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Growth kinetics of CdS in germanium oxide glassy matrix

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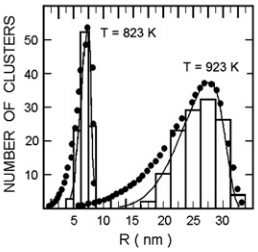
HIGHLIGHTS

- ► CdS microcrystals in glassy matrix of Germanium oxide.
- ► Mean average radius, dispersion, volume agrees with $t^{1/3}$, $t^{2/3}$ and t laws.
- ► Results fits Lifshitz–Slyozov–Wagner distribution modified to limit in origin.
- ► Growth process analyzed with evaporation overcome by precipitation rate.
- ► First order approximation showed a good agreement with the t law.

GRAPHICAL ABSTRACT

Histogram of size distribution in samples annealed at 823 K and 923 K. Dots are the Lifshitz–Slyozov distribution, and solid lines are the modified Lifshitz–Slyozov distribution.



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ABSTRACT

In the present work we revisit the size data of CdS microcrystals previously collected in the glassy matrix of Germanium oxide. The CdS clusters analyzed using electron microscopy images have shown a wurtzite structure. The mean average radius, dispersion and volume evaluated from the histograms showed good agreement for $t^{1/3}$, $t^{2/3}$ and t laws, respectively. We observed that the amount of microcrystals remains constant throughout the heat treatment process, as well as that the radii distribution has a lower limit and increases with heat treatment. The distribution of radii follows a distribution similar to the Lifshitz–Slyozov–Wagner distribution limited in the origin. Discussions led to the conclusion that the growth of CdS is a process that occurs after the fluctuating nucleation and coalescence phases. We then analyze the growth process, assuming that the evaporation is overcome by the precipitation rate, stabilizing all clusters with respect to dissolution back into the matrix. The problem was simplified neglecting anisotropy and the assuming a spherical shape for clusters and particles. The low interface tension was described in terms of an empirical potential barrier in the surface of the cluster. The growth dynamics developed considering that the number of clusters remains constant, and that the minimum size of these clusters grow with time, as the first order approximation showed a good agreement with the t law.

1. 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 very difficult to obtain, and have potential applications in non-linear optics [1–8]. Many studies have reported the optical properties of CdS and CdSe clusters [9–21]. The size variation of the clusters in heat treatment have been analyzed using the theoretical model of coarsening developed almost 40 years ago [22–37]. This theory successfully and qualitatively predicts coarsening kinetics and optical properties, but does not provide the quantitative determination of parameters such as

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the 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 them has found general acceptance and none takes into account all the factors that can affect coarsening [38–40].

It was pointed out that two stages must be distinguished in the cluster growth process in a supersaturated solution. In the first stage, fluctuating nucleation centers are formed, and their growth occurs directly outside 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 as smaller ones dissolve.

The exact asymptotic solution of the problem, derived by Lifshitz and Slyozov [23,24] and independently by Wagner [25], shows that the cluster average volume grows as t and the total number of cluster decreases as the t^{-1} power law. Clusters larger than a critical radius $|R_{CR}|$ are unstable with respect to the degree of supersaturation and growth. Clusters smaller than $|R_{CR}|$, are also unstable, and dissolve back into the matrix. The precipitation rate dominates and the degree of super saturation decreases, leading to a time-dependent increase of $|R_{CR}|$. Thus, as the process progresses, increasingly larger particles have radii below $|R_{CR}|$, causing a shrinking, which increases the average radius of stable clusters, and decreases the number of clusters per unit volume [14].

The size distribution of the CdS clusters [14,15] shows the characteristic vanishing of the distribution above certain values of cluster radius, predicted by LSW (Lifshitz–Slyozov–Wagner) model and the characteristic abrupt increase to a maximum, near 2/3 of the maximum radius. However the size distribution is slightly broader and shows no continuous tail extending to a vanishing radius. The measurements show a well-defined size cutoff at R_m, below which no clusters have been found. The R_m value was not the same for all measurements and actually grows with heat treatments over time as the $t^{1/3}$ power law [14]. These observations show strong evidence that the clusters' growth is not a pure coarsening process.

