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**KINETICS OF DIFFUSIVE GROWTH OF
CdS CRYSTALS IN GLASSY MATRIX**

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Abstract

A method of microscopic description was applied to the study of time dependence of the cluster size distribution in CdS doped GeO₂ glassy matrix. It was assumed that clusters were grown by the condensation flux of CdS particles at the cluster surface and neglecting the evaporation flux. The rate equation for the number of CdS particles and clusters were found to obey a complex integro-differential equation, which with the law of matter conservation, describe the growth process. Evaluation of first order approximation and the experimentally determined time dependence of the average radius, dispersion and volume were found to obey the $t^{1/3}$, $t^{2/3}$ and t laws, respectively.

KEYWORDS: Cds, clusters, growth, diffusion, glasses.

PACS — 90: 61.50Cj

I. INTRODUCTION

CdS and CdSe clusters in glassy matrices have become one of the most interesting topics in the study of low-dimensional materials, because they are not so difficult to obtain it, and have potential applications in non linear optics¹⁻⁷. A large number of reports on the optical properties of CdS and CdSe clusters have been appeared⁸⁻¹⁷. The size variation of the clusters in heat treatment have been analysed using the theoretical model of coarsening, developed almost 30 years ago¹⁸⁻³³. This theory successfully predicts coarsening kinetics and optical properties qualitatively, but not the quantitative determination of parameters such as effective diffusion coefficient and interfacial energy. A number of modifications of the basic theory, taking into account several important factors affecting coarsening kinetics, have been proposed but none of these has found general acceptance and none takes account all the factors which can affect coarsening³⁴.

It was pointed out that two stages must be distinguished in the cluster growth process in a supersaturated solution. In the first stage, occurs the formation of the fluctuating nucleation centers and their growth is directly out of the supersaturated solution. In the second stage, the clusters are fairly large and the degree of supersaturation becomes extremely small. The main process is the coalescence, in which larger microcrystals grow by dissolution of smaller ones.

The exact asymptotic solution of the problem, derived by Lifshitz and Slyozov (LS), shows that the cluster average volume grows as t and the total number of clusters decreases as t^{-1} power law. Clusters larger than a critical radius $|R_{CR}|$ are unstable with respect to the degree of supersaturation and they growth. Clusters smaller than R_{CR} , are also unstable, and dissolve back into the matrix. The precipitation rate dominates and the degree of supersaturation decreases, leading to a time-dependent increase of R_{CR} . Thus, as the process progresses, increasingly larger particles have radii below R_{CR} , causing

then a shrinking, which moves the average radius of stable clusters to higher values and decreasing the number of clusters per unit volume¹⁷.

The size distribution of CdS clusters¹⁶⁻¹⁷ shows the characteristic vanishing of the distribution above certain values of cluster radius, predicted by the LS model and the characteristic abrupt increasing to a maximum, near 2/3 of the maximum radius. However, the size distribution is slightly broader and shows no continuous tail extending to vanishing radius. The measurements show a well-defined size cut off at R_m , below which no clusters have been found. The R_m values were not the same for all measurements and actually grows with heat treatments time as the $t^{1/3}$ power law¹⁷. These observations show a strong evidence that the clusters growth are not a pure coarsening process.

The objective of this work is the study of the growth process in which the coarsening rate is overcome by the precipitation rate, stabilizing all clusters with respect to dissolution back into the matrix. The aim is to obtain a better understanding of the growth process in glassy matrix.

II. A MODEL FOR CONDENSATION CURRENT

Let us now set up a microscopic description of the growth of CdS clusters. As the number of clusters is constant, we assume that the nucleation of new clusters have ceased and that the precipitation rate dominates, stabilizing all clusters with respect to dissolution back into the matrix. Then, the consistency of this description will be checked through the evaluation of the asymptotic time dependence of average R^3 and comparing it with the experimental data.

To simplify the problem, the anisotropy was neglected and the spherical shape for clusters and particles is assumed. The diffusion flux of the dissolved substance per unit of cluster surface is composed by a condensation flux and an evaporation flux, which is

neglected because precipitation rate dominates.

In the condensation, which occurs at the saturated condition, we can determine the transport flux of the dissolved substance per unit of cluster surface, following the originally applied theory of the colloidal flocculation.

The clusters are formed from the condensation of CdS particles overcoming a potential barrier. This potential is probably produced by the local geometrical relaxation, of particles needed for the condensation of new substances. Here, we consider the potential U , which includes the potential barrier. A sketch of U is shown in Figure 1.

