PHYSICAL REVIEW B VOLUME 30, NUMBER 11 1 DECEMBER 1984

Investigation of cluster growth in Al-Zn-Mg systems with analysis of time-scaling properties

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(Received 29 May 1984)

The decomposition of Al-Zn-Mg alloys is investigated by diffuse neutron scattering, and the structure function $S(\vec{q},t)$ is analyzed with respect to scaling properties. Beginning after the first minutes of decomposition, $S(\vec{q},t)$ shows time-scaling behavior within the q range investigated over a time interval where the total clustered volume increases by a factor of 5. Moreover, the present results may be described by Gibb's capillarity equation for the droplet equilibrium during the whole decomposition process; i.e., a proportionality between critical radius and mean radius of gyration is observed. Both properties are not in contradiction with the basic assumptions of the Lifshitz-Slyozov theory on the evolution of the cluster distribution function, whereas the well-known $R \propto t^{1/3}$ behavior for the asymptotic real-time evolution of the cluster growth, however, could not be found in the present alloy. The experimentally observed time dependence expressed in power laws $R \propto t^{a}$ shows exponents much lower than most theoretical estimates. In principle, this discrepancy may be removed if, within the Lifshitz-Slyozov description, a time-dependent diffusion constant is assumed.

I. INTRODUCTION

The decomposition of alloys quenched into a two-phase region has been the subject of numerous recent theoretical and experimental investigations.[1–15] The transformation of a uniform mixture of A and B atoms to well-separated A- and B-rich domains is characterized by an ordering wave with $\vec{k}=\vec{0}$. In contrast to ordering processes with finite wave vectors, i.e., order-disorder transitions, in the case of clustering, transport of matter takes place over large distances, and therefore diffusion plays a predominant role. The description of the diffusion process is a main difficulty in understanding clustering phenomena: The quenched alloy is in a two-phase nonequilibrium state, and therefore the use of the diffusion constant appearing in Fick's law, which describes small deviations from a single-phase equilibrium, may no longer be adequate. Moreover, the influence of the defect structure specific for each material and its history, e.g., vacancies, dislocations, grain boundaries, elastic distortions, etc., should be taken into account. For instance, the existence of impurity-vacancy and divacancy complexes induces nontrivial dependences of the diffusion on alloy composition.[16–18] On the other hand, the discovery of dynamical scaling properties for the late stages of decomposition revealed time-independent characteristics of the structure function, i.e., time is scaled out and the structure function remains self-similar during its evolution towards equilibrium.[2–7] The influence of diffusion is then implicitly contained in a time-dependent scaling parameter $R(t)$.

The dynamical scaling is visible in the structure function, $S(q,t)$, which can be obtained directly by diffraction experiments. This was first observed for the late stages of decomposition with computer simulations on an Ising lattice,[4] and was subsequently confirmed in various real cases (binary and ternary alloys,[11–13,19] glasses,[9] and fluids[19]), so that one may write[3–6]

$$\tilde{S}(q,t)=R^{3}(t)F(qR(t)) , \tag{1}$$

where F is a time-independent scaling function [q denotes the length of the wave vector, $q=(4\pi/\lambda)\sin\theta$]. However, in the computer simulations and some real experiments, the structure function $S(q,t)$ was normalized as[4,11]

$$\tilde{S}(q,t)=\frac{S(\vec{q},t)}{\sum\limits_{\vec{q}} q^{2}S(\vec{q},t)\delta\vec{q}} \tag{2}$$

in order to obtain relation (1). The sum was taken over the $\vec{q}$ points where appreciable intensity was observed. This leads to

$$S(q,t)=J(t)F(qR(t)) , \tag{3}$$

where $J(t)$ is defined by combining (1) and (2). It has been shown recently that, in the case of computer-simulation results, R and J/R^{3} are proportional to mean cluster radius and total volume of clusters, respectively.[19] This interpretation, verified in the case of dilute alloys, is consistent with the classical works by Guinier[20] and Gerold.[21] During the final stage of decomposition, however, where dynamical scaling is supposed to occur, the total cluster volume J/R^3 should have nearly attained its equilibrium value, and should remain constant[19] so that Eq. (3) is equivalent to (1).

