelling an alloy undergoing decomposition is based either on nucleation and growth process described by the LSW theory or by a spinodal decomposition [Gunton 1983]. The LSW theory predicts a well-defined size distribution f(R) of regularly shaped clusters of radii R having sharp interfaces [Lifshitz and Slyozov 1961]. The corresponding scattering cross section for monodisperse distribution of equal size is

$$\frac{d\sigma(qR)}{d\Omega} == \left(\frac{V_R \Delta\rho 3j(qR)}{qR} \right)^2 .$$

This gives for $qR << 1$ a Guinnier law $d\sigma(qR)/d\Omega \sim exp(-q(3R/5)/3)$, and for large $qR >> 1$ a Porod law $d\sigma(qR)/d\Omega) \sim q^{-4}$. However, a specially chosen size distribution $f(R)$ can in principal produce a power law dependence of $d\sigma(q)/d\Omega$ in the experimentally observed q regime by providing appropriate weight factor in the following way

$$\frac{d\sigma(qR)}{d\Omega} == \int f(R) \left(\frac{d\sigma(qR))}{d\Omega} \right) dR .$$

Such a distribution should be highly asymmetric with its peak at very small radii $R/\langle R \rangle \sim 0.1$ having a very long tail for large particle sizes. However, such a distribution cannot result from the known nucleation and growth theories [Lifshitz and Slyozov 1961]. This result is not changed essentially, if a distance distribution $G(D,R)$ of interacting particles of radii R and distance D is taken into account.

It is pertinent to note that the scattering intensity of a spinodal decomposing system normally exhibits a maximum in dσ/dΩ at some q_{max} with approximate power laws q^2 for $q < q_{max}$ and q^{-4} for $q >> q_{max}$. In the conventional experiments on decomposition, a power law with some exponent does not extend over more than an order in q.

From the above facts, the above authors conclude that both the LSW and the spinodal decomposition fail. The SANS data were therefore interpreted in terms of a fractal-like decomposition [Wiedenmann 1994]. However, the kinetics could not be explained by the authors. We will comment on this aspect later.

DIRECTIONS OF POSSIBLE THEORETICAL APPROACH

From the discussion presented in the previous section, we believe that any theoretical approach could take the following points with reasonable level of confidence.

a) The discussion in the previous section suggests that there is sufficient evidence that the Invar alloy is undergoing decomposition and there is evidence for the existence of a miscibility gap which is narrow centered around the Invar alloy composition.

b) The structure of the decomposition changes with increasing annealing time. As for the quantitative aspects, we need to explain the change in the exponent as a function of annealing time with the exponent value α in the range 2 - 3.

c) It may be important to consider the magnetic aspects the alloy for the decomposition kinetics.

d) Lastly, it may be crucial to offer some explanation of the scaling structure at the early annealing times.

It must be stated that the above experimental results are intriguing in several respects. First, the scattering intensity shows no conceivable maxima which is normally expected of a decomposing system. Second, this is coupled with the existence of a scaling regime, even though the interval is only one to two orders, and which is rather unusual since there have been no such reports for alloys undergoing decomposition. It is pertinent to note here that scaling regimes are seen in conventional spinodal decomposition from the intermediate time scales when nonlinearities govern the time development. In this regime normally dynamic scaling is resorted to, where in the typical length scale (which corresponds to the mean length of the A rich regime) constitutes the key to casting the structure factor in the scaled form. However, for the present case, this length scale cannot be fixed due to the absence of the peak in the structure factor. Even so, it may be still worthwhile to represent the structure factor for various times in this fashion by resorting to intelligent guess. In addition, in the present case, the scaling regime if anything is larger for smaller times, unlike the spinodal.

So the primary objective of any theory is to give a reasonable explanation for the existence of scattering intensity with only scaling property, (i.e. without a maxima) for all values of times. Due to this above unusual scaling feature, it may be worthwhile to split the problem into three parts each of which should be considered in parallel. They are given according to the time evolution of the decomposition process.

i) Starting from sudden deep quenches of solution annealed (1513 K , 48 h), the system already contains extended heterogeneities. The SANS scattering intensities for small $q < 3$ nm^{-}№ are different for different samples and were attributed to grain boundaries. However, they are not considered to influence the decomposition kinetics during the subsequent annealing [Wiedenmann 1994]. In fact, the existence of initial inhomogeneities may have a bearing on further decomposition.

ii) To consider the time development of the decomposition kinetics by considering the magnetic order parameter coupled appropriately with the concentration variable.

iii) It is possible that some additional relaxational kinetics may be involved. One way of including this is to carry out a Monte Carlo simulation.

