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Chemical Synthesis Methods for Controlling Morphology and Achieving Uniform Arrays of Metal Nanoparticles in Semiconductor Films

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Abstract

The optical properties of metal nanoparticles are significantly influenced by their morphology, and varying of their shape leads to the appearance of a number of interesting properties. The presence of sharp edges and vertices in nanoparticles with non-spherical shapes, such as polyhedral, pentagonal and, in particular, icosahedral, leads to enhanced electric-field confinement. Enhanced optical and electrical properties of nanoparticles and semiconductor films with embedded nanoparticles make them promising for various applications, including photovoltaics, optoelectronics, and light-emitting devices. Since the properties of nanoparticle-based nanocomposites are determined not only by the morphology of nanoparticles, but also by their distribution in the film volume, it is necessary to develop methods for producing nanoparticles with the possibility of controlling and varying their morphology, as well as their introduction into semiconductor films to obtain uniform arrays. This article gives a review of relevant studies, the main focus is on chemical synthesis, as one of the most common methods for producing metal nanoparticles.

Keywords: Nanocomposites; Metal and semiconductor nanoparticles; Morphology of nanoparticles; Chemical synthesis

1. INTRODUCTION

The incorporation of metal nanoparticles (NPs) into semiconductor films can significantly affect and enhance their optical, electrical, and optoelectronic properties. Embedding metal NPs into semiconductor films aims to leverage the unique properties of NPs, such as localized surface plasmon resonance (LSPR), quantum confinement effects, and enhanced light scattering. Besides that, for example, LSPR of metal NPs can significantly enhance the optical absorption cross-section of semiconductor films without increasing their physical thickness.

This approach has been explored for various applications, including photovoltaics, optoelectronics, and light-emitting devices [1–3]. The enhanced optical and electrical properties of nanoparticle-based semiconductor films make them suitable for applications where high sensitivity and efficiency are required [2–4]. For instance, it was shown that metal NPs such as Au, Ag and Al can enhance light absorption and scattering in semiconductor films [2,3,5,6]. The incorporation of Au, Ag and Cu NPs into TiO_2 thin films has been shown to increase their absorption efficiency, particularly in the visible and near-infrared regions, which is promising for solar cells applications [5,7,8]. It has also been observed that the addition of NPs in thin films decreases the optical bandgap, refractive index and extinction coefficient of films [9], QD-based polymer nanocomposites exhibit size-dependent luminescence properties [10], embedded Sn NPs in Si thin films demonstrates the near-field enhancement effect, which can improve optoelectronic performance of films [1].

The optical properties of metal NPs, such as their absorption, scattering and extinction spectra, are significantly influenced by their morphology. Spherical NPs are the simplest and most widely studied NPs shape. For spherical NPs, the LSPR is characterized by a single reso-

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nance peak in the extinction spectrum due to their symmetry, the position and intensity of this peak depend on the NPs size and the dielectric properties of the surrounding medium [11–13].

But despite their simplicity, compared to anisotropic shapes spherical NPs are less efficient in applications requiring strong electric-field enhancement, for instance, for surface-enhanced Raman spectroscopy [14,15]. Elongated NPs, such as ellipsoidal, rodlike, triangular and disc plates, exhibit more complex optical properties compared to spherical NPs. The anisotropic shape leads to the appearance of multiple plasmon modes, which are redshifted compared to spherical NPs [16–20]. Elongated NPs shapes demonstrate a variable LSPR peak position, which can be shifted based on changing the NPs aspect ratio (ratio of the longest to the shortest dimension) and depending on the purpose [12]. For example, triangular Au NPs with higher aspect ratio exhibit stronger extinction efficiencies [21].

The presence of sharp edges in NPs lead to enhanced electric-field confinement, so cubic NPs and NPs with more complex shapes, such different polyhedrons, can provide unique optical responses [14,22]. More complex NPs shapes lead to multiple LSPR peaks due to varying edge lengths and vertex angles, but a higher symmetry of NPs results, on the contrary, in a decrease in the number of peaks. Thus, cubic NPs exhibit multiple surface plasmon resonances, as the truncation increases, a number of faces and vertices also grows, the main resonance is blue-shifted, and the number of secondary resonances decreases [23,24]. The high number of faces in icosahedral NPs leads to a reduction in the number of LSPR modes compared to less symmetric shapes, since the increased symmetry averages the contribution from individual faces [23]. The high symmetry and the presence of sharp vertices of icosahedral NPs also results in a blue shift and more sharpness in the absorption maxima and increased local field enhancements compared to other shapes [25–27]. In general, pentagonal NPs exhibit distinct LSPR resonances due to their five-fold symmetry [28]. These shapes are less studied but have the potential for applications requiring specific resonance wavelengths.