The shape of scaling function $F(x)$ is found to be similar throughout different classes of substances,[19] although discrepancies have been reported recently in the case of a magnetic alloy.[12]

The interpretation of the time evolution of $R(t)$, however, is essentially dominated by diffusion effects. Although an *ab initio* formulation has been given by Binder *et al.*,[2] the resulting equations may not be solved in general, and assumptions concerning the diffusion process and growth mechanism (coagulation, coalescence, etc.) are necessary. Depending on these assumptions, various forms are derived for the asymptotic time behavior of $R(t)$ expressed generally as power laws $R(t) \propto t^a$, where a may take values between $\frac{1}{6}$ and $\frac{1}{2}$, in agreement with computer simulations.[4] General discussions were given recently by Binder[2] and Furukawa,[22] and the seminal work on the subject is the study by Lifshitz and Slyozov (LS) giving $a=\frac{1}{3}$.[23]

The exponents obtained experimentally vary considerably from one substance to another,[10–13] and even for a given alloy with a change in temperature.[11] The values of a are often comparable to the theoretical values,[12] but are sometimes also much lower than expected.[10,11]

In fact, despite the enormous experimental literature on decomposing alloys, only a few experiments have been carried out for critical comparison with theoretical calculations. The major problems are the data evaluation when only a few small clusters are present in an alloy, and also the difficulty of comparing time scales between the theoretical calculations and different experiments, because the kinetics, and therefore the relevant time scales, may vary by many orders of magnitude through a small change in temperature or alloy composition, and also from one substance to another.

We have carried out an experiment on the decomposing alloy Al-Zn-Mg, designed to follow the decomposition over the widest possible time interval. The alloy has been chosen because of the similarity to Al-Zn, which has been used widely as a model system.[24] Mg is added to control the kinetics, so that the early stages of decomposition may also be observed. As both Zn and Mg participate in the decomposition through the formation of Guinier-Preston (GP) zones,[25] Al-Zn-Mg is considered here to be a pseudobinary Al-(Zn,Mg) alloy.

The structure function has been measured using diffuse neutron scattering on quenched samples, Al—2.41 at. % Zn—1.30 at. % Mg and Al—2.36 at. % Zn—3.01 at. % Mg, annealed at different temperatures with aging times ranging from 5 min to 4.5 yr. First, the dynamical scaling behavior of the structure function is investigated and the results are analyzed with respect to the various scaling theories. An additional time-scaling relation between the mean cluster radius and the supersaturation, which may also be obtained from these data, has already been published in a letter.[26] These results are used to derive a generalization of the usual scaling equation (1) accounting for decomposition in Al-Zn-Mg over nearly the entire time interval investigated. Finally, the diffusion-controlled properties are analyzed via the time evolution of $R(t)$ at different temperatures. In particular, an analysis of the data using the Lifshitz-Slyozov concept[23] is carried out in which a time-dependent diffusion constant is needed to obtain agreement between theory and experiment.

II. EXPERIMENTAL DETAILS

The samples were two sets of platelets of polycrystalline aluminum-zinc-magnesium alloys with compositions as indicated in Table I. The platelets were homogenized for several hours at 460 °C. In order to change the number of the initially quenched-in vacancies, three different quenching procedures were applied. Some of the platelets were quenched directly in ice water (quench 1), while others were removed from the furnace, held for 10 sec in air, and then dropped into ice water (quench 2); a third group of platelets was first dropped into boiling water and after 10 sec finally quenched into ice water. The neutron-scattering measurements were done at liquid-nitrogen temperature on the D7 spectrometer for diffuse scattering located at a cold-neutron-guide position of the Institut Laue-Langevin, Grenoble. An incident-neutron wave length of 4.9 Å was used, and a q range from 0.25 to 1.5 Å^{-1} was investigated. The diffuse neutron scattering was measured as a function of annealing time for three temperatures: 25 °C, 50 °C, and 70 °C.