The aim of this work is the analysis of the size distribution of CdS using an empirical distribution, obtained by modifying the LSW distribution to include the suppression of the continuous tail to the vanishing radius. Also, the mean average radius, dispersion and volume are fit for $t^{1/3}$, $t^{2/3}$ and t laws, respectively.

2. Experimental

The data of CdS grown in germanium oxide glassy matrix used in the present report were collected from a previous report from Arai et al. [15] and Ph.D. Thesis of one of the author's [41]. A mixture of GeO_2 (87.7%), $NaGeO_3$ (8.92 mol%), Cd (0.9 mol%) and S (2.59 mol%) powders was sealed in an evacuated double-covered quartz ampoule (1×10^{-6} Torr). It was heated up at 873 K for 12 h while being rocked and heated up to 1423 K for 8 h to form CdS particles. After that, it was quenched by cooling in air at room temperature. The resulting ingot was sliced with a conventional wire saw. Each slice was annealed for various times at temperatures listed in Table 1.

3. Results

The micrographs have shown that the CdS clusters have a wurtzite structure and are fairly well separated. Therefore the radius of these clusters was measured along their diameter. Fig. 1 shows the histogram of size distribution in samples annealed at 823 K and 923 K by 30 min.

Table 1
The annealing conditions and the average parameters.

Temperature (K)	Time (minute)	Scaled time (minute)	N_0	$\langle R\rangle$ nm	$\langle \Delta R^2\rangle$ $(nm)^2$	$\langle R^3\rangle$ $(nm)^3$
773	60	2	228	4.0	2.3	93
823	30	5	102	7.5	0.9	441
873	10	10	207	9.3	4.2	921
873	30	30	231	11.8	10.1	1995
873	60	60	221	16.1	11.2	4717
923	30	215	127	25.4	15.8	17622

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 reported micrograph data [15,41]. We observe that N_0 remains almost constant.

In Table 1, we also show the scaled time, t_s, defined to allow the comparison between the data obtained at different temperatures and times. The scaling was performed by assuming a $t^{1/3}$ law for $\langle R\rangle$, observed for the CdS [14,15].

Fig. 1a shows an excellent overall agreement between the data and the mean average volume law. Fig. 1b shows an excellent overall agreement between the data and the mean average dispersion $\langle \Delta R^2\rangle = \langle R^2\rangle - \langle R\rangle^2 \propto t_s^{2/3}$.

The analysis of the present data also reinforces the observation reported by Potter and Simmons [14] of a minimum size, R_m, below which no cluster could be found. The existence of clusters of size below the observation capacity was discarded, in view of the observation that the smallest clusters grown at a temperature of 923 K for 30 min are larger in absolute value than those clusters grown at a temperature of 823 K for 30 min, as shown in Fig. 2. The histograms of Fig. 2 also show no continuous tail extending to a vanishing radius.

We observe in Fig. 2 that the LSW distribution (dots) [23,24] fits the larger cluster distribution well, but fails to predict the absence of small clusters.

The excess of small clusters can be suppressed from the LSW model, assuming a minimum size, R_m, below which no cluster was found [14]. Accordingly, the Lifshitz–Slyozov distribution is modified by replacing the u^2 factor by $(u-u_m)^2$ and with $P(u)=0$ for $u<u_m$, where $u=R/\langle R\rangle$. The modified LSW distribution is then given by

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

where A is a constant.

Fig. 3 shows the distribution in function of the normalized radius $u=R/\langle R\rangle$ for the overall data of CdS, containing glassy matrices annealed for 60 min at 773 K, 30 min at 823 K, 10 min, 30 min and 60 min at 873 K, and 30 min at 923 K. A good agreement with the data was obtained for $A=88$ and $u_m=0.3$ shown as solid lines. Therefore, since $\langle R\rangle$ follows the $t^{1/3}$ law, we conclude that R_m also follows the $t^{1/3}$ law.