INSERT FIGURE 1

The density of current in the direction of clusters having potential U is

$$\vec{J} = -\vec{\nabla}(D\bar{n}) - \frac{D\bar{n}}{kT} \vec{\nabla}U \quad , \quad (1)$$

where \bar{n} is the density of particles which is dependent on time and on the distance from the condensation surface, T is the absolute temperature and D is the diffusion constant.

For spherical symmetry, the density of current has only radial component:

$$J = - \left[\frac{\partial(D\bar{n})}{\partial r} + \frac{D\bar{n}\partial U}{kT \partial r} \right] \quad . \quad (2)$$

Here, evaporation flux is neglected.

The current of substance is then given by $I = -4\pi r^2 J$, which from equation (2) one obtains:

$$\frac{I \exp(U/kT)}{r^2} = -4\pi \frac{\partial}{\partial r} (D\bar{n} \exp(U/kT)) \quad , \quad (3)$$

Assuming that I is independent of r , from equation (3) one obtains:

$$I(R, t) = -4\pi D_0 f(R) n(t) \quad , \quad (4)$$

where $f(R) = R^2\eta(R)$, $\eta(R) = \left[R^2 \int_R^\infty \frac{\exp(U/kT)}{r^2} dr \right]^{-1}$, with $\bar{n}(R) = 0$, $n = \lim_{r \rightarrow \infty} \bar{n}$, $\lim_{r \rightarrow \infty} U(r) = 0$, $D_0 = \lim_{r \rightarrow \infty} D$. The term $\eta(R)$ is the reflection factor for the process.

Here we assume that the formation of nuclei have ceased, i.e., the nuclei reached a critical size from which the nucleation rate is negligible. Also, we assume that the nucleation process is a very fast process. The actual density of particles will be assumed to be equal to $n(t)$ and the density of particles at the beginning of the second stage will be n_0 , and nearly equal to the initial density of particles at the beginning of the first stage. Finally, we disregard the fusion process of two or more clusters, since doping concentration is small and the observed clusters are fairly well separated.

Considering that the clusters are distributed along several sizes, the variation $n(t)$ is given by:

$$\frac{dn(t)}{dt} = -4\pi D_0 n(t) \sum_{k=m}^{\infty} f(x_k) \rho_k, \quad (5)$$

from equation (4), where ρ_k is the density of number of clusters of size R_k , $R_k = ak^{1/3}$ is the radius of the cluster with k atoms of radius a and m is the critical minimum number of clusters. Then the critical minimum radius is $R_m = am^{1/3}$.

The current over each cluster of radius R_k , is $I(R_k, t) = -4\pi D_0 f(R_k) n(t)$, from equation (6). The current over all clusters of radius R_k is $I(R_k, t) \rho_k$. The number of cluster of radius R_k decreases through the radius increasing process by absorption of particles by $-I(R_k, t) \rho_k \delta t$. The number of clusters with radius k increases through the radius increasing process from the clusters of radius R_{k-1} by absorption of particles by $I(R_{k-1}, t) \rho_{k-1} \delta t$. The variation of the clusters of radius R_k will be $-[I(R_k, t) \rho_k - I(R_{k-1}, t) \rho_{k-1}] \delta t$. Then the time variation of ρ_k is given by:

$$\frac{\partial \rho_k}{\partial t} = -[I(R_k, t) \rho_k - I(R_{k-1}, t) \rho_{k-1}], \quad (6)$$

for $k > m$. As in the present case $\gg 1$, one obtains:

$$R_k - R_{k-1} \cong \frac{a^3}{3R_k^2} \quad (7)$$

From (5), (6) and (7) one obtains:

$$\frac{dn}{dt} = -4\pi D_0 n \sum_{k=m}^{\infty} f(R_k) N_k (R_k - R_{k-1}), \quad (8.a)$$

$$\frac{\partial N_k}{\partial t} = -\frac{a^3}{3(R_k - R_{k-1})} \left[\frac{I(R_k, t) N_k}{R_k^2} - \frac{I(R_{k-1}, t) N_{k-1}}{R_{k-1}^2} \right], \quad (8.b)$$

where $N_k = \frac{3R_k^2 \rho_k}{a^3}$ is the number of clusters with k atoms per radius.