For comparison, some additional measurements were carried out, using a conventional x-ray small-angle-scattering spectrometer with Cu K_α radiation ($\lambda=1.54$ Å), on samples of Al-Zn-Mg of longer annealing times at room temperature, showing no significant difference in the scaled structure function, as well as in the R-versus-t behavior, when compared to the neutron-scattering measurement, so that the pseudobinary approximation Al-(Zn,Mg) seems reasonable.

III. DYNAMICAL SCALING

Diffuse scattering of all samples was measured between 0.25 and 1.5 Å^{-1}, and showed after background correction, in all cases, a flat portion in the q region around 1 Å^{-1}. This was interpreted as being the sum of incoherent and Laue monotonic scattering, and this constant was subtracted from the scattered intensity. The intensity curves were then normalized for sample volume, and, as described in Ref. 19, double-logarithmic plots of $S(q)$ versus q of the so-obtained structure functions $S(q)$ were

TABLE I. Composition of investigated Al alloys.

	Alloy I composition		Alloy II composition	
	(wt. %)	(at. %)	(wt. %)	(at. %)
Zn	5.65	2.41	5.55	2.36
Mg	1.13	1.30	2.63	3.01
Si	0.008		0.008	
Cu	0.006		0.006	
Fe	0.003		0.002	

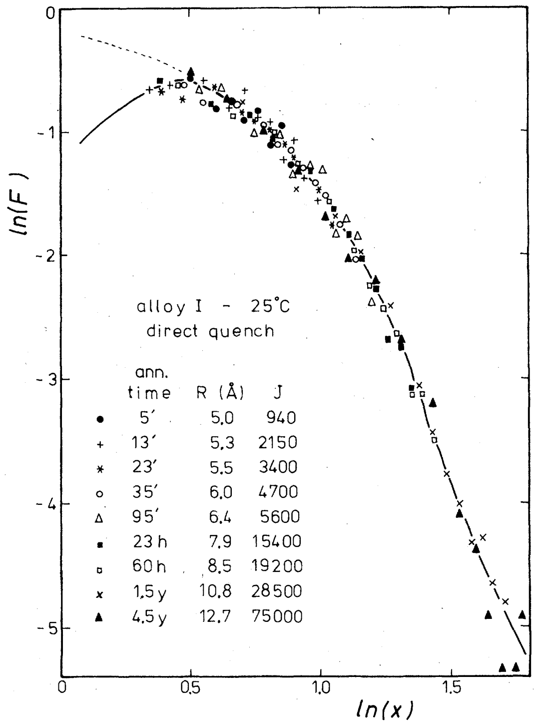

FIG. 1. Double-logarithmic plot of all measured scattering curves $S(q,t)$ in directly quenched (quench 1) Al—2.4 at.% Zn—1.3 at.% Mg (alloy I) aged between 5 min and 4.5 yr at room temperature. Scales are reduced, with the values of J and R indicated in the figure so that $F(x)=S(q,t)/J(t)$, $x=qR(t)$ [Eq. (3)]. The solid line is the fitted scaling curve, which is in very good agreement with computer-simulation results (Refs. 4 and 19). The dashed line represents the Gaussian approximation at small wave vectors.

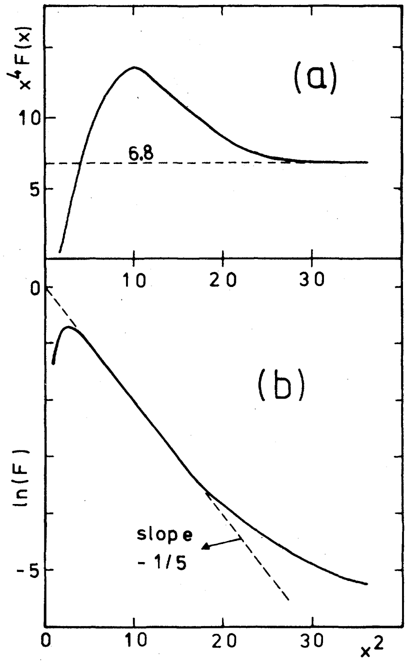

FIG. 2. Scaling function $F(x)$ (solid line in Fig. 1) in a (a) Porod and (b) Guinier plot. The domain of validity of both approximations, i.e., $F(x) \propto 1/x^4$ and $F(x)\approx e^{-x^2/5}$, respectively, may be seen.