Therefore, it is important to understand how different shapes, such as spherical, ellipsoidal, flakes and fibers, affect the optical properties of metal NPs, as well as to learn how to obtain different NPs morphology and also control the shape of NPs during their producing depending on technological tasks.

Chemical synthesis is the main method for obtaining metal NPs up to hundreds of nanometers in size with various shapes (spherical, ellipsoidal, in the form of flakes, fibers, etc.) [29,30]. Using the chemical synthesis method, it is possible, among other things, to obtain NPs with complex morphology and axes of symmetry prohibited by the laws of classical crystallography, such as pentagonal and, in particular, icosahedral particles [31–33]. Chemical synthesis is not only one of the most common methods for producing metal NPs, but it also makes it possible to create uniform, ordered arrays of NPs in semiconductor films. But it is a complex process that involves simultaneous precise control over NPs shape and size, as well as their distribution and incorporation into the film volume. That is why the development of methods for producing NPs with the possibility of controlling and varying their morphology, as well as their introduction into semiconductor films or direct synthesis in their volume to obtain uniform two-dimensional arrays is an important area of research in the field of materials science and nanotechnology.

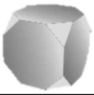
Studies of NPs devoted to variation of their morphology and introduction into semiconductor films were not considered well. In this article the set of relevant studies were identified and categorized based on the NPs synthesis method (Table 1) and method of nanocomposite films producing (Table 2).

2. PRODUCING NANOPARTICLES WITH VARIOUS MORPHOLOGIES

A review of experimental studies has shown that chemical synthesis methods allow to obtain NPs with different morphologies, and often this requires only varying synthesis parameters. It was shown that NPs size and shape depend on various factors, such as solution concentration and solvent type, evaporation rate and drying process. Key factors also include the choice of reducing/stabilizing agents, reaction kinetics, and environmental conditions, which collectively influence nucleation, growth speed, and final particle characteristics. For example, the NPs shape can change depending on the growth parameters, such as molecular structure of polyols, from wire to versatile rod, cube, and sphere [34].

Silver and gold, in particular, have been intensively studied owing to their numerous applications that include catalysis, surface-enhanced Raman scattering (SERS), as well as chemical and biological sensing [35]. Ag NPs display several interesting properties that could be improved through their morphology control [36]. Shape control of metal NPs has received considerable attention in recent years because of the strong correlation between the shape and the chemical, physical, electronic, optical, magnetic, and catalytic properties of NPs [35]. For example, investigation of effects of crystal shape on the catalytic performance of Ag NPs for hydrogen generation from formaldehyde solution shown that silver nanocubes were more active than nanorods, nanowires, and nanospheres [34]. Au icosahedral particles demonstrated better efficiency

Table 1. Comparison of methods for Ag NPs producing.

NPs shape	Synthesis method	Morphology control method	Ref.
sphere 	chemical reduction	the choice of reducing agent and reaction conditions (temperature, pH, concentration)	[58,59]
	sol-gel	annealing temperature	[43,44]
	modified polyol synthesis	reflux and microwave irradiation, temperature	[60–63]
cube  truncated cube 		varying the molecular structure of polyols	[34,64,65]
		light radiation	[66]
	polyol synthesis with seed catalysis	changing the concentration of Na_2S in the solution	[45,67]
	polyol synthesis	etching of twinned particles and seeds, leaving only the single-crystal particles	[35,68,69,70]
plate: triangular  disc 	modified polyol synthesis	reflux and microwave irradiation, temperature	[61,71]
	polyol synthesis with seed catalysis	varying the concentration of ascorbic acid	[72]
		regulating reaction conditions	[73–76]
		type of DNA: poly C and G - triangular plate, poly T - disc plate	[77,78]
wire, rod 	polyol synthesis with seed catalysis	changing the concentration of Na_2S in the solution	[67,79,80]
	modified polyol synthesis	varying the molecular structure and concentration of polyols (1,2 PG, 1,3 PG)	[34,81,82]
polyhedral: tetrahedral  octahedral  pentagonal, in particular, icosahedral 	green synthesis with microwave heating	microwave irradiation time, silver precursor and capping agent concentration	[83]
	polyol synthesis	facet-selective capping agents	[84]
	hydrothermal method	molar ratio between PVP and $AgNO_3$	[85]
	chemical reduction	double reductant method	[86]
	chemical reduction	photochemical selection	[87,88]
		stepwise growth of tetrahedral units	[89]
	polyol synthesis with seed catalysis	thermal regrowth of decahedral NPs	[90]
		surface blocking with sodium polyacrylate	[91]
	hydrothermal method	amount of ammonia	[92]

for surface-enhanced Raman spectroscopy compared to spherical particles with similar sizes [37].

