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PLASMONIC NANOMATERIALS FORMED BY ION EXCHANGE IN GLASS: PROPERTIES AND APPLICATIONS

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Abstract. Plasmonic materials based on noble metal nanostructures exhibit unique optical properties in the visible range. Currently, plasmons supported by metal nanoparticles or propagating along metal interfaces are attracting increasing attention in sensorics, medicine, imaging, nanophotonics, and optoelectronic technologies. This article has provided a brief overview of two main plasmonic modes: surface plasmon polaritons at metal interfaces and localized plasmons in nanostructures. It also discussed the physical principles of plasmon-enhanced sensors, such as colorimetric, plasmon-enhanced fluorescence, and surface enhanced Raman scattering ones. These sensors are widely used in healthcare, security, food processing, and environmental monitoring. The fabrication of plasmonic nanostructures in glasses using ion exchange and their application for detecting chemical compounds and biological objects was discussed.

Keywords: localization of light, nanostructure, ion exchange, plasmon, electromagnetic field amplification, sensor

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ПЛАЗМОННЫЕ НАНОМАТЕРИАЛЫ, ФОРМИРУЕМЫЕ МЕТОДОМ ИОННОГО ОБМЕНА В СТЕКЛЕ: СВОЙСТВА И ПРИМЕНЕНИЕ

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Аннотация. Плазмонные материалы на основе наноструктур благородных металлов обладают уникальными оптическими свойствами в видимом диапазоне. В настоящее время плазмоны, которые поддерживаются металлическими наночастицами или распространяются вдоль металлических интерфейсов, привлекают все большее внимание в сенсорике, медицине, визуализации, нанофотонных и оптоэлектронных технологиях. В статье представлен краткий обзор двух основных плазмонных мод: поверхностных плазмонных поляритонов на металлических интерфейсах и локализованных плазмонов в наноструктурах, а также физических основ плазмонно-усиленных датчиков, таких как колориметрические сенсоры, сенсоры на основе плазмонно-усиленной флуоресценции и сенсоры на основе гигантского комбинационного рассеяния. Такие датчики широко используются в здравоохранении, безопасности, пищевой промышленности и мониторинге окружающей среды. Рассмотрено получение плазмонных наноструктур в стеклах методом ионного обмена и их применение для детектирования химических соединений и биообъектов.

Ключевые слова: локализация световой волны, наноструктура, ионный обмен, плазмон, усиление электромагнитного поля, сенсор

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Introduction

It is well known that the electromagnetic properties of noble metals, such as gold, silver and copper, at optical wavelengths closely resemble those of an electron gas. Classical electron theory predicts and numerous experiments confirm a negative real part of the dielectric permittivity for these metals at or near optical wavelengths [1]. Consequently, charge oscillations can propagate along the surface of noble and several other metals at optical frequencies. Surface charge oscillations dominate the electromagnetic response of nanoparticles with sizes of the order of several nanometers.

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A surface plasmon arises from collective oscillations of conduction electrons in nanostructured metal systems [2]. A unique property of such systems is that they can concentrate optical energy at the nanoscale via surface plasmons. If the external excitation frequency matches the surface plasmon frequency, surface plasmon resonance (SPR) occurs.

Both the intensity and the position of the SPR depend strongly on the size, shape, and composition of the nanostructures, as well as on the dielectric properties of the surrounding medium [5]. This sensitivity opens the avenue for spectroscopic methods for monitoring the interactions between the analyte and the sensor surface in real time [6]. Moreover, SPR-based sensors require only minute amounts of analyte and use label-free techniques, making it possible to forgo any preliminary preparation [7]. As a result, this approach offers a highly sensitive, versatile, and efficient technology that has been rapidly developing over the past two decades.

Surface plasmon polaritons and plasmons in nanoparticles

Plasmon resonance typically takes two distinct forms (Fig. 1):
surface plasmon polaritons (SPPs) propagating along metal–dielectric interfaces;
localized surface plasmon resonance (LSPR) in metal nanoparticles [8].