made. In order to test the data for dynamical scaling behavior, an attempt was made to superpose the structure functions by shifting the logarithmic scales. This was indeed possible for all measurements, except for the as-quenched samples, which showed, in the q range investigated, a nearly flat intensity. Following Ref. 19, we obtained by this method a scaling description of the structure function as given by Eq. (3). The time-independent scaling function F is normalized[19] so that R may be identified with the classical Guinier radius[20] obtained by fitting $S(q,t)$ to a Gaussian,

$$S(q,t)\approx J(t)\exp\left[-\frac{R^2(t)}{5}q^2\right], \qquad (4)$$

in the appropriate q range. The Guinier radius, which is a mean radius of gyration, is related to the mean radius by a constant factor (~ 1.3), as shown in the case of computer simulations.[19] $F(x)$ also shows $1/x^4$ decay at high x values, in agreement with the Porod law[27] and calculations by Furukawa.[3] The scaling function $F(x)$ obtained by the superposition method is in excellent agreement with the scaling function obtained by computer simulations in the case of an A—5 at.% B alloy at $T/T_c=0.59$.[4] This is seen in Fig. 1, where data for the alloy I annealed at room temperature between 5 min and 4.5 yr are shown, together with the scaling function from Ref. 19. The domains of validity of the Porod and the Guinier laws [Eq. (4)] are shown in Fig. 2. The present experiment confirms dynamical scaling in Al-Zn-Mg for both alloys studied, three temperatures, and different quenching procedures from the maximum of the structure function until high q, including the regions of the Guinier- and Porod-law behavior. No data could be obtained, however, in the very-small-q region characterizing

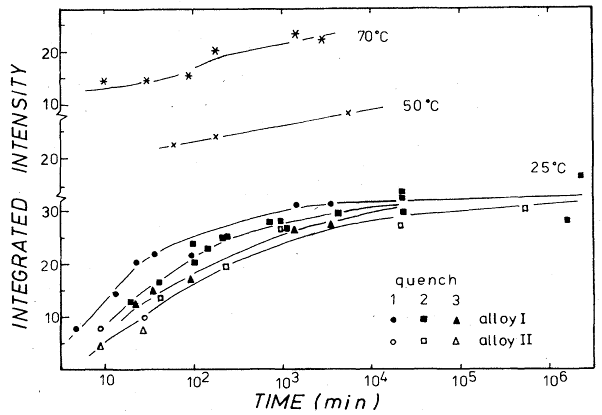

FIG. 3. Integrated intensity J/R^3 versus logarithm of time for all measurements, i.e., at 25 °C for both alloys and three quenching procedures, and at 50 °C and 70 °C for alloy I. An additional normalization to the same total content in Zn and Mg has been performed in order to permit a comparison of both alloys.

the cluster interference.[20] Moreover, in contrast to the usual scaling concept,[2–6] scaling was observed in the present experiment even at early times of decomposition and over a time interval where the total volume of clusters, V, first increases by more than a factor of 5, reaching a nearly constant value after a few weeks (see Fig. 3), whereas dynamical scaling formulation has generally been considered to hold for the asymptotic time-evolution period where V is already close to its final value.[2–6]

IV. THE CAPILLARITY EQUATION

Once the Laue scattering has been subtracted from $S(q,t)$ the total cluster volume $V(t)$ is, for low supersaturation,[21] proportional to

$$\int_0^\infty S(q,t)q^2dq = J(t)/R^3(t)\int_0^\infty F(x)x^2dx$$

[Eq. (3)], and therefore to $J(t)/R^3(t)$. Since $V(t)$ varies throughout the decomposition process, it provides an intrinsic parameter for the advancement of the phase separation. It therefore may provide an inner time scale of the system. In a recent paper,[26] referred to as I, it has been shown that R and V verify, for Al-Zn-Mg, a relation independent of the quenching procedures, even though different time evolutions of the cluster radius R were observed. For both alloys and all temperatures, it was shown in I that