Some common techniques of synthesis NPs include chemical reduction, photochemical synthesis, electrochemical synthesis, solvothermal and polyol processes. Among various methods, the solution-phase synthesis seems a promising route in the preparation of metal nanostructures because of its characteristics of low cost, high yield, and simplicity. In the general synthesis of NPs, the reduction of inorganic salts and their subsequent capping is the common procedure. The size, shape, and composition of the metal NPs can be well controlled by adjusting the reaction parameters such as temperature, precursor type, and order of addition of reactants. Additionally, metal salts and acids such as $FeCl_3$, $NaNO_3$ or HCl can be added to obtain new shapes.

Among these methods, chemical reduction is one of the most widely used methods for synthesizing NPs. The NPs morphology can be controlled by adjusting the concentrations of the precursor, reducing agent, and capping agent, as well as the reaction conditions [38]. Method of photochemical synthesis involves the reduction of metal

Table 2. Comparison of methods for producing nanoparticle-embedded semiconductor films.

Method	NPs material	Ref.
RF sputtering	Au, Ag, Cu	[3,5,6]
Sol-gel	Ag	[43,44]
Chemical deposition	SiO_2, ZnS	[104]
Deposited from colloidal solution	CdS, CdSe	[4,105]
Sintering of colloids	$CuInS_2$, CuInGaSe	[97,98]
Flux sublimation technique	TiO_2, ZnO	[106,107]

ions under UV light. The size and shape of NPs can be controlled by varying the intensity of UV light and the concentration of stabilizing agents [39]. Electrochemical synthesis allows for precise control over the morphology of NPs by adjusting the applied potential and current density. For example, electrodeposition from an aqueous solution of metal precursor can produce dendrites or spherical NPs depending on the concentration of solution [40]. Methods of solvothermal and polyol processes involve the reduction of metal salts in a solvent at elevated temperatures. The morphology of NPs can be controlled by adjusting the reaction time, temperature, and the presence of nucleants or growth inhibitors [41,42]. The variation of the molecular structure of polyols can be an additional powerful way to control the shape of metal NPs. For example, Ag NPs of different shapes, from nanosphere to nanowire, versatile rod, and nanocube have been obtained quantitatively with different types of polyol [34,36].

As a precursor in the chemical synthesis of Ag NPs silver nitrate ($AgNO_3$) is widely used due to its versatility, stability, and cost-effectiveness. $AgNO_3$ dissolves easily in water and other solvents, making it suitable for various synthesis methods, including sol-gel [43,44], photochemical reduction, microwave-assisted synthesis, and electrochemical methods [45,46]. In eco-friendly approaches, plant extracts (e.g., Moringa oleifera) act as both reducing and stabilizing agents during Ag NPs formation from silver nitrate [47].

In most methods, silver nitrate serves as the source of silver ions, which are reduced to elemental silver by a reducing agent [45,46,48]. Silver nitrate is reduced by agents such as sodium borohydride ($NaBH_4$), trisodium citrate (TSC), or ethylene glycol, resulting in metal silver NPs [49], another study used silver nitrate with ascorbic acid and glutathione to form colloidal silver NPs [50]. Strong reductants like $NaBH_4$ produce small, monodisperse NPs but limit size control [51]. Weaker agents like TSC enable slower reduction rates, allowing better control over shape and size distribution, which leads to shape-specific crystallization and NPs anisotropic shapes [52–55]. The use of ascorbic acid as a weak reducing agent has been shown to slow down the growth regime, leading to the formation of specific morphologies such as icosahedral particles [56].

Stabilizing agents play a crucial role in controlling the morphology of Ag NPs by stabilizing specific crystal facets and directing the growth of NPs. Stabilizing agents like polyvinylpyrrolidone (PVP), aminopropyltriethoxysilane (APS) and dodecanethiol are often added to control particle size and prevent aggregation [46], they also direct morphology to grow in specific crystal facets. For example, PVP yields spherical or prismatic Ag NPs [39,54,57], β-cyclodextrin has been used to synthesize NPs with multiply twinned icosahedral morphology [56].

