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DIRECTIONAL AND SHORT-RANGE ORDERING KINETICS IN METALLIC ALLOYS, CRYSTALLINE AND AMORPHOUS

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<u>Résumé</u>. Nous présentons les méthodes d'analyse de la mise en ordre à courte distance et de l'établissement de l'ordre directionnel anélastique et leur application à l'étude des alliages métalliques, cristallins et amorphes. L'accent est mis sur la détermination de la mobilité atomique et des propriétés des défauts ponctuels. Les renseignements d'ordre structural qu'apporte la relaxation Zener au sujet de la transition ordre-désordre et de l'ordre directionnel auto-induit sont également discutés.

<u>Abstract</u>. This presentation describes the methods based on analysis of stressinduced directional ordering and short-range ordering and their application to the study of metallic alloys, crystalline and amorphous. It focuses on the determination of the atomic mobility and point defect properties. It discusses also the structural information which can be gained by Zener relaxation studies about the order-disorder transition and self-induced directional ordering phenomena.

I. INTRODUCTION

The state of short-range order is an important characteristic of concentrated alloys, since it reflects the existing interatomic forces between the constitutive atoms and influences a number of physical properties. A convenient description of the local atomic environments is provided by consideration of the Warren-Cowley parameters. An interesting feature of these parameters is that they are accessible to experimental determination by use of diffraction methods. X-rays or neutrons. However, the inherent sensitivity of these methods is in general not high enough to enable detailed study of the variations of the degree of order which occur during thermal treatments or irradiation. The object of the present paper is to describe indirect methods, resistometric and anelastic, which offer better potential to the determination of the ordering kinetics. In turn, these bring information about the atomic mobility and the fundamental properties of the elementary point defects involved. In some cases, additional information about structural aspects is also obtainable, in particular in conditions when order-disorder transition or selfinduced ordering phenomena take place, as discussed in the second part of this brief survey. The kinetic aspects are presented first.

Basically the physical parameter which will be considered is the rate at which local order evolves, in a solid initially at equilibrium, in response to a sudden modification of temperature or the application of a mechanical stress. This rate, τ^{-1} , is directly proportional to the atom jump frequency, v_{at} . Consequently, it is the product of the instantaneous concentrations c_d of the defects responsible for atomic mobility, and mean rate, v_d , at which atom defect interchanges occur. Hence

 $\tau^{-1} = \chi_{v} v_{at} = \chi_{c} c_{d} v_{d}$ (1)

 χ denotes the mean efficiency of an atom jump in promoting ordering by the vacancy or the interstitialcy mechanism. It is of the order of unity for vacancies [1].

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From the atomic diffusion coefficient D being related to the ordering characteristic time by $D = \lambda^2/6\tau$, where λ is the distance that an atom moves in one atomic jump [1], it is apparent that, by measuring τ , we obtain information about atomic transport. Ordering times of the order of 10⁵ seconds are readily measured. They are equivalent to diffusion coefficients of 10^{-21} cm²xs⁻¹, compared to the usual limits of $10^{-18} - 10^{-19}$ cm²xs⁻¹ ior tracer and other diffusion methods.

This inherent sensitivity has several important consequences : (i) ordering experiments can be conducted in a much lower temperature range than conventional diffusion experiments. Stated differently, atomic mobility can be studied in conditions of very low vacancy concentrations, typically 10^{-9} at⁻¹, which means that mostly monodefects are present; (ii) by combining diffusion and ordering data, coverage of 7 to 9 decades in c is possible as reported in α -CuZn and α -AgZn solid solutions [2,3]; (iii) since equilibrium short-range order is established with an average of a few atom jumps per atom, the desired information about atomic mobility is gained in a time lapse during which the constitutive atoms are not allowed to diffuse over more than a few interatomic distances. Thus ordering can be properly analyzed in out-of-equilibrium solid solutions and metallic glasses also.

II. EXPERIMENTAL APPROACHES, RESISTOMETRIC AND ANELASTIC

The two approaches most frequently followed to investigate ordering kinetics are mechanical after-effect experiments and electrical resistivity measurements. It is recalled that the resistivity associated with short-range order, $\Delta \rho_{SRO}$, referred to the one of a random solid solution, $\rho_{\alpha=0}$, is given by [4]

$\Delta \rho_{SRO} / \rho_{\alpha=0} \simeq \sum Z_i \alpha_i Y_i$

The α_j are the Warren-Cowley parameters, Z_j , coordination numbers and Y, a function which depends on the number of conduction electrons per atom and details of the electronic structure. Depending on these details, resistivity can be increased or decreased when the degree of SRO increases. Interestingly, by combining resistivity and neutron diffraction measurements, Wagner et al. [5] have shown that, over a wide temperature range which spans 300 K, $\Delta \rho_{SRO}$ is directly proportional to the first Warren-Cowley parameter, the one for the first coordination shell.