$$\frac{R_0}{R(t)} = 1 - \frac{V(t)}{V_\infty}\,, \tag{5}$$

where R_0 and V_∞ are temperature- and alloy-composition-dependent constants. A discussion of this R-versus-V curve has shown that at the beginning V increases with nearly constant R, which may indicate predominant nucleation. At the end, however, V reaches a constant value, whereas R still increases, indicating the presence of a coarsening regime. This leads to an interpretation of R_0 as a critical radius at the beginning and V_∞ as an equilibrium volume fraction at the end of the decomposition. Furthermore, it was shown in I that in the case of low supersaturation, which is a good approximation here, Eq. (5) may be transformed, using the lever rule, to

$$R(t) = R_0\frac{\delta c_0}{\delta c(t)}\,, \tag{6}$$

where δc is the supersaturation and δc_0 its initial value. This is basically Gibb's capillarity equation, defining the equilibrium of a droplet in a surrounding phase due to its surface tension, which, however, is written here for the mean cluster radius.

The results of I allow one to derive an extension of the classical scaling equation (1) for the structure function. Replacing $J(t)$ by $V(t)R^3(t)$ in Eq. (3), and using (5), one obtains

$$\frac{1}{V_\infty}S(q,t) = R^3(t)\left[1 - \frac{R_0}{R(t)}\right]F(qR(t))\,. \tag{7}$$

In the limit of large R, where classical scaling should apply,[2–6] (7) reduces again to the usual scaling equation (1). With this correction of (1), it is then possible to describe, within the q range shown in Fig. 1, the decomposition of Al-Zn-Mg over a wide time interval, and even at the beginning of cluster growth, when $R(t)$ starts with small values close to R_0.

V. TIME EVOLUTION OF THE MEAN CLUSTER SIZE

The two time-invariant results of the preceding sections may be related to basic results of the Lifshitz-Slyozov theory of coarsening.[23] The capillarity equation (6) found in this experiment is in accordance with the equality of the mean and critical radius, which is a main result of the LS theory.[23] The time-scaling behavior of the LS cluster distribution function would also produce a scaling of the structure function. The time evolution, however, of the cluster size within the LS assumptions, is expressed as

$$\frac{d}{dt}(R^3) = \tfrac{4}{9}DR_0\delta c_0\,, \tag{8}$$

where D is the diffusion constant and R_0 and δc_0 are defined in Eq. (6). With D constant, an integration of (8) leads to the well-known $R(t) \propto t^{1/3}$ behavior. The experimentally observed values of R are plotted versus $t^{1/3}$ in Fig. 4 for both alloys and all temperatures investigated. However, no straight line is observed, even for the very late times of decomposition, so that no $t^{1/3}$ behavior is found in this experiment.

We then tried a description of the time evolution of R as a power law in the form

$$R(t) = (\lambda t)^a\,. \tag{9}$$

For alloy I and three temperatures, R has been plotted versus time on double-logarithmic scales in Fig. 5. When straight lines are fitted to the data, the same exponent, $a \approx 0.08$, is obtained for all three temperatures. The values of the temperature-dependent constant λ are indicated in the figure.

The classical theoretical $t^{1/3}$ behavior was derived by LS theory, assuming bulk diffusion. Within the same framework, but assuming diffusion along grain boundaries or dislocation pipes, exponents of $\frac{1}{4}$ and $\frac{1}{5}$, respectively, were later obtained.[28,29] More recent treatments of cluster growth during decomposition led to exponents be-

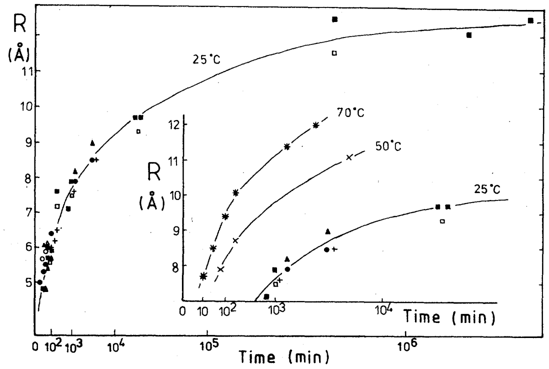

FIG. 4. Mean radius versus $t^{1/3}$ for all measurements. The symbols are defined as in Fig. 3.