Reaction conditions, such as temperature, pH, reaction time, concentration, and the presence of external salts, also affect the parameters of the resulting NPs. Higher reaction temperature accelerates reduction and NPs growth, favoring larger NPs [41,52,54]. Solution pH level affects reduction kinetics and stabilizing agent effectiveness, alkaline conditions often enhance Ag^+ reduction [45,54]. High pH values promote faster reduction rates, leading to the formation of rod-like and spherical NPs, while low pH values result in triangular or polyhedral NPs due to slower reduction rates [55]. Alkaline conditions can lead to the formation of larger NPs or aggregates, while acidic conditions may result in smaller, more dispersed particles [93,94]. The ratio of silver salt to reducing agent determines atom availability, affecting nucleation speed and particle growth. Higher silver salt concentrations initially increase NPs yield but may lead to heterogeneity over time [45,48]. At high concentrations of silver nitrate, excess precursor ions promote rapid nucleation, leading to smaller NPs, while controlled growth phases enable shape tuning [45,48,95]. A higher ratio of reducing agent to precursor can lead to faster reduction rates and the formation of smaller NPs [51].

By optimizing these parameters, researchers achieve tailored Ag NPs morphologies for different applications [52,96]. To produce Ag NPs with a complex morphology, specialized chemical methods are used that control nucleation, growth, and shape selectivity. For instance, icosahedral and, in particular, pentagonal Ag NPs are synthesized through specialized approaches leverage photochemistry, seed-mediated regrowth, and oxidative etching [86,87,90,91]. Such methods let to achieve high shape yields (>90%) and produce NPs with sizes ranging from 50 to 200 nm.

A wealth of chemical methods has been developed for the synthesis of silver and gold nanostructures that have well-controlled shapes, including triangular plates, cubes, wires, rods and others (Table 1). These techniques allow for precise control over NPs size, shape, and material, which are critical for achieving desired optical and electrical properties. Most of these methods, however, still require improvement in terms of yield, purity, and monodispersity of synthesis before they will find use in commercial applications [35].

3. METHODS FOR OBTAINING A UNIFORM ARRAY OF NANOPARTICLES ON A SURFACE OR WITHIN A SEMICONDUCTOR FILM

The study of methods for obtaining a uniform array of NPs on a surface or within a semiconductor film has shown two common approaches - embedding previously produced NPs into semiconductor films or synthesizing NPs directly within the volume of these films. One of the key challenges in the use of nanoparticle-embedded semiconductor films is material compatibility. The NPs must be compatible with the semiconductor material in terms of their chemical and physical properties to ensure optimal performance [1,2]. Another challenge is the scalability and uniformity of nanoparticle-embedded semiconductor films. The synthesis and integration of NPs into semiconductor films must be scalable and uniform to achieve consistent performance across large areas [97,98].

An example of the first approach is the method of adsorption of pre-synthesized CdSe NPs onto silicon wafers using various adsorption promoters like APTES, PEI, or PDDA [99]. It was shown that NPs array shape also depends on various factors, such as solution concentration and solvent type, evaporation rate and drying process (Table 2). For example, the NPs array morphology can undergo multiple transitions during the drying process, changing from hexagonal to cubic, tetragonal, and back to cubic symmetry [100].

The method of sintering of colloidal NPs involves the deposition of NPs on substrates, followed by thermal treatment to remove organic ligands and sinter the NPs to form thin films, such as $CuInS_2$ and $CuIn_xGa_{1-x}Se_2$ [97,98].

Chemical reduction method is one of the most widely used techniques for synthesizing metal NPs. This method uses metal salts (nitrates or chlorides) as metal precursors, chemicals like sodium borohydride or alcohols as reducing agents that reduce metal ions to neutral atoms, and stabilizer agents like surfactants or polymers to prevent aggregation and control size. By optimizing solutions composition and concentration uniform NPs can be achieved, which can then be deposited onto semiconductor films to create ordered arrays [101,102].

Green synthesis method uses biological agents such as microorganisms or plant extracts to produce NPs, so this method is the eco-friendliest and avoids toxic chemicals. Although this method is less common for use in semiconductor manufacturing, it has the potential for sustainable synthesis of NPs [102,103].