The observation that the number of clusters remains almost constant with the annealing time is quite different from the t^{-1} power law predicted by the LSW model for the time variation number of clusters. Although the LSW model predicts the $t^{1/3}$ power law for the average radius data, the existence of a minimum cluster size and the number of clusters that stay constant along time annealing shows that the growth of CdS clusters does not follow a pure coarsening process.

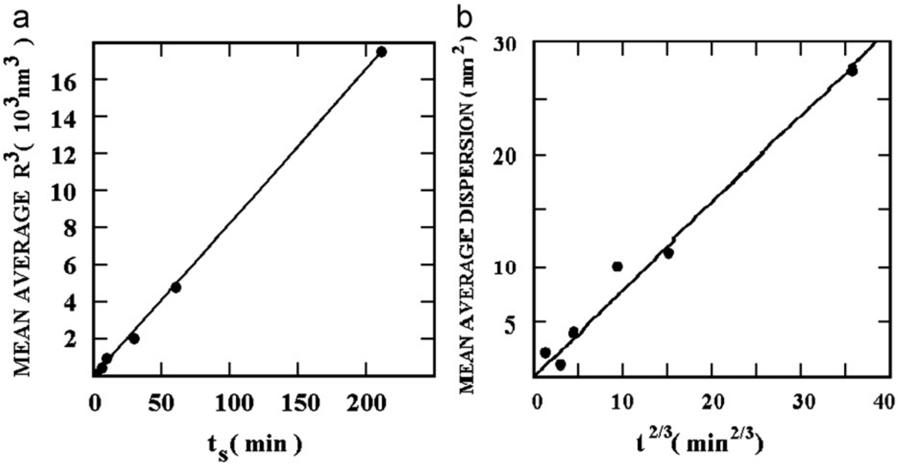

Fig. 1. The average behavior of $\langle R^3 \rangle$ and the average square radius dispersion.

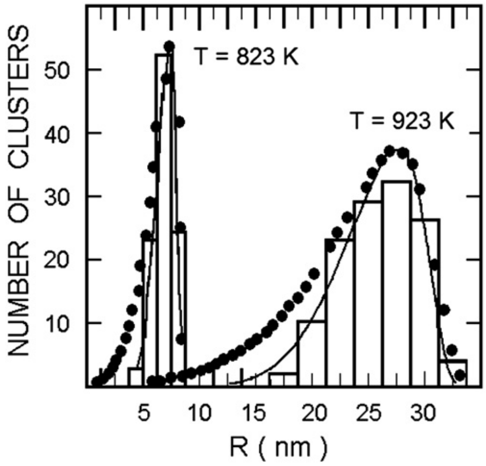

Fig. 2. Histogram of size distribution in samples annealed at 823 K and 923 K by 30 min. Dots are calculated with the Lifshitz–Slyozov distribution, and solid lines are calculated with the modified Lifshitz–Slyozov distribution.

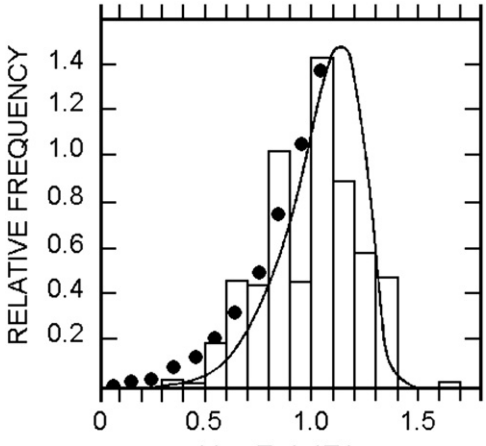

Fig. 3. Comparison of the Lifshitz–Slyozov (dots) and modified Lifshitz–Slyozov (solid line) distribution function with the size histograms for CdS containing glassy matrices annealed by 60 min at 773 K, 30 min at 823 K, 10 min, 30 min and 60 min at 873 K and 30 min at 923 K.