In the limit of very small values for the relation $(R_k - R_{k-1})$ (large clusters), the equations (8) becomes:

$$\frac{dn}{dt} = -4\pi D_0 n(t) \int_{R_m}^{\infty} f(R) N(R, t) dR, \quad (9.a)$$

$$\frac{\partial N(R, t)}{\partial t} = -\frac{4\pi a^3}{3} D_0 n(t) \frac{\partial}{\partial R} \left[\frac{f(R)}{R^2} N(R, t) \right], \quad (9.b)$$

where $N(R, t) dR$ is the number of clusters of size between R and $R + dR$. The limit of $N(R, t)$ for large values of R must vanish because fusion process of two or more clusters is disregarded in the present description.

III. PARTICLE CONSERVATION

The microscopic description of the CdS clusters growth using equations (9) is open with respect to the total number of particles. The glassy matrix doped with CdS, conserve the number of particles. Then, a conservation condition must be found to complete the desired description.

The number of atoms in each cluster of radius R is given by $(4\pi R^3/3)/(4\pi a^3/3)$. The total density of clusters of radius R is equal to the number of particles times the density of clusters of size R , i.e., $(R/a)^3 N(R, t) dR$. Then the total numbers of particles, Φ , at any instant of the process is given by:

$$\Phi(t) = n(t) + \int_{R_m}^{\infty} \left(\frac{R}{a}\right)^3 N(R, t) dR \quad (10)$$

The time derivative of this number must vanish. Then, calculating the derivative of equation (10) and using equation (9.b), one obtains:

$$\frac{d\Phi}{dt} = \frac{dn}{dt} - \frac{4\pi}{3} D_0 n \int_{R_m}^{\infty} R^3 \frac{\partial}{\partial R} \left[\frac{f(R)}{R^2} N(R, t) \right] dR \quad (11)$$

Integrating by parts equation (11) one obtains:

$$\frac{d\Phi}{dt} = \frac{dn}{dt} + 4\pi D_0 n \int_{R_m}^{\infty} f(R) N(R, t) dR - \frac{4\pi}{3} D_0 n [Rf(R)N(R, t)]_{R_m}^{\infty} \quad (12)$$

From equation (9.a), and (10), applying the particle conservation $\frac{d\Phi}{dt} = 0$ and regarding that equation (5) has been set by assuming a critical minimum radius ($N(R_m, t) = 0$) we obtain the condition given by:

$$\lim_{R \rightarrow \infty} Rf(R) N(R, t) = 0 \quad (13)$$

The analysis of the potential shown in figure 1 shows that $Rf(R)$ increases as R^4 . Assuming a square potential barrier of width ΔR and height V_0 , we obtain the limit given by:

$$\lim_{R \rightarrow \infty} Rf(R) N(R, t) = \lim_{R \rightarrow \infty} R^4 N(R, t) / \Delta R \exp(V_0/kt) \quad (14)$$

The distribution $N(R, t)$ does not vanishes for larger R . Therefore equation (14) leads to the condition that precipitation in big clusters are innhibited by the increasing of the potential barrier V_0 .

The size distribution of clusters predicted by the LS model is given by:

$$N(u) = \begin{cases} \frac{A u^2 \exp[-1(1-2u/3)]}{(u+3)^{7/3}(3/2-u)^{11/3}} & , u < 1.5 \\ 0 & , u \geq 1.5 \end{cases} \quad (15)$$

where $u = R/\langle R \rangle$, $\langle R \rangle$ is the average value for R . The Cds cluster distribution is very similar to this distribution except that there is no continuous tail extending to vanishing radius.¹⁶⁻¹⁷ The continuous tail of equation (15) can be eliminated by replacing the factor u^2 by $(u - u_m)^2$ and assuming $P(u) = 0$ for $u < u_m$, with $u = R_m/\langle R \rangle$. Here u_m describes the critical minimum radius defined when equation (5) was obtained.

Another, important observation in the previous reports is that $N(R, t)$ can be scaled for time and $\langle R \rangle$. Therefore, we assume that $N(R, t)$ is of the following form:

$$N(R, t) = N(u) N(t) \quad (16)$$

where $N(u)$ has an implicit dependence on time through $u(t)$ and $N(t)$ has an explicit dependence on time.