In addition to embedding pre-synthesized NPs, the possibility of in situ synthesis of NPs directly within semiconductor films has also been studied. This approach offers the advantage of better integration and uniform distribution of NPs within the film volume.

Some studies have focused on synthesizing NPs directly within the film volume, such as the formation of PbSeCd chalcogenide NPs in thin films [9].

Sputtering technology is a physical deposition method adapted from semiconductor manufacturing that uses plasma to break down bulk metals into NPs. These NPs are then deposited onto a substrate, forming a thin film. This method avoids the use of harmful chemicals and offers excellent control over NPs distribution, making it environmentally friendly and scalable [108].

Chemical bath deposition (CBD) has been used to synthesize NPs directly within semiconductor films. For example, CdS NPs have been incorporated into CdS thin films using CBD, followed by thermal annealing to improve their optoelectronic properties [4].

Hydrothermal synthesis method allows for precise control over NPs size and morphology and their uniform integration into semiconductor films. In this method NPs are grown directly on substrates by heterogeneous reaction under conditions of high temperature and high pressure in an autoclave [109,110].

Light-assisted synthesis (print-light-synthesis) is the most innovative approach, which combines the synthesis and patterning of metal NPs in a single step – first, metal precursor solutions are deposited on substrates, then high-intensity light irradiation converts the precursors into NPs directly on the substrate or film surface. This eliminates the requirement for stabilizing agents and allows for spatial resolution deposition, enabling the creation of ordered arrays [111].

Intercalation and Reduction is another innovative approach involving the intercalation of metal ions into layered semiconductor films, followed by reduction to form NPs. For example, copper ions have been intercalated into titania nanosheet films and reduced to form copper NPs within the interlayer space [106].

Sonoelectrodeposition combines ultrasound waves with electrodeposition to synthesize metal NPs. Ultrasound enhances mass transport and nucleation rates during deposition that allow it to produce uniform metal NPs (like Ag and Pt), which can be incorporated into semiconductor films [109].

Future research should focus on developing new materials and methods for the synthesis and integration of NPs into semiconductor films. This includes the exploration of new NPs materials, such as graphene and transition metal dichalcogenides, and the development of novel synthesis techniques, such as in situ synthesis and 3D printing [105,106].

4. CONCLUSION

The shape of metal nanoparticles is a powerful parameter for varying their optical properties. By producing nanoparticles into specific geometries, it is possible to adapt their optical properties to specific tasks and applications. Elongated NPs and NPs with sharp edges and multiple faces, such as polyhedral, icosahedral and, in particular, pentagonal, suitable for applications where high sensitivity are required, for instance, in surface-enhanced Raman spectroscopy and sensing applications.

The integration of nanoparticles into semiconductor films has emerged as a powerful approach to enhance the optical, electrical, and optoelectronic properties of these films. This technique has been explored for various applications, including solar cells, LEDs, and photodetectors, where high efficiency are required. However, to realize the full potential of this technology, issues such as material compatibility, scalability, and uniformity need to be resolved.

In conclusion, it should be noted that research on nanoparticles in semiconductor films continues to develop, with a focus on obtaining different morphology and improving such simple producing methods as chemical synthesis, which allows to obtain nanostructures suitable for various applications in electronics, optics and energy conversion, and to modify their properties over a wide range.

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Методы химического синтеза для контроля морфологии и получения однородных массивов металлических наночастиц в полупроводниковых пленках

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Аннотация. На оптические свойства металлических наночастиц существенное влияние оказывает их морфология, и изменение их формы приводит к появлению ряда интересных свойств. Наличие острых краев и вершин у наночастиц несферической формы, а именно формы многогранника, например, пентагональной и, в частности, икосаэдрической, приводит к лучшей локализации электрического поля в наночастице. Уникальные оптические и электрические свойства наночастиц и полупроводниковых пленок с внедренными наночастицами делают их перспективными для различных применений, включая фотовольтаику, оптоэлектронику и светоизлучающие устройства. Поскольку свойства нанокомпозитов на основе наночастиц определяются не только морфологией наночастиц, но и их распределением в объеме пленки, необходимо разработать методы получения наночастиц с возможностью контроля и варьирования их морфологии, а также их введения в полупроводниковые пленки для получения однородных массивов. В этой статье дается обзор соответствующих исследований, основное внимание в которых уделяется химическому синтезу, как одному из наиболее распространенных методов получения металлических наночастиц.

Ключевые слова: нанокомпозиты; металлические и полупроводниковые наночастицы; морфология наночастиц; химический синтез