Below we comment on each of the aspect based on known facts in the literature and some simple calculations that we have carried out.

EFFECT OF LARGE SCALE HETEROGENEITIES

In the following we attempt to include the effect of heterogeneities into the early stages of decomposition by assuming the Cahn-Hilliard's theory. Consider the early stages of spinodal decomposition where the local concentration deviation

$\delta c(r,t) = c(r,t) - \langle c \rangle$ with the average concentration $\langle c \rangle$ is given by

$$\Delta c(r,t) = \int d^3 q \exp(iqr) \exp(R(q)t) .$$

Here $R(q^2)=-Mf''q^2- MKq^4$ is the Cahn-Hilliard amplification factor, which is time independent. Due to the large heterogeneities we assume that that different regions of the sample is undergoing decomposition with slightly different dominant wavelength q_{max}. To include the effect q_{max}, we introduce a distribution of q_{max} given by $f(q_{max})$ into the above equation. Then the average structure factor takes the form

$$¿S(q)>=\int dq_{max}\, S(q, q_{\max}) f(q_{\max}).$$

The corresponding average correlation function $<G(r)>$ is the Fourier transform of $<S(q_{max}>$. The results have to be compared with the spatial particle-particle correlation function for a mass fractal structure of dimension D [Teixera 1986]

$$G(r)|_{fractal}=(D/4\pi)\xi_{\max}^{D} r^{D-3}\exp(-\xi_{\min} t).$$

The three dimensional Fourier transform of this function gives the desired power law in the range $\xi_{max} < q < \xi_{min}$ given by

$$S(q)=\int G(r)\exp(iqr)d^3 r=4\pi\int r^2[\sin(qr)/q$$

For D=2 the particle-particle correlation function $G(r)|_{fractal}$ and G(r) obtained for the early stages of spinodal decomposition has vary similar form if a broad distribution of q_{max} is considered so that the $G(r)$ is smeared out. This result may be improved if the higher order correction term according to Cook and Langer [Gunton 1983] is taken into account. Thus, it appears that spinodal decomposition in the early stages can be described by power laws for $q > q_{max}$.

COUPLED MAGNETIC AND COMPOSITIONAL ORDER PARAMETERS

From the discussion in section 2, it is clear that magnetic fluctuations are present during the decomposition. Thus a natural approach would be to consider a coupled Ginzburg-Landau type description for the two order parameter variables [Gunton 1983]. Let m(r,t) represent the magnetic order parameter. Then the spinodal decomposition of the system should be governed by the following equations.

$$\frac{dc(r,t)}{dt}=-\Gamma_c \Delta\frac{\delta F(c,m)}{\delta c};$$

$$\frac{dm(r,t)}{dt}=-\Gamma_m \Delta\frac{\delta F(c,m)}{\delta m}.$$

Here $\Delta=\nabla^2$, Γ is the mobility and $F(c,m)$ is the free energy density whose form has to be chosen appropriately for the present problem. The input for this choice has to be obtained from the literature on the magnetic properties of the Fe Ni system. In the above equation the relaxation of the nonconserved magnetic order parameter is much faster than the conserved order parameter. Thus, the competition between the two relaxing variables could give rise to much more complicated time development than in one variable. In the present case, since the miscibility gap is very narrow, particular attention must be made to include this feature in the free energy expression.

FAILURE OF DLA LIKE MODELS

A different approach could be based on a diffusion-limited cluster-cluster aggregation (DLA). Computer simulations have shown that irreversible aggregation processes can lead to ramified fractal aggregates [Meakin 1983, Kolb et al. 1983]. However, the minimum fractal dimension D for the Euclidian dimension d=3 obtained by this process is always significantly larger than 2 (D=2.4) which would not explain the observed value α=2. No time dependence of the fractal dimension with increasing cluster size is predicted.