The total number of clusters in the present model is given by:

$$N(t) = \int_{R_m}^{\infty} N(R, t) dR \quad (17)$$

The time variation of $N(t)$, using equation (11b) is:

$$\frac{dN(t)}{dt} = \frac{4\pi a^3}{3} D_0 n \left[\frac{f(R)}{R^2} N(R, t) \right]_{R_m}^{\infty} \quad (18)$$

Equation (18) vanishes, since $N(R_m, t) = 0$ and the limit for $R \rightarrow \infty$ also vanishes from equation (13). Therefore $N(t) = N_0$, a constant, which is consistent with that is expected for systems where precipitation rate dominates.

IV. APPROXIMATE SOLUTION

Let us now analyse the time dependence of the average value of R^3 , through a first order approximation to the solution of equations (9).

The moments of clusters distributions are given by:

$$\langle R^e \rangle = \frac{\int_0^\infty R^e N(R, t) dR}{\int_0^\infty N(R, t) dR} \quad (19)$$

From equations (10) and (19) one obtains:

$$\langle R^3 \rangle_t = \langle R^3 \rangle_0 + \frac{a^3}{N_0} [n_0 - n(t)] \quad (20)$$

The density of particles $n(t)$ varies slowly because the degree of glassy matrix supersaturation is small (only small doping amount of CdS particles was introduced) and the diffusion of these particles through the glass network is a slow process. From equation (9.a) and (20) assuming a first order approximation for $n(t)$ one obtains:

$$\langle R^3 \rangle_t = \alpha t \quad (21)$$

where $\alpha = 4\pi D_0 n_0 a^3 \int_{R_m}^\infty f(R) N(R, 0) dR / N_0$.

Since $N(R, t) = N(u) N(t)$, equation (19) is given by:

$$\langle R^e \rangle = \frac{\langle R \rangle^e}{N_0} \int_{u_m}^\infty u^e N(u) du \quad (22)$$

From equations (21) and (22), the average value for R and the dispersion $\langle (\Delta R)^2 \rangle = \langle R^2 \rangle - \langle R \rangle^2$ are given by:

$$\langle R \rangle = \beta t^{1/3} \quad (23.a)$$

$$\langle (\Delta R)^2 \rangle = \gamma t^{2/3} \quad (23.b)$$

where β and γ are parameters.

V. EXPERIMENT AND RESULTS

The samples were prepared in two steps. First, base glass doped with Cd and S atoms/ions was produced by the melting method. Second, the base glasses were annealed. A mixture of GeO_2 (87.7 mol%), NaGeO_3 (8.82 mol%), Cd (0.9 mol %) and S (2.59 mol%) powders was sealed in an evacuated double-covered quartz ampoule (1×10^{-6} Torr) to avoid the danger of an accidental explosion during the cooling process. Then, it was heated up to 600°C for 12 hours while being rocked, and heated up to 1150°C for 8 hours to form CdS particles by the chemical reaction between Cd and S atoms. After, it was quenched by cooling in air at room temperature. The electron microscope image of the base glass obtained shows no CdS cluster with size larger than 4 nm. The resulting ingot was sliced with a conventional wire saw. Each slice was annealed for various times at 600°C and for 60 min at 500°C , in air.

The micrographs of slices annealed for 30 min at 550°C and 650°C have been also reexamined⁴.

The micrographs have shown that the CdS clusters have the wurtzite structure and are fairly well separated.

The size of the clusters was measured along their diameter. Table 1 shows the number of clusters, N_0 , the mean radius, $\langle R \rangle$, and the average volume per $(3/4\pi)\langle R^3 \rangle$, obtained from the micrographs. We observe that N_0 remains almost constant, showing that the above developed description can be used for the analysis of these data.

INSERT TABLE 1

The measured samples were annealed at 500°C by 60 min, 550°C by 30 min, 600°C by 10, 30 and 60 min and 650°C by 30 min.

Figure 2 shows that the LS model (dots) gives a good description of the size distribu-

tion, with excess of clusters at small radius.

INSERT FIGURE 2

This excess can be suppressed if it assumes a minimum size, R_m , below of which no cluster was found¹⁷. Accordingly, the LS distribution is modified by replacing the u^2 factor by $(u - u_m)^2$ and with $P(u) = 0$ for $u < u_m$. Then, the modified LS distribution is given by:

$$P(u) = \begin{cases} A \frac{(u - u_m)^2 \exp[-1/(1 - 2u/3)]}{(u + 3)^{7/3} (3/2 - u)^{11/3}} & , \quad u_m < u < 1.5 \\ 0 & , \quad u \geq 1.5 \text{ and } u \leq u_m \end{cases} \quad (24)$$

where $u = R/\langle R \rangle$ and A a constant. A good adherence with the data was obtained for $A = 88$ and $u_m = 0.3$ (solid line).