The existence of a minimum cluster size and the number of clusters constant along time annealing was also observed in the growth of grains of α-(FeCo) in amorphous $Fe_{68}Co_{17}B_{15}$ [42]. It was also observed that in octadecene CdSe, the number of nuclei remains constant during the synthesis. The nucleation process was observed to be very fast and the initial size prior to the growth was determined to be 1.0 nm and 0.8 nm for lower and highest oleic acid concentrations [43].

4. Modeling the growth process

The average radius data follows the $t^{1/3}$ power law predicted by the LSW model. However, we found the minimum size of crystals in the growth process, and the number of clusters remain constant, in contrast with the absence of minimum size and t^{-1} power law for the number of clusters predicted by the LSW model.

The LSW theory has as its starting point neglect anisotropy and assumes the nanocrystals to be spherical of radius R. The equilibrium concentration on the surface of nanocrystals is assumed as the sum of the concentration of saturated solution plus the contribution of the interphase surface tension. Assume also that the degree of supersaturation is small and overlooks the interaction between the nanocrystals. There is a critical radius below which the nanocrystals dissolves and above which the nanocrystals grows. From these considerations is generated a continuity equation of distribution of sizes of nanocrystals and an equation for the conservation of matter. The LSW solution is basically an asymptotic solution of these equations for a given initial distribution. To solve this issue examines the asymptotic variation of the sizes of nanocrystals. The grain size below the critical nanocrystals decrease and disappear and the larger grains grow. However as the size decreases the critical supersaturation increases and overlaps the nanocrystals that are growing. So there will always be decreasing and growing nanocrystals. We observed in this study a process in which smaller nanocrystals do not diminish in size during the heat treatment process. A possibility that occurs at the moment is that increase of the critical radius with decreasing supersaturation does not exceed the rate of rise of the nanocrystals, modifying the condition of solution of the equations.

The optical absorption spectra analysis [10] showed that the evolution of CdS in a glassy matrix is dominated by the homogeneous nucleation of particles that are 1.4–1.8 nm in radius, followed by a ripening stage, during which larger particles grow at the expense of small particles. Since the average radius ranges from 4 to 25 nm, the process described in the present report must be happening after the ripening stage.

As the LSW model does not describe the after-ripening stage, here we suggest an empirically based set of equations to describe this process for the CdS evolution in a glassy matrix.

As the number of clusters is constant, we assume that the nucleation of new clusters has 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 by means of the evaluation of the asymptotic time dependence of the average $\langle R^3 \rangle$ and comparing it with experimental data.

Although small-angle X-ray scattering showed that polyhedral model is needed to account for the surface of the scattering particles [44], to simplify the analysis, anisotropy is neglected and a spherical shape for cluster and particles is assumed. A condensation flux and an evaporation flux compose the diffusion flux of the dissolved substance per unit of cluster surface. The evaporation flux is neglected because the precipitation rate dominates. The condensation flux of the dissolved substance per unit of cluster surface is determined following the originally applied theory of the colloidal flocculation.

The X-ray absorption fine structure measurement at the Cd K edge of CdS nanocrystals grown in borosilicate glass showed a low interface tension, and suggests that glass-embedded nanocrystals are more stable than bare or capped nanocrystals of the same size [11]. We describe this stability in terms of an empirical potential barrier.

The clusters are formed when the condensation of CdS particles overcomes the potential barrier. This potential probably results from the energy need for the relaxation of the cluster structure during the process of condensation of new substances. Here, we consider the potential barrier U sketched in Fig. 4.

