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U. Woggon

Optical Properties of Semiconductor Quantum Dots

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With 126 Figures

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Preface

Systematic research on quantum dots began in the early 1980s with the identification of quantum confinement in small, nanocrystalline semiconductor inclusions in glasses and colloids, though the use of semiconductor-doped glasses as edge-filters or as Q-switches in lasers is much older. The "quantum dot" has been rapidly developed to an intensively investigated model system of basic research, extending the physics of reduced dimensions to all three space coordinates. A variety of theoretical calculations have been published concerning, for example, the determination of energy states and wave functions. At that time, comparison with experimental results was limited by the problem of how to manufacture quantum dots with reproducible properties. It looked like a failing attempt to confirm the emerging theory of three-dimensional spherical confinement by reliable experimental results. As an efficient tool to monitor the electronic properties of quantum dots, methods of linear and nonlinear optics have been successfully established in experiments. Thus, the first detailed information could be gained by optical spectroscopy in the studies of the development from clusters to the solid state with growing dimensions of the crystallites.

This work is aimed at pointing out to the reader the current knowledge about the optical properties of quantum dots. Through discussions of experiments into linear and nonlinear optics and into electro-optics, the intrinsic electronic properties of three-dimensionally confined semiconductors will be illustrated. In choosing the topics of the chapters of this book, I tried to touch on all important experimental activities in the field of quantum dots.

The book starts with the illustration of the growth and precipitation process of the crystallites in different environments, including epitaxial growth. To characterize the intrinsic electronic properties we compare theoretical calculations of the confined energy levels with experimentally identified energy positions found by using the great variety of spectroscopic methods available. The development from one-electron–hole-pair states to two-pair states and many-particle systems will follow. I will deal with some corrections due to deviations from the ideal particle-in-a-spherical-box problem, introduced, for example, by peculiarities in the band structure, the finite height of the potential barriers, or by differences in the dielectric constants between the semiconductor and the host material. Since knowledge of not only the line

positions but also the line width is necessary to understand the optical spectra exhibited by the quantum dots, we will study the different dephasing mechanisms in quantum dots. The analysis of phase relaxation (and energy relaxation) is still at its beginning and I will give a few first examples. However, this field is going to be a central topic of future work. Furthermore, I will look at physical properties that are basic for possible applications. The physical origin of nonlinear and electro-optic properties will be discussed in the context of their exploitation in device concepts. Competitive mechanisms that lower the nonlinear or electro-optic response will be analyzed. The advantage that could be obtained by using matrix materials other than glass will be discussed. The review covers all semiconductor materials suited for creating zero-dimensional structures, starting from the wide-gap I–VII and II–VI compounds, and going via the group-IV materials and III–V compounds through the whole visible spectrum up to the near-infrared spectral range.

In spite of the work done during the last decade our current knowledge about quantum dots is rather fragmentary. The research on semiconductor materials of lower dimensions has finally left its infancy but is still far from being mature. For the next few years this field of science promises many new problems. In any case, the "quantum dot" is a model system in which we can study subjects from many different fields of science and no single semiconductor bulk system allows us to involve such a great diversity of knowledge.

In writing this book, I have benefitted from numerous discussions with many colleagues and students. Special thanks go to C. Klingshirn for his continuous interest and support, and for many valuable discussions. Many of the results could be only obtained due to close and fruitful collaborations, and I am indebted to A. Uhrig, M. Saleh, O. Wind, H. Giessen, W. Langbein, M. Portuné, V. Sperling, F. Gindele, A. Lohde, and H. Spöcker for their contributions. I am also very grateful to many colleagues for encouraging discussions and suggestions. It is a pleasure for me to thank L. Banyai, M.G. Bawendi, Al.L. Efros, A.I. Ekimov, B. Fluegel, D. Fröhlich, S.V. Gaponenko, O. Gogolin, B. Hönerlage, H. Kalt, S.W. Koch, M. Müller, S. Nomura, N. Peyghambarian, U. Rössler, I. Rückmann, L. Spanhel, and E. Tsitsishvili. Direct financial support for this work has been given by the Deutsche Forschungsgemeinschaft.