In resistivity methods, a typical procedure is one in which a sample is heat treated at a moderately high temperature and quenched in order to freeze-in the equilibrium degree of SRO characteristic of the anneal temperature. Then resistance is measured at a reference, low temperature. Cycling the anneal temperature gives rise to a reversible resistivity effect which reflects the temperature dependence of the equilibrium degree of SRO. Next, isochronal or isothermal anneals after quenching enable to follow the return of the as-quenched metastable disordered state towards the equilibrium as exemplified in section 3.

In Zener relaxation methods, the procedure is different. A change in the state of stress is applied to the sample at the appropriate time in the anneal or irradiation schedule and the strain response is studied as a function of time. The anelastic strain relaxation is then analyzed by methods described in the literature [1,7,10]. It is only recently that the more elaborate formalism developed in the anelastic field was applied to the treatment of the resistivity results [6,8]. Prior work (see for example [9]) had shown that the kinetics were identical for the two processes, short-range ordering and directional ordering, as could be expected from the two base mechanisms being very similar. A unique property of the methods based on analysis of the directional order, anelastic or magnetic [10, 11] is that owing to the delayed creep method proposed initially by Berry and Orehotsky [10] sensitivity remains the same during the whole time range from the initial defect population to indefinitely long times. [10, 11]. This makes possible the accurate study of the buildup or decay times for a defect super-(or sub-) saturation. Hence important parameters such as defect life-times and hence sink densities are inferred. Thermal generation of vacancies at internal sources can also be studied [12,13]. The evolution of a defect population is less easily followed by resistivity methods and care must be exercised

about the derivation of the true build-up or decay kinetics of a supersaturated defect population.

III. VACANCY FORMATION AND MOBILITY PARAMETERS

As concerns the determination of the activation parameters relevant to vacancy defects, both approaches offer comparable potential. We shall consider quench and resistivity experiments. A typical behaviour is illustrated in Fig. 1 for an α -Cu-30 at. % Zn solid solution. Quenches from 300 °C were followed by isochronal anneals at successively increased temperatures. Two types of ordering profiles can be distinguished depending on the sink density. If high, no evolution occurs until about 150 °C when the atomic mobility becomes high enough. Then the frozen-in disorder gets progressively eliminated until the equilibrium SRO at the anneal temperature is reached at about 240 °C. If the temperature is increased further, equilibrium line is followed. The dotted curve indicates the corresponding evolution of the vacancy supersaturation during the same anneal sequence.



<u>Fig. 1</u> (left) isochronal evolution of the resistivity excess induced by a quench from 300° C, in Cu-30 at% Zn. The two patterns shown are typical of specimens with a high and a low sink density, respectively. The upper curves are experimental. The lower curves indicate the estimated corresponding vacancy supersaturations (right scale) [9].

<u>Fig. 2</u> (right) Arrhenius representation of the ordering rates measured as follows (a) In conditions of equilibrium vacancy concentrations. The activation energy, 1.59 eV, refers to self-diffusion by a monovacancy mechanism. (b) As a function of the quench temperature, as derived from the initial slopes of a series of isochronal curves traced at 48° C. (c) As a function of the anneal temperature after quenches from 340° C. Vacancy formation and migration enthalpies, respectively are inferred.

When sink density is high, the excess vacancies are annealed out rapidly during the first anneals, that is in a very limited number of jumps, such that they cannot produce any significant ordering. After a brief transient period, only equilibrium vacancies are still present and ordering takes place by means of these defects. Consequently, only the selfdiffusion energy can be inferred. In contrast to this, in the case of a well annealed material with very low sink density, the supersaturated vacancies diffuse over long distances before annihilation. Along their diffusion path they produce a large amount of ordering. This results in a supplementary ordering stage which occurs at lower temperatures (fig. 1). But simultaneously the vacancy supersaturation is progressively decreased. When it vanishes the ordering is stopped. A second ordering stage is observed when equilibrium vacancies become again operative. It is located in the same temperature region as in the preceding case. Then, clearly, by analyzing the kinetics in the first ordering stage, direct information about the vacancy parameters can be gained. By changing the quench temperature, that is the vacancy concentration, the formation energy of vacancies is determined ; similarly by varying the anneal temperature. The migration energy of these defects is obtained. Thus the three activation enthalpies which

characterize the monovacancies can be studied by the same method, at neighbour temperatures. Fig. 2 is an illustration of the results for the α -Cu-30 at. % Zn alloy under consideration.