The density of current in the direction of clusters with a potential barrier U is given by

$$\overrightarrow{J} = -\nabla(D\overline{n}) - \frac{D\overline{n}}{kT}\nabla U, \tag{2}$$

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

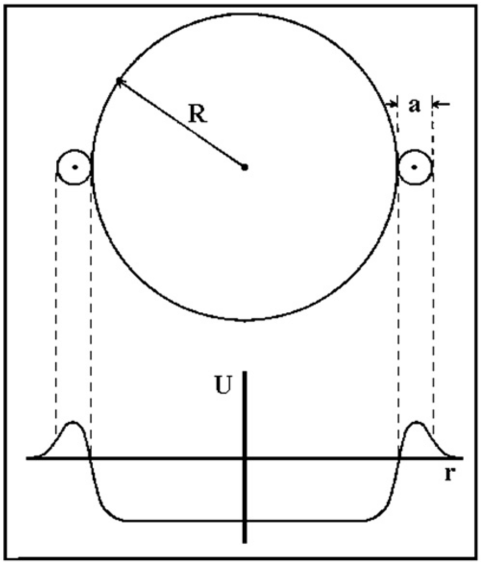

Fig. 4. Sketch of the spherical potential energy of a CdS particle in the CdS cluster.

Neglecting evaporation flux and assuming spherical symmetry, the density of the current only has a radial component given by

$$J = -\left[\frac{\partial(D\overline{n})}{\partial r} + \frac{D\overline{n}}{kT}\frac{\partial U}{\partial r}\right] \tag{3}$$

The current of the substance is then given by $I = -4\pi r^2 J$, which, using Eq. (3), then gives

$$\frac{I\exp(U/kT)}{r^2} = -4\pi\frac{\partial(D\overline{n}\exp(U/kT))}{\partial r} \tag{4}$$

Integrating Eq. (4) from R to ∞, assuming that I is independent of r, $n = \lim \overline{n}_{r\to\infty}$, $\lim U(r)_{r\to\infty} = 0$, $D_0 = \lim D_{r\to\infty}$ and $I(R,t)$, Eq. (4) gives

$$I(R,t) = -4\pi D_0 f(R) n(t) \tag{5}$$

where R is obtained at $\overline{n}(R) = 0$, $f(R) = R^2\eta(R)$ and

$$\eta(R) = \left[R^2\int_R^\infty \exp\frac{(U/kT)}{r^2}dr\right]^{-1}$$

is the reflection factor for the process. Here, since the number of cluster remains constant, we assume that the formation of nuclei has ceased, i.e., the nuclei reached a critical size at which the nucleation rate is negligible. We also 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, 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 and summing over the current of particles in the direction of all clusters gives the rate equation

$$\frac{dn(t)}{dt} = -4\pi D_0 n(t)\sum_{k=m}^{\infty} f(x_k)\rho_k \tag{6}$$

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 $I(R_k,t) = -4\pi D_0 f(R_k)n(t)$. 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 $I(R_k t)\rho_k\delta t$. 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, for $k > m$, the time variation of ρ_k is given by

$$\frac{\partial\rho_k}{\partial t} = -\left[I(R_k,t)\rho_k - I(R_{k-1},t)\rho_{k-1}\right] \tag{7}$$

Using $R_k = ak^{1/3}$ and $k \gg 1$ we obtain $R_k - R_{k-1} \approx a^3/3R_k^2$. Then from Eqs. (6) and (7) are rewritten as

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

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

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

In the limit of very small $(R_k - R_{k-1})$ (large clusters), the Eqs. (8a) and (8b) become

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

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

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.

The microscopic description of the CdS clusters growth using Eqs. (9a) and (9b) is open with the respect to the total 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 number of particles, Φ, at any instant of the process is given by

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

This equation gives the conservation condition of the growth process.

We verify the consistency of the growth process described by Eqs. (9) and (10) in the following way.

First, the time derivative of this number must vanish because the number of particles is conservative. This condition can be verified by calculating the derivative of Eq. (10). Using Eq. (9b), we find

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

Integrating this equation by parts, we obtain

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

Since there are no clusters below R_m, we have the condition $N(R_m,t)=0$. Also, because $N(R,t)$ vanishes for large R, $Rf(R)N(R,t)$ also vanishes. Then, using Eq. (9a), $d\Phi/dt$ vanishes, giving a constant value for Φ, which satisfies the conservation of particles.