Karlsruhe, September 1996 *Ulrike Woggon*

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1. Introduction

Present semiconductor physics appears to be the physics of systems of reduced dimensionality. Artificially made semiconductor structures show a surprising variety of new interesting properties that are completely different from solid-state bulk materials and have never been observed there. The fabrication of single or periodic potential wells by simply combining two semiconductor materials of different bandgap energies and with spatial dimensions confining the motion of electrons and holes, results in many impressive possibilities for engineering of the semiconductor properties. Two-dimensionally layered material systems exhibiting quantum confinement in the direction of the growth axis are widely investigated and their study represents a new, rapidly developing field in solid-state physics.

It is understandable that scientists' efforts are directed to further decrease the dimensions to quasi-one-dimensional or zero-dimensional structures. One possibility for obtaining zero-dimensional structures is the inclusion of spherical semiconductor particles in a dielectric, transparent matrix. Such a structure is mesoscopic in all three dimensions, i.e. its radius is large compared to the lattice constant, but comparable to the spatial extension of the wave functions of excitons, electrons or holes in the corresponding bulk semiconductor material. To define these particles many different terms have been used such as quantum dots (QD), nanocrystals, microcrystallites (MC), Q-particles, or nanoclusters. The small semiconductor spheres of, for example, II–VI or I–VII compounds can be grown in different matrices, such as glasses, solutions, polymers or even cavities of zeoliths, and by different manufacturing processes, for example by melting and annealing processes, by organometallic chemistry or by sol-gel techniques. Evidence for quasi zero-dimensional structures has likewise been obtained by investigating the epitaxial growth on highly mismatched substrates. It results in the development of small islands on the substrate surface with high regularity and sufficiently small sizes to show quantum confinement.

Quantum dots are very attractive and interesting objects for scientific research of three-dimensionally confined systems. Their investigation requires an insight into many different branches of knowledge such as solid-state physics, molecular physics, photochemistry, nonlinear optics and ultrafast spectroscopy, materials sciences and structural analysis. The simplest, naturally

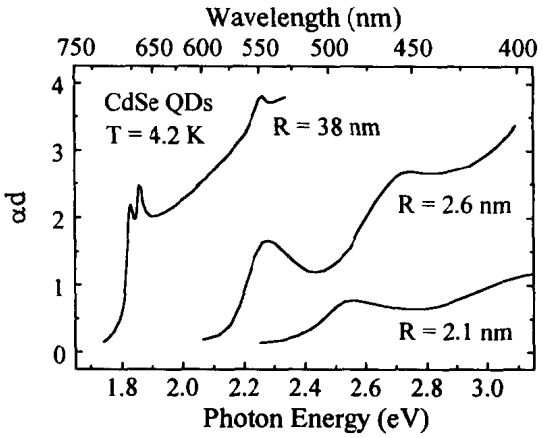

Fig. 1.1. Spectra of linear absorption of CdSe nanocrystals with different radii R = 2.1 nm, 2.6 nm and 38 nm, embedded in a borosilicate glass matrix, data taken from Ekimov et al. (1985a, 1993)

given zero-dimensional systems are spherical semiconductor nanocrystals embedded in glasses or organic matrices. This has proved to be the ideal model system for the study of basic questions of three-dimensional confinement in semiconductors. Beyond basic research there is always the question of possible applications. Promising ideas exist regarding the fields of integrated optics (active elements in waveguide structures, fast switching devices, light-emitting and laser diodes) and of physical chemistry (solar energy conversion, photocatalysis).