IV IRRADIATION STUDIES

In conventional irradiation studies, vacancies and self-interstitials are produced at low temperatures and stored before being analyzed during postirradiation annealing treatments. A somewhat different approach is one in which the irradiation is conducted at temperatures when both created defect species, self-interstitials and vacancies, have a significant mobility. Then the dynamic behavior of the corresponding defect populations is studied directly under flux. The physical parameter to be considered is the enhanced atomic mobility : it is monitored by the enhanced ordering rate $\tau^{-1} = X_V(c_V, eq^+c_V)v_V+X_ic_iv_i$ where the subscripts v and i refer to vacancies and self-interstitials, respectively. For the analysis of the results, use is made of the general balance equations proposed initially by Damask and Dienes [14] to describe the radiation enhancement of atomic mobility :

$$\frac{\partial c_{\mathbf{v}}}{\partial t} = \sigma \epsilon \phi - 2c_{\mathbf{i}}(c_{\mathbf{v},\mathbf{eq}} + c_{\mathbf{v}}) (v_{\mathbf{i}} + v_{\mathbf{v}}) - \rho c_{\mathbf{v}} v_{\mathbf{v}}$$
$$\frac{\partial c_{\mathbf{i}}}{\partial t} = \sigma \epsilon \phi - 2c_{\mathbf{i}}(c_{\mathbf{v},\mathbf{eq}} + c_{\mathbf{v}}) (v_{\mathbf{i}} + v_{\mathbf{v}}) - \rho c_{\mathbf{i}} v_{\mathbf{i}}$$

In these expressions, the production of freely migrating defects is due to flux ϕ , with efficiency equal to the product of the displacement cross-section σ and the number ϵ of uncorrelated Frenkel pairs emitted by primary knock-on. Their destruction is both by mutual annihilation characterized by a recombination factor Z and by elimination at fixed sinks having a density ρ ; $c_{V, eq}$ is the equilibrium vacancy concentration at the irradiation temperature. Without entering into details, one can feature the evolution pattern with time-of-irradiation for the resulting relaxation rate as follows. First, on application of flux, a build-up transient occurs, until a quasi-stationary maximum is reached. On further irradiation, elimination at fixed sinks, such as dislocations, starts being significant, resulting in a decreased rate, until a stationary level is reached.

It is readily shown that the quasistationary and the stationary rates are governed by the faster and the slower moving defect species, respectively and related very simply to the jump rates of these defects by

$$\tau_{qst}^{-1} \approx \begin{bmatrix} \sigma \epsilon \phi \\ z \end{bmatrix} \mathbf{x} \mathbf{v}_{f} \end{bmatrix}^{1/2} \qquad \tau_{st}^{-1} = \begin{bmatrix} \sigma \epsilon \phi \\ z \end{bmatrix}^{1/2}$$

Thus experimental observation of such a profile is able to bring detailed information about the defect mobilities and all the other parameters which control the dynamics of the defect population under permanent flux [16, 17]. Investigations of this type were conducted in our laboratory with employment of mechanical after-effect measurements [15-19]. An inverted torsional pendulum with negligible inertia was adapted to work in-line with a Van de Graaff accelerator. A resonant cavity system was devised also to perform in situ anelasticity measurements in the core of a nuclear reactor. Both devices enabled rate measurements to be performed continuously during the irradiation. Fig. 3 shows a rate curve traced in a Ag-24 at % Zn alloy, at 60°C, in a constant electron flux. It can be seen that, after a rapid rise, a quasistationary maximum occurs, after some hundred seconds of irradiation, in accordance with the pattern predicted by the model. The interrupted line indicates the result of a computer calculation obtained by plugging into the balance equations the numerical values for the various physical parameters involved, as determined in specific studies [15-18]. Agreement between the calculated and experimental curves is good. Among the results, one point is to be emphasized. From the ratio of the quasistationary to the stationary rates being about two and a half, it is Inferred that the mobilities of vacancies and self-interstitials differ by only a factor of six, which is surprisingly small Deeper insight is brought by consideration of activation energies, fig. 4. For this purpose, the rate profile was traced at various temperatures. Activation energies of 0.56 eV and 0.82 eV for the migration of the faster and the slower moving defects were found.