Second, we verify the time dependence of the total number of clusters given by

$$N(t) = \int_{R_m}^{\infty} N(R,t)dR \qquad (13)$$

The time variation of $N(t)$ using Eq. (9b) is given by

$$\frac{dN(t)}{dt} = \frac{4\pi a^3}{3} D_0 n \left[\frac{f(R)}{R^2} N(R,t)\right]_{R_m}^{\infty} \qquad (14)$$

From the conservation conditions, Eq. (14) vanishes and $N(t)$.

The above considerations of the number of particles and number of clusters shows that the present description of the growth process is self-consistent.

The average value for R^ℓ is given by

$$\langle R^\ell \rangle = \frac{\int_{R_m}^{\infty} R^\ell N(R,t)dR}{\int_{R_m}^{\infty} N(R,t)dR} = \frac{\int_{R_m}^{\infty} R^\ell N(R,t)dR}{N_0} \qquad (15)$$

Then, from Eqs. (10) and (15), one obtains

$$\langle R^3 \rangle = \langle R^3 \rangle + \frac{a^3}{N_0}[n_0 - n(t)] \qquad (16)$$

Since the degree of supersaturation in a glassy matrix is small (only a small doping amount of CdS particles was introduced) and the diffusion of these particles through the glass network is a slow process, we can assume a first order expantion for $n(t)$. Then, from Eqs. (9a) and (16), one obtains

$$\langle R^3 \rangle = \alpha t \qquad (17)$$

where

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

Eq. (17) agrees with the experimental dependence shown in Fig. 1.

5. Conclusions

In the present work, we report an analysis of the size distribution of CdS clusters grown in the GeO_2 glassy matrix, for several temperatures and heating times. The size distribution is similar to that predicted by the LSW model. A modification of this distribution, suppressing the continuous tail that extends to the vanishing radius, shows good agreement with the data. The mean average radius, dispersion and volume evaluated from the histograms showed good agreement for $t^{1/3}$, $t^{2/3}$ and t laws, respectively. Discussion led to concluding that the present observed growth process is a phase, after the fluctuating nucleation and coalescence phases.

Here, we use the absence of the continuous tail extending to the vanishing radius as a remark regarding the cluster growth modeling. We assume that the evaporation is overcome by the precipitation rate, stabilizing all clusters with respect to dissolution back into the matrix. The problem was simplified by neglecting anisotropy, and assuming a spherical shape for cluster and particles. The low interface tension was described in terms of an empirical potential barrier in the surface of the cluster. The growth dynamics were developed considering that the number of clusters remains constant, and that the minimum size of these clusters grows with time, as the first order approximation showed a good agreement with the t law for $\langle R^3 \rangle$.

Therefore, we conclude that the present growth dynamics process given here has a good possibility of describing the third stage of cluster growth process in CdS. The present model, however, describes the observed time dependence laws; it is still incomplete because it neglects the term that describes the evaporation process. Also, solving the integro-differential equations is very difficult and complex. Finally, chemical effects that are often ignored, i.e., mechanisms that may lead to progressive re-solution of the smaller nanocrystals as the temperature (or annealing duration) is increased [45] should be considered. An example of such effects is given in a somewhat different context in nanocrystals nucleation in silver doped silicate glass [46], and was apparently also applied to the case of PbS in silicate glass[47].

The next study to be done must be the introduction of the effect of evaporation flux. According to Tolman [48] surface tension can be expected to decrease with the decrease in droplet size over a wide range of circumstances. A model in terms of the size-dependent surface energy found that the surface tension drops monotonically with the size of the droplet in the region of nanometer [49]. Thus, on the basis of these studies and by the introduction of evaporation flow, we will be able to analyze the intriguing experimental observation of cessation of evaporation in CdS small nanocrystals described in this work and also an attempt to explain the cut-off on the small nanocrystals side of the LSW distribution.

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