The growth of quantum dots in glasses is one of the oldest and most frequently used techniques. The first hints of the existence of small inclusions of, for example, CdSe and CdS in silicate glasses causing its yellow to red color were connected to the development of X-ray analysis and published in the early 1930s by Rocksby (1932). Since the 1960s, semiconductor doped glasses have been widely applied as sharp-cut color filter glasses in optics. The concept of quantum confinement, and by that the distinction between the coloring of the glasses by *changes in stoichiometry* of CdS_xSe_{1-x} mixed crystals or by *size changes* of the binary nanocrystals, was introduced by Efros and Efros (1982), and confirmed experimentally by Ekimov and Onushenko (1984). At the same time, the change of color of semiconductor colloidal solutions has been discussed in the context of quantum confinement effects by Rosetti et al. (1984). A period of control of the growth process of these nanocrystalline semiconductors followed in the 1980s combined with a detailed investigation of their linear and nonlinear optical behavior.

As demonstrated in Fig. 1.1, a first approach in understanding the behavior of quantum dots is mainly the investigation of their optical properties, in particular of their absorption spectra. Using the data first reported by Ekimov et al. (1985a, 1993) the size-dependent change of the absorption spectra is plotted in Fig. 1.1 for CdSe-doped glasses. Nanocrystals of large radii (e.g. R = 38 nm) show the typical spectrum of bulk CdSe. The spec-

trum is characterized by the sharp band-edge and, close to that, by the series of exciton states. Three peaks are located at the energy of the A-exciton ($E = 1.826\,\mathrm{eV}$), B-exciton ($E = 1.85\,\mathrm{eV}$) and C-exciton transitions [($E = 2.26\,\mathrm{eV}$, $T = 1.8\,\mathrm{K}$, (Landolt–Börstein 1982)] originating from the spin–orbit and crystal-field splitting of the valence-band states. The spectral positions of the absorption peaks shift to higher energies and the lines become broader with decreasing sizes of the nanocrystals. The explanation for these spectral changes in the absorption spectra by a *size*-dependent effect was the beginning of the intensive research on three-dimensional quantum confinement in solid-state semiconductor composite materials.

The main intention of this book is an approach to the three-dimensional quantum confinement in semiconductors by analyzing the optical properties of the corresponding semiconductor structures. To do this, one has to consider the absorption coefficient of an ensemble of quantum dots inside a transparent matrix. The averaged absorption spectrum $\bar{\alpha}(\omega)$ can then be expressed by

$$\bar{\alpha}(\omega) = \frac{p}{\bar{V}_{\mathrm{QD}}} \int \mathrm{d}R \, \frac{4\pi}{3} \, R^3 \, P(R) \, \alpha_{\mathrm{QD}}(\omega, R) \tag{1.1}$$

with p the volume fraction of the semiconductor material, $\bar{V}_{\mathrm{QD}}$ the average quantum dot volume, R the radius and $P(R)$ a characteristic distribution function for the dot sizes, as well as $\alpha_{\mathrm{QD}}(\omega, R)$ the absorption coefficient of a single quantum dot. As a result of the quantum confinement effect, the absorption coefficient α_{QD} is strongly dependent on the radius R of the dot. The absorption spectrum is given by a series of Lorentzian lines for the ground and excited states at energies $E^j_{\mathrm{QD}} = \hbar\omega_j$, with homogeneous line widths Γ_j and oscillator strengths f_j

$$\alpha_{\mathrm{QD}}(\omega, R) \sim \sum_j f_j(R) \, \frac{\frac{\hbar\,\Gamma_j(R)}{2}}{(E^j_{\mathrm{QD}}(R) - \hbar\omega)^2 + \left(\frac{\hbar\,\Gamma_j(R)}{2}\right)^2} \, . \tag{1.2}$$

Equations (1.1) and (1.2) show that one needs information about the radius R, the size distribution $P(R)$, and the semiconductor volume fraction p to correlate the appearance of structures in the absorption spectrum to, for example, electronic states of the quantum dots, as well as suitable relations for the size dependence of $E(R)$, $\Gamma(R)$ and $f(R)$, the energy, homogeneous line broadening and oscillator strength, respectively. When the quantum dots are additionally exposed to, for example, electric fields, pressure or high photon densities, further interesting problems appear since then quantum confinement and external forces combine and influence the energy, line broadening and oscillator strength of the optical transitions.