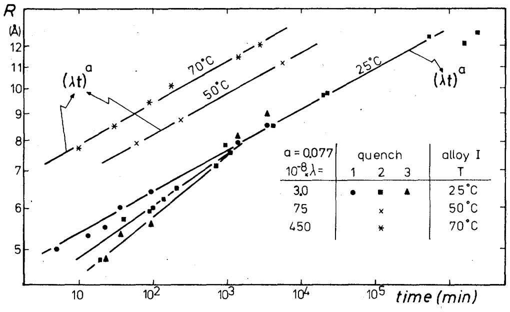

FIG. 5. Double-logarithmic plots of the mean radius versus time for alloy I. The later times have been fitted with Eq. (9).

tween $\frac{1}{6}$ and $\frac{1}{2}$.[2–4,22] The value of a obtained in the present experiment is therefore much lower than any existing theoretical estimate.

It may be argued that one does not yet observe the asymptotic time behavior and that there is still strong nucleation of clusters. In the present case, however, the total cluster volume (Fig. 3) does not increase significantly between 1 and 4 yr of aging at room temperature, so that strong nucleation seems very improbable at this stage.

As discussed in the case of highly degenerate systems,[14,15] a slow increase of $R(t)$ (i.e., a logarithmic behavior) may arise from the fact that the driving force for coarsening (i.e., the surface tension) becomes ineffective. However, the finding that the capillarity equation (6) holds during the entire decomposition process of Al-Zn-Mg does not support such a hypothesis in the present case.

On the other hand, the only discrepancy between the present experiment and the LS theory may be removed and the experimentally observed time dependence obtained, if an "effective diffusion constant" D_{LS} is introduced which is allowed to vary during decomposition. Within this formulation, Eq. (8) defines D_{LS}, which is then computed with the present data and plotted in Fig. 6 versus R. The numerical values for R_0 and δc_0 have been taken from paper I and are listed in Table II. In Fig. 6 the logarithmic scales for D_{LS} have been shifted by an amount Q/k_BT, where Q has been taken to be 1.0 eV. By this procedure, the data for the three temperatures fall on the same curve so that D_{LS} appears as

$$D_{\mathrm{LS}}(R,T)=g(R)e^{-Q/k_BT}, \tag{10}$$

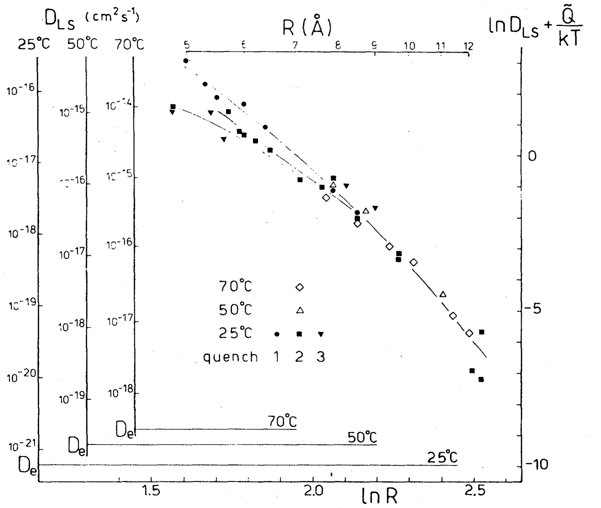

FIG. 6. Using the R-versus-t curves (Fig. 5), D_{LS} is computed from R using Eq. (8) (see text). The constants in Eq. (8) are taken from Table II. The logarithmic scales for D_{LS} have been shifted by Q/k_BT, where $Q=1.0$ eV (right-hand-side scale), leading to a superposition of data at greater R for all three temperatures for alloy I. The values, D_e, of the diffusion constant extrapolated from high temperature to 25 °C, 50 °C, and 70 °C are also indicated.

where g is the function of R represented in Fig. 6. It should be noted that the diffusion D_e at high temperatures in the same alloy has an activation energy of 1.2 eV,[17] whereas $Q=1.0$ eV. D_e has been extrapolated into the two-phase region and is indicated in Fig. 6. D_{LS} is higher than D_e by several orders of magnitude and decreases strongly, when the clusters are growing, and, even after very long annealing times, D_{LS} is still higher than D_e.