The histograms of samples annealed at 550° and 650°C by 30 min in Figure 3, clearly show no continuous tail extending to $u = 0$. They show a well defined size cut off, below of which no clusters is measured.

INSERT FIGURE 3

In table 1 we show the scaled time, t_s , defined to allow the comparison between the data obtained at different temperatures. The scaling was performed by assuming the $t^{1/3}$ power law observed for the CdS clusters¹⁶⁻¹⁷. Figures 4 and 5 show an excellent overall agreement between data and LS model.

INSERT FIGURES 4 AND 5

The data given in table 1 show that, at 600°C, the number of clusters remain almost constant with the annealing time. This observation is quite different from the t^{-1} power law predicted by the LS model.

Although the LS model predicts t^3 power law for the average radius data, the existence of a minimum cluster size and the constant number of clusters through different annealing times, shows that growth of CdS clusters does not follow a pure coarsening process.

VI. CONCLUSIONS

The growth process of clusters in glassy matrix has been analysed assuming that the coarsening rate is overcome by the precipitation rate, stabilizing all clusters with respect to dissolution back into the matrix.

The growth dynamics is described by the coupled differential equations (9.a) and (9.b). Fls. the exact solution is very complex, and beyond the scope of the present report, a first order approximation solution has been given showing that the mean values of radius, dispersion and volume follow $t^{1/3}$, $t^{2/3}$ and t laws, respectively.

Size distribution have been measured for CdS clusters grown in GeO₂ glassy matrix, for several temperatures and heating times. It has been observed that the number of clusters remains almost constant, which satisfies the condition that the precipitation rate dominates the growth process.

The mean average radius, dispersion and volume have been evaluated from the histograms and showed good adherence for $t^{1/3}$, $t^{2/3}$ and t laws, respectively.

The size distribution has been found to be similar to that predicted by the LS model. A modification of this distribution suppressing the continuous tail extending to vanishing radius show good adherence to the data, reinforcing the previous reports.^{16,17} As a matter of fact, the absence of the continuous tail extending to vanishing radius, has been used as the remark for the present given cluster growth modelling.

The empirical size distribution given by equation (15), together with the observed time and $\langle R \rangle$ scaling of this distribution, show a good adherence with the assumption (16)

used in the development of the first order approximation $t^{1/2}$, $t^{2/3}$ and t laws for mean radius, dispersion and volume, respectively.

The good adherence between the theoretical modelling and the experimental observations suggest that it is profitable to exert efforts to solve equations (9). The evaluation process requires the knowledge of the cluster distribution of sizes $N(R, t)$. Then, an analysis of the first stage of growth must be given to obtain $N(R, t)$.

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FIGURE CAPTIONS

Figure 1. Sketch of the potential energy for spherically assumed CdS particles in the CdS cluster.

Figure 2. Comparison of the LS (dots) and modified LS (solid line) distribution function with the experimental histograms generated from CdS containing samples annealed for 60 min at 500°C, 30 min at 550°C, 10 min, 30 min and 60 min at 600°C and 30 min at 650°C.