The size-dependence of the optical properties of quantum dots has been one of the main subjects of research work over the last decade. The enormous growth of experimental data (e.g. Ekimov et al. 1985a, 1991; Borelli et al. 1987; Brus 1991; Bawendi et al. 1990a; Henglein 1988; Wang 1991a), and the

increasing exactness of the theoretical concepts (e.g. Banyai and Koch 1993; Haug and Banyai 1989; D'Andrea et al. 1992) resulted in a steadily growing understanding of the electronic and optical properties of quantum dots. In the present book an attempt has been made to provide an overview of the variety of phenomena which can influence the physical properties of quantum dots, starting with the matrix material, size, structure, and interfaces, but considering also excitation densities, external and internal fields, lattice properties etc. The choice of topics covered here is determined by experimental points of view. However, this work is being written in a period of rapid development and therefore without the claim of presenting a complete discussion of all experiments carried out in this field during the last few years.

We will mainly concentrate on quantum dots of II–VI semiconductors embedded in glass matrices, but consider also other semiconductor compounds as well as other types of matrix materials. The widespread quantum dot systems based on II–VI materials show absorption structures in the visible and near-ultraviolet part of the spectrum and are therefore compatible with a great number of laser sources used for experiments. The experimental techniques applied comprise almost all standard experiments of linear and nonlinear optics such as absorption, steady-state and time-resolved luminescence, pump-and-probe spectroscopy, and degenerate and non-degenerate four wave mixing. In this work I start with the growth process of the nanocrystals and then consider under which conditions a nanocrystal can be defined as a quantum dot. I will demonstrate that the application of experimental methods taken from nonlinear optics of bulk materials is a powerful tool for identifying energy states and homogeneous line broadening. As a main topic, the new, specific properties related to excitons and biexcitons in three-dimensionally confined systems will be discussed. I will deal with the different mechanisms of phase relaxation and their influence on three-dimensional confinement. The interaction with the matrix, the specific aspect of optics of composite materials and the role of interfaces will be further topics. Furthermore, I will give a short overview of experimental results which have been obtained when applying external fields. Possible applications will be discussed as well.

Currently, quantum dots derived from III–V compounds are also being studied intensively. They are prepared by etching techniques of two-dimensionally confined layered structures, ion implantation or island-like epitaxial growth. The detailed analysis of the properties of these structures is beyond the scope of this work. However, a brief survey will be given accompanied by a list of corresponding references. Recent summaries of the development in the field of interesting transport properties of quantum dots, such as single electron transport and Coulomb blockade, can be found, for example, in Kuchar et al. (1990), Merkt (1990), Reed (1993), Geerligs et al. (1993) and in the references therein.

Over the last three years enormous progress could be observed in the investigation of quantum dots obtained from indirect-gap semiconductor ma-

terials, for example Si-nanoclusters. Although we cannot provide a comprehensive presentation of this field, a short summary of the first studies will be given, taking into account that indirect-gap quantum dots are of some specific interest. For more information I refer, for example, to Littau et al. (1993), Takagahara and Takeda (1992), Brus (1994) and references therein.

In the field of theory the influence of Coulomb interaction, the description of the electron and hole states, the electron-phonon coupling, the problem of the dielectric confinement and of the interface, high-density phenomena, field action etc. have been treated during the last five years. A comprehensive representation of the theory of semiconductor quantum dots may be found in the book of Banyai and Koch (1993). Therefore a detailed review of the results of theory of quantum dots has been omitted. I refer to theory only in the case where the theoretical results are complemented or confirmed by the presented experiments.

Finally, I should mention that the concept of quantum dots is already included in modern textbooks (Peyghambarian et al. 1993; Haug and Koch 1993; Klingshirn 1995). For further information about quantum confinement, the book by Bastard (1988) provides a very good comparison of the physics of two-dimensionally confined layered systems and three-dimensionally confined quantum dots.