The essential picture which emerges from the results is the inversion in the mobilities of the vacancies and the self-interstitials [16,17]. The physical reason is that the self interstitials are slowed down by the presence of the undersized solute atoms at the extent that they become slower than the vacancies, as is probably the case in all substitutional alloys with a marked difference in atom size between the constitutive elements.



<u>Fig. 3</u> Relaxation rate versus time of irradiation in an Ag-24 at % Zn solid solution exposed to a constant electron flux of $1.6 \times 10^{11} \text{ cm}^{-2} \text{s}^{-1}$, at 60°C [15]. Both the solid line and the data points are experimental. The former was derived from the direct relaxation curve by local slope analysis [10]. The data points are initial slopes of a family of relaxation curves obtained by alternately loading and unloading the specimen. It is apparent that relaxation experiments yield the ordering rate with the same accuracy over several decades in the time scale.

<u>Fig. 4</u> Arrhenius plot for the anelastic ordering rate in Ag-24 at % Zn [15]. The left curve refers to the relaxation rate measured in zero flux, under thermodynamical equilibrium conditions for the vacancy concentration. The other two curves indicate the ordering rates for the quasistationary and the stationary conditions under the same electron flux as for the preceding figure.

All the other parameters which intervene in the above balance equations can be determined equally well, in particular recombination volumes [18]. The specific interest of this present approach is that it enables exploration of a temperature range, generally 0.3 to 0.5 of the absolute melting temperature, in which experimental data are scarce Another interesting potential of the ordering methods is that they enable one to discriminate between close-pair or correlated recombination or else, in the case of irradiations with heavy particles, those defects which are confined to their native displacement cascades and the long-range diffusing ones. An application to the determination of the efficiency of neutron displacement cascades in producing free defects, that is defects escaping recombination or clustering within the cascade, has been reported [19].

V. SELF-INDUCED DIRECTIONAL ORDERING

Another area of recent progress is the study of self-induced ordering. Indeed, a parameter of particular interest is the relaxation strength, Δ , which characterizes directional ordering. The various theories for the Zener relaxation predict a temperature dependence of the type $\Delta = \text{To}/(\text{T-T}_{\text{C}})$ [1,20]. T₀ is a constant and T_c, the critical temperature for self-induced ordering. A non-zero value of T_c means that the free energy levels depend not only on the stress but also on the existing state of order in the alloy. In terms of relaxing entities, this means that these would all spontaneously align themselves into a single orientation, if the necessary atom movements could occur. This phenomenon is analogous to the onset of ferromagnetism below a Curie temperature, while the $1/(\text{T-T}_{\text{C}})$ dependence of Δ is akin to the Curie- Weiss law for the paramagnetic susceptibility above the Curie temperature.

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A situation of this sort could recently be observed experimentally, in an α -Au-30 at % Ni solid solution. It is recalled that the gold-nickel system exhibits complete solid solubility above a miscibility gap. Preliminary anelastic and resistivity experiments have shown that the solid solution can be retained by quenching and the evolution of short-range order in the metastable phase analyzed at temperatures well below the domain of solid solubility, without any disturbances produced by the eventual advent of unmixing [21]. More recent work by Halbwachs [22] and by Mazot et al. [23] has indicated that the relaxation strength Δ follows a Curie-Weiss law, in the temperature range examined, from 310°C to 200°C. It is worth noticing that, at 200°C, the anelastic strain is more than twice the elastic one (fig. 5). By extrapolation, a critical temperature of 175 ± 10°C is inferred.

When appropriate mobility conditions are met, a remarkable behaviour of the solid solution is observed. As soon as temperature is lowered below T_C , in conditions of no applied stress, large permanent strains spontaneously occur, which are manifested by shape variations. This effect is entirely reversed by increasing the temperature above T_C [22]. It is thought that self-induced atomic rearrangements result in the formation of local domains in which the macroscopic strain would be unidirectional. Direct evidence of this ferroelasticity by electron microscopy would be of interest as well as examination of the detailed features of this transformation above T_C .



Fig. 5 (left) Reciprocal relaxation strength versus temperature in the α -Au-30 at % Ni solid solution ; determinations were by use of mechanical after-effect measurements (C) [22] and of a variable frequency torsional pendulum (+) [23].

Fig. 6 (right) Same in the vicinity of the critical order-disorder temperature. T_c , in Au₃Cu. Triangles are from prior work [24] and circles from present observation.