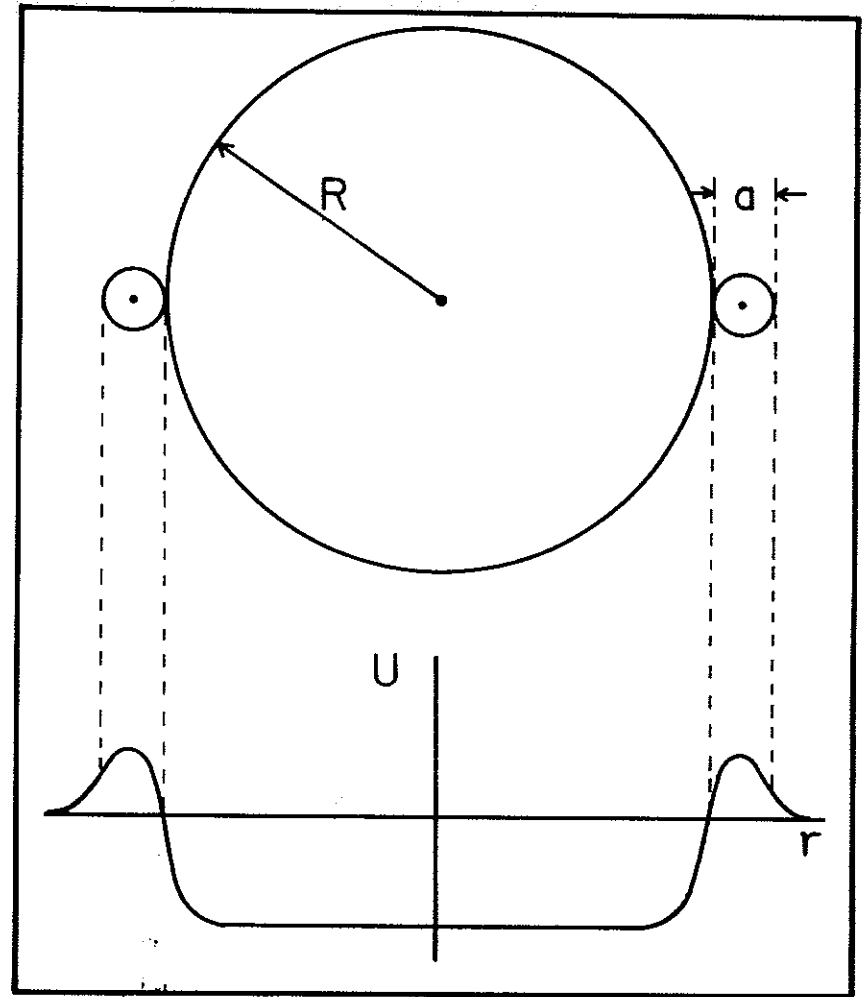
Figure 3. Histogram of size distribution in samples which are annealed at 550 and 650°C by 30 min. Dots are calculated with the LS distribution and solid line with the modified LS distribution.

Figure 4. The average R^3 behavior is shown as the t power law .

Figure 5. The average square radius dispersion behavior is shown as the $t^{2/3}$ power law.

Table 1 — The Annealing Conditions and the Average Parameters Obtained.

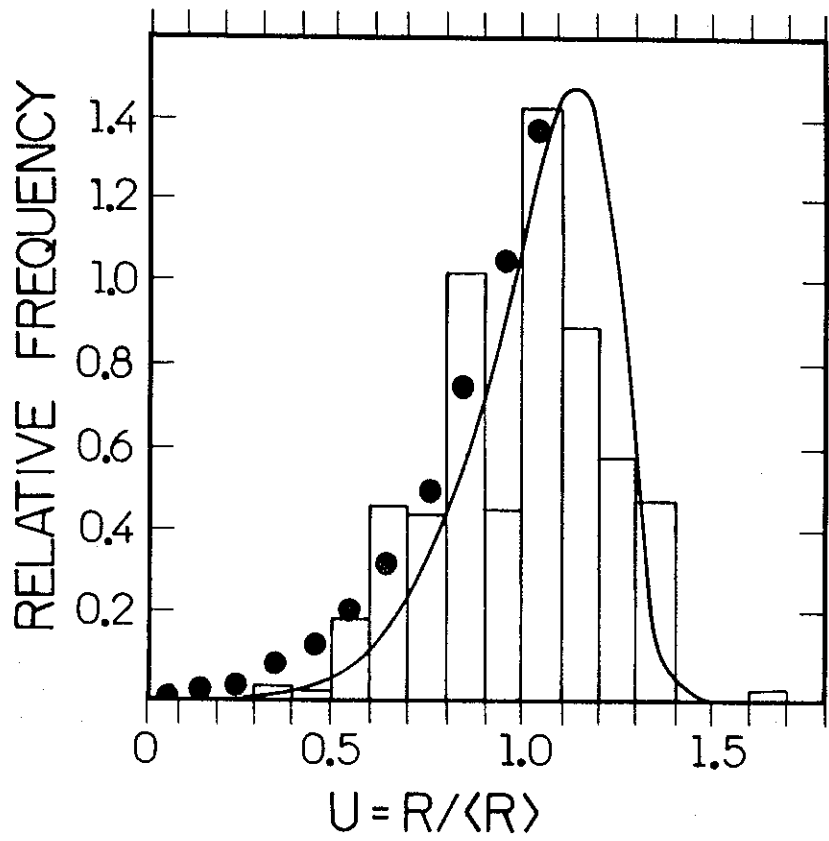
Temperature (°C)	time (min)	Scaled time (min)	N_0	$\langle R \rangle$ (nm)	$\langle (\Delta R)^2 \rangle$ (nm) ²	$\langle R^3 \rangle$ (nm) ³
500	60	2	228	4.0	2.3	93
550	30	5	102	7.5	0.9	441
600	10	10	207	9.3	4.2	921
600	30	30	231	11.8	10.1	1995
600	60	60	221	16.1	11.2	4717
650	30	215	127	25.4	15.8	17622