A result concerning a similar large discrepancy of the diffusion constant in the miscibility gap when compared to the values extrapolated from the single-phase region has been found recently in Al-Zn. For an Al-Zn alloy in which a $t^{1/3}$ behavior has indeed been reported, the obtained constant D_{LS} was higher than D_e by a factor of 10^4, and it was not possible to explain this difference by the amount of quenched-in vacancies.[13]

TABLE II. Critical radius R_0 and the final volume fraction V_∞ (within a multiplicative constant) of clusters, as determined in paper I (Ref. 26), as well as the initial supersaturation δc_0 and the solubility c_α of Zn and Mg estimated from V_∞ (Ref. 30).

Alloy	Composition (at. %)	Temperature (°C)	R_0 (Å)	V_∞ (a.u.)	c_α (at. %)	δc_0 (at. %)	$\frac{4}{9}R_0\delta c_0$ (Å)
II	5.4	25	5.1	98	1.6	3.8	0.086
I	3.7	25	3.8	54	1.6	2.1	0.035
I	3.7	50	4.3	47	1.9	1.8	0.035
I	3.7	70	5.2	40	2.1	1.6	0.036

VI. CONCLUSION

The decomposition kinetics of some Al-Zn-Mg alloys has been investigated by means of diffuse neutron scattering, and the time-scaling results are obtained and discussed in terms of the Lifshitz-Slyozov theory of coarsening.[23] This theory starts with a time-scaling assumption of the cluster distribution[23] describing a situation where the system remains self-similar between two steps in the time evolution, when simultaneously the length scale is changed (such scaling assumptions are also used in more recent theories.[2,3] Using the classical diffusion equation, LS then derive, for the late stages of decomposition, the equality between mean and critical radius, as well as a time behavior of the mean radius as $t^{1/3}$. In the present experiment the structure function showing dynamical scaling behavior also remains self-similar during the decomposition. Moreover, the time-scaling relation between mean radius and supersaturation [Eq. (6)] was found during all stages of cluster growth, which can be related to the equality between mean and critical radius in the LS theory. However, the most well-known result of the LS theory, i.e., the time behavior of the mean cluster radius $R(t)$ as $t^{1/3}$, has not been observed. The time behavior of R fitted as a power law t^a yields an exponent a smaller than any theoretical estimate.[2–6,22,23] It should be pointed out that these LS relations, which are indeed verified in Al-Zn-Mg, are time-scaling relations, so that, in fact, diffusion has been scaled out, whereas the actual time evolution of $R(t)$ is a diffusion-controlled property.[31]

An estimate of the diffusion constant just after quenching using Eq. (8) [derived by LS (Ref. 23)] gave a value higher than the value extrapolated from high-temperature measurements by a factor 10^4-10^5. This is observed even when the LS theory is applied to alloys where $R(t)$ behaves approximately as $t^{1/3}$, as reported by Simon *et al.*[13] in Al-Zn and Al-Zn-Ag. Finally, the remaining discrepancy between LS theory and the time evolution of $R(t)$ in Al-Zn-Mg may be removed if an effective diffusion constant is introduced, which, after the quench, is much higher than the value extrapolated from high temperature and decreases strongly during decomposition.

ACKNOWLEDGMENT

The authors would like to thank the Fonds zur Förderung der wissenschaftlichen Forschung in Österreich for financial support.

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[30] V_∞ is affected by an unknown multiplicative constant due to the experimental setup, but knowing that $\delta c_0 \propto V_\infty$ and $\delta c_0 = \bar{c} - c_\alpha$ ($\bar{c}$ is the mean concentration of Zn plus Mg in the sample), the values of c_α and δc_0 may be crudely estimated, supposing that c_α depends only on temperature (and is the same for both alloys at 25 °C). The values obtained for c_α are good agreement with other measurements [cf. V. Gerold, J. E. Epperson, and G. Kostorz, J. Appl. Crystallogr. **10**, 28 (1977)].

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