VI. SHORT-RANGE VERSUS MEDIUM OR LONG-RANGE ORDER

A more complex case is when, in addition to short-range ordering, longer-range ordering phenomena take place. The AuCu system is the archetype of long-range ordering alloys. In particular a number of relaxation experiments have been conducted in the disordered solid solutions (for a recent survey, see ref. [25]). We will focus on the Au₃Cu composition and on critical phenomena in the vicinity of the order-disorder transition; the critical temperature is 200° C. Recent quench and resistivity measurements have shown that the temperature variations of resistance associated with equilibrium short-range order exhibit a pronounced positive deviation from the extrapolation of the high temperature equilibrium line [28]. It is accompanied by a marked enhancement of characteristic ordering times, the so-called critical slowing down. A parallel X-ray determination of the Warren-Cowley parameters has shown that, in similar conditions, an extension of the spatial range of the local order was occurring [27] Zener relaxation measurements in our laboratory bring further arguments along this view. It is seen in Fig. 6 that the temperature dependence of the relaxation strength exhibits marked deviations from the usual Curie-Weiss law in the vicinity of the critical order-disorder temperature. Interestingly, a similar effect has been reported in Cu3Au close to the critical temperature

of 390 C [251. Thus, just like Zener relaxation is decreased or even absent in a crystal with high degree of long-range order, a high degree of local order causes a reduction in the strength of the relaxation.

VII. AMORPHOUS ALLOYS

Amorphous alloys have received much attention in the last few years. A quite interesting and important feature of them is the structural relaxation which occurs in the amorphous regime during aging at subcrystallization temperatures. This relaxation involves the reduction and redistribution of the "free volume" characteristic of the amorphous condition. It implies also modifications to the nature of the local chemical atomic coordinations. For convenience the corresponding atom rearrangements are subdivided into changes in topological short-range order (TSRO) and compositional short-range order (CSRO). We have studied these phenomena by resistivity and anelasticity methods. The most striking aspects of the results are as follows : first, quench and resistivity experiments of the type already described have enabled separation of two effects, of reversible and non-reversible character, respectively. Fig. 7 is an illustration of the resistivity pattern that was observed in a number of alloys [28]. Starting from the asamorphized condition, progressive annealing by isochronal temperature steps results in a resistivity peak. The isochronal first heating anneal sequence was stopped periodically. Then anneal and quench cycles were performed at successively decreased and increased temperatures. Also a second heating sequence was conducted. Reversible changes akin to the ones found in crystalline alloys were observed. Thus the above treatments enable separation of the chemical short-range ordering, manifested by a resistance increase, from the topological, medium-range ordering revealed by a non-reversible resistance decrease.



Fig. 7 Resistance variations, measured 4.2 K, in three specimens which were given successive 10min. isochronal anneals : (a) first heat up, (b) temperature cycles after prestabilization at 618 K. Curve c was obtained in conditions of prolonged isothermal treatments.

Fig. 8 Length variations measured during annealing at 575 K, after direct heating to this temperature.

Fig. 9 Evolution of strain, in $Ni_{35}Ti_{65}$ preannealed for about a day at 575 K and several hours at 555 K before the stress cycle was started.

Second, analysis of the reversible resistivity effect shows that ordering kinetics are of Gaussian type with an extremely wide distribution of relaxation times, characterized by

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half-width of the order of 4. Interestingly, the shape of the curves for down and up anneat cycles cannot be explained if ordering occurs homogeneously. The experimental profile is understood as showing that the material is a conglomerate of ordering domains-at an unspecified scale larger than some interatomic distances – each characterized by its own evolutionary degree of CSRO [29].

Finally, first evidence of stress-induced directional ordering in a metallic glass was brought in melt-spun Ni₃₅Ti₆₅ by realization of combined length and mechanical aftereffect experiments (in tension). The length variations associated with the structural relaxation were measured directly at the anneal temperature of 575 K. Initially a rapid contraction is observed (fig. 8). It is progressively slowed down and tends towards an asymptotic level for anneal times of the order of 10^5 seconds. This treatment was intended to stabilize the structural state of the specimen before a stress cycle was conducted, at the temperature of 555 K. A stress of about 500g mm⁻² was first applied. The instantaneous resulting strain is about 2.2 x 10^{-4} . Creep is also observed. When the stress is released, most of the strain is recovered, as appears in Fig. 9. Analysis of the kinetic features of this reversible strain effect has shown that they are very close to the ones found in similar structural conditions for the reversible resistivity effects. This indicates that the relaxing entities are the same for the two processes. In analogy to similar effects in substitutional crystalline alloys, the observed anelastic effect was traced to stress-induced directional ordering [30].

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