← 1 text column →

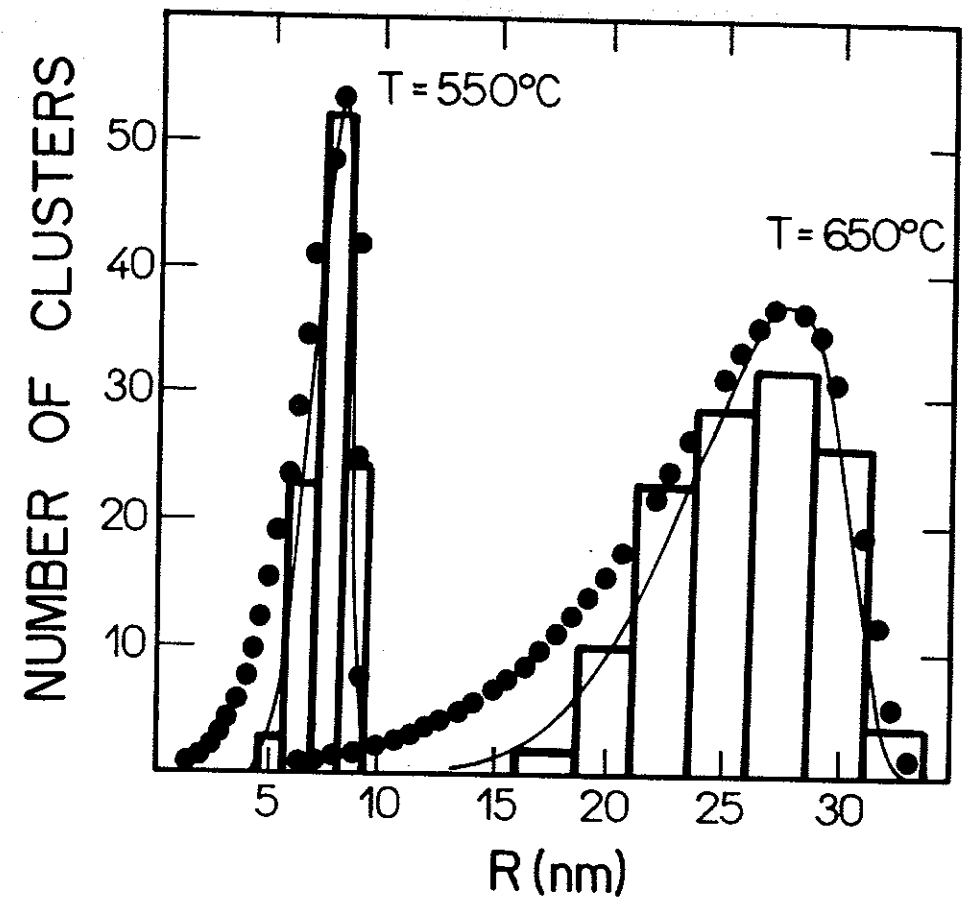
FIG. 4

Phys. Rev.
Kinetics of Diffusive Growth of Cells.