In our opinion the formation of diffusion limited aggregates (DLA) like fractal structure is suspect for variety of reasons. First, diffusion limited aggregates are formed only in the limit of zero concentration, ie., a highly fluctuating situation where the aggregation proceeds with single monomers aggregating in an irreversible manner. This clearly is not consistent with the physical picture of an alloy decomposition where neither the zero concentration (ie., single particles being present in the neighbourhood of the aggregating point) is satisfied, nor the particles aggregate in an irreversible way. Second, the fractal dimension of such a cluster is D=1.7 in Euclidean dimension d=2 and D=2.4 in d=3. Further, the fractal dimension remains constant as a function of time. As can be seen these points are against the known facts in the present case. However, if there is possibility of the formation of concentration fluctuations with a ramified geometry, such a situation cannot be ruled out.

Granting the formation of DLA like structure, further evolution of these initial structures cannot be described in the framework of the theories given above. The structures are determined by the overall growth and additional atomic rearrangements on local scale due to thermal relaxation. They contain a dissolution and reaggregation of atoms and/or atom diffusion into regions of higher, local concentrations but smaller concentration gradients. There are several examples of a thermal relaxation reported in the literature [References in Irisawa et al. 1985, Toyoki 1985]. They predict the evolution of a ramified structure into a more compact form.

MONTE CARLO SIMULATION

In parallel to the analytical approaches, it is necessary to undertake a Monte Carlo simulation to get further insight into various mechanisms leading to ramified fractal structure and also to understand increase in fractal dimension as a function of time. Furthermore, in order to describe the transition from an spinodally created ramified structure into a more compact cluster-like structure, an appropriate description of the geometrical atomic arrangements is necessary. In the presence of small scale inhomogeneities arising from the magnetic

aspects of the alloy, different types of relaxation effects could be envisaged. It is worthwhile to investigate the effect of such relaxations on the kinetics [Ananthakrishna 1996]. From the experimental side, only the structure factor *S(q)* in the limited scattering vector interval $\xi_{min} < q < \xi_{max}$ is known. The required quantity is therefore the spatial density-density correlation function $G(\Delta R) = \langle \rho(\Delta R+R)\rho(R) \rangle$ with the main contribution in the length scale $\xi_{min}^{-1} > l > \xi_{max}^{-1}$ because *S(q)* is the Fourier transform of $G(\Delta R)$. It has to be checked whether it is possible to use the computer simulation results to construct the structure factor S(q), but also to model the initial and the final state of the structure.

COARSENING REGIME

In the final state of the decomposition, the alloy consists of well-shaped clusters with sharp interfaces between the matrix phase and the precipitated phase. These clusters are quite large and compact. Their contribution to the SANS scattering intensity are well described by a Porod law $d\sigma(q)/d\Omega \sim q^{-4}$. This late stage is obtainable under irradiation. The proton irradiation accelerates the relaxation into a more compact form by direct ballistic re-dissolution inside the ramified structure, but also be an enhanced growth and coarsening due to enhanced-diffusion.

DECOMPOSITION UNDER ENERGETIC PARTICLE IRRADIATION

An interesting test of this model is provided by the ion- and neutron irradiation results. The ion-irradiation already destroys the initial state of the decomposition in the spinodal region leading to long-wave fluctuations. Such process can be understood assuming a underlying thermal spinodal decomposition, however with a shift of a length scale due to ballistic diffusion under irradiation (Abromeit and Krishan 1986, Abromeit and Martin 1999). No thermal relaxation occurs in such structure. The fluctuation is conserved also after long times, which is in accordance with the experimental findings.

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Fe-Ni ИНВАР СПЛАВ КАК ПРИМЕР ФРАКЦИОННОГО РАСПАДА

С. Абромайт и Ж. Анантакришна

Обсуждаются важные экспериментальные результаты облученных и необлученных Ni-Fe-сплавов, а также некоторые аппроксимации для объяснения различных противоречивых характеристик. Мы подчеркиваем известные результаты, полученные в экспериментах с рассеянием, и предлагаем возможные направления для теоретических исследований, чтобы объяснить кинетики разложения FeNi ИНВАР сплава.

Fe-Ni ІНВАР СПЛАВ ЯК ПРИКЛАД ФРАКЦІЙНОГО РОЗПАДУ

С. Абромайт та Ж. Анантакришна

Обговорюються важливі експериментальні результати опромінених та не опромінених NiFe-сплавів, а також деякі апроксимації для пояснення різних суперечливих характеристик. Ми підкреслюємо відомі результати, отримані в експериментах з розсіянням, та пропонуємо можливі напрямки для теоретичних досліджень, щоб пояснити кінетику розкладу FeNi ИНВАР сплава.