2. Growth of Nanocrystals

This chapter deals with the problems of the growth process of nanocrystals, i.e. with the analysis of the laws of growth, the final sizes of the nanocrystals and expected size distributions. Different procedures have been reported for the growth of nanocrystals, such as the growth inside the cavities of a zeolith, or the growth in an organic environment by stabilization of the nanocrystals by organo-metallic ligand molecules at the surface (Chestnoy et al. 1986; Henglein 1988; Wang et al. 1989, 1995; Bawendi et al. 1990a; Bagnall and Zarzycki 1990; Spanhel and Anderson 1991). In glasses, it is common practice to describe the growth of nanocrystals by the model of condensation from a supersaturated solid solution (Lifshitz and Slezov 1961; Ekimov et al. 1985a, 1991).

The understanding of the growth process is a prerequisite for the understanding of all basic properties of three-dimensional confinement in semiconductor doped glasses. Often, the description of quantum dots starts with the introduction of different classification schemes, for example the characterization of the confinement range with respect to the ratio of the radius R of the nanocrystal to the Bohr radius a_B of the exciton in the corresponding bulk material (strong, medium or weak confinement). Further classification is possible with respect to the treatment within the frame of the effective mass approximation or within the concepts of cluster physics. Other interesting parameters are the volume-to-surface ratio, the ratio between the confinement energy and the phonon energies, and the differences in the dielectric constants between the semiconductor and the matrix material.

Thus, the growth and the following characterization procedure have to provide a minimum of data before the study of basic electronic and optical properties of quantum dots becomes meaningful. The most important information to be obtained from growth analysis concerns the sizes, the size distribution, stoichiometry, structure and the interface configuration of the nanocrystals.

2.1 Growth of Nanocrystals in Glass Matrices

2.1.1 The Diffusion-Controlled Growth Process

How big will nanocrystals be if they are grown in a certain matrix, over a fixed time and within a given range of temperature? How much time is needed to achieve a certain mean radius and what size distribution is then obtained? To describe the growth process it is interesting to look for models giving answers to these questions.

In the course of a real growth process, however, several growth stages may occur and sometimes even coexist. For example, we know the nucleation process with the formation of stable nuclei, the normal growth stage where these nuclei grow from the supersaturated solution and, at low values of supersaturation, the onset of the competitive growth where the larger particles grow due to the dissolution of smaller particles. For simplification, theoretical description commonly starts with the separate analysis of these different stages of growth to obtain simple analytical expressions.

The attempts to find a functional description of the growth process, or generally of phase transitions, go back to the early 1930s (Becker and Döring 1935). The main idea of the classical homogeneous nucleation theory consists in the ansatz that the phase transition proceeds (at constant temperature) between a supersaturated vapor of n monomers (molecules) and droplets containing $i\,n$ ($i \geq 2$) monomers. The starting point is the rate equation determining the number of monomers n inside the droplets (clusters). This equation relates the gain g_n and the losses l_n, where the size of the clusters changes by the gain or the loss of one monomer

$$\frac{\mathrm{d}n}{\mathrm{d}t} = g_n - l_n \,. \tag{2.1}$$

The resulting change in the monomer density $\mathrm{d}n/\mathrm{d}t$ is determined by the difference between the number of monomers impinging on and evaporating from the cluster through the surface into the supersaturated vapor phase. The variety of the occurring growth mechanisms is expressed by introducing the explicit terms for g_n and l_n. For instance, for the growth of thin films the gain g_n can be determined by surface diffusion, interface transfer (nonthermic transport), or cluster movement and unification (Turnbull 1956; Chakraverty 1967; Abraham 1974; Landau and Lifshitz 1974; Koch 1984). The losses l_n were derived by analyzing the energy balance between surface and volume energy contributions of the condensed phase.

The primary aim of growth analysis is to evaluate asymptotic functions for the cluster size distribution. Involving detailed expressions for g_n and l_n in (2.1) and combining this equation with the matter conservation law and the equation of continuity, the cluster growth laws have been calculated. One of the most famous models is the Lifshitz–Slezov model (Lifshitz and Slezov 1961). It is often used to fit the growth process of semiconductor nanocrystals in glasses and is therefore presented here.