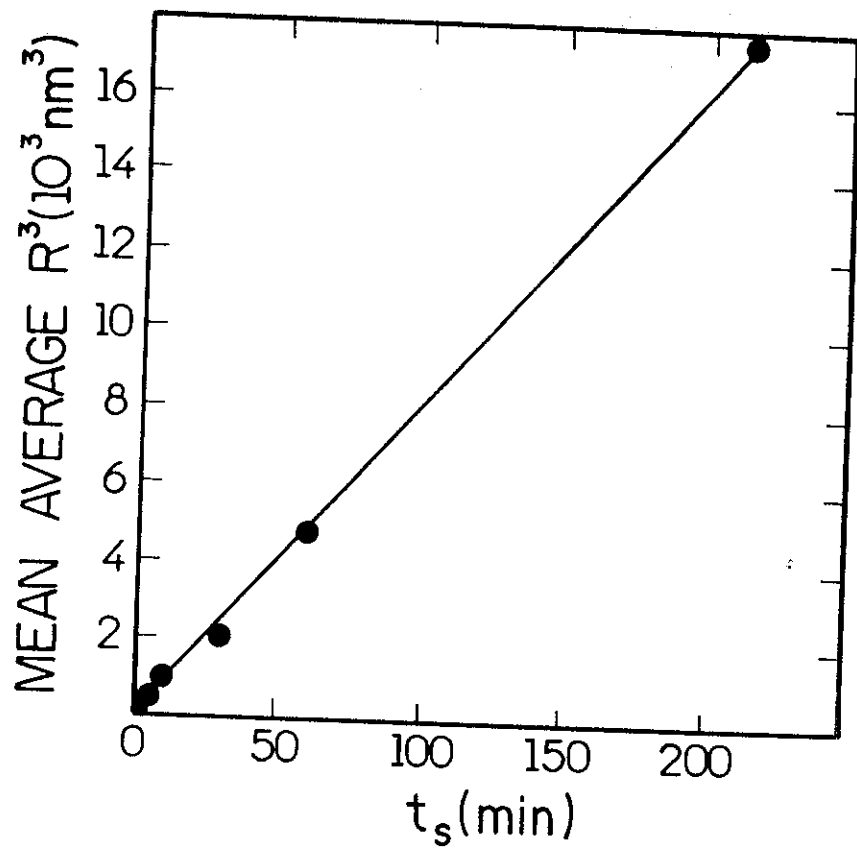
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Phys. Rev. **FIG. 2**
Kinetics of Diffusive Growth of Cds



→ 1 text column →

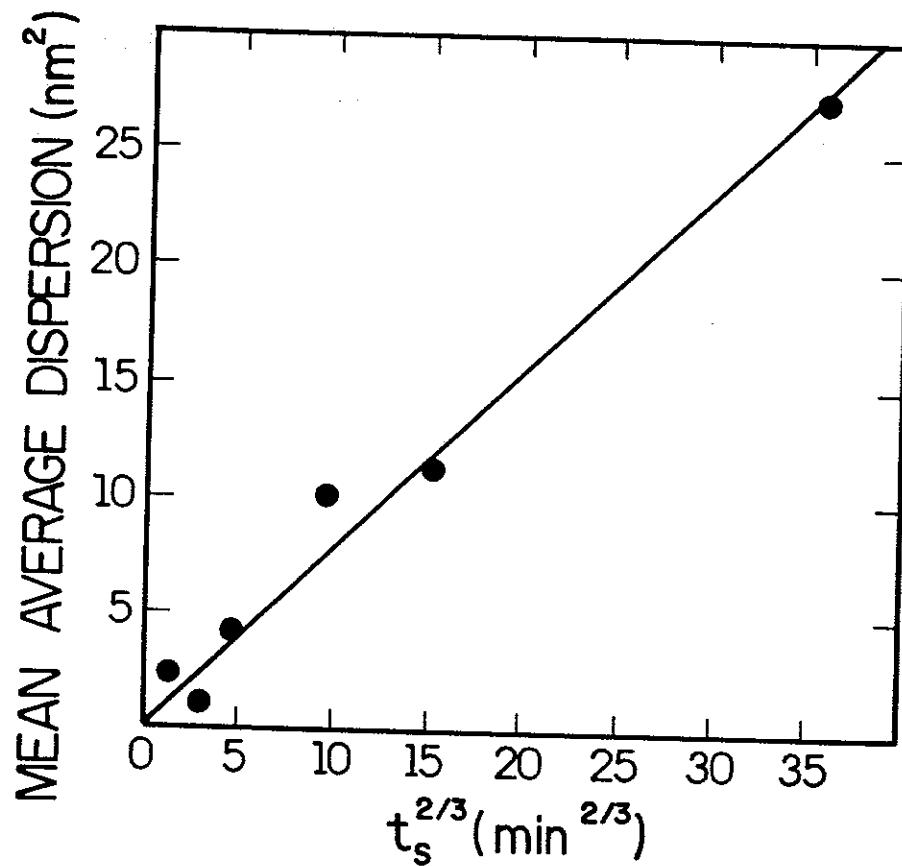
Phys. Rev. **FIG. 3**
Kinetics of Diffusive Growth of Cds.



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Phys. Rev.
Kinetics of Diffusive Growth of CdS

FIG. 4



← 1 text column →

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Kinetics of Diffusive Growth of CdS

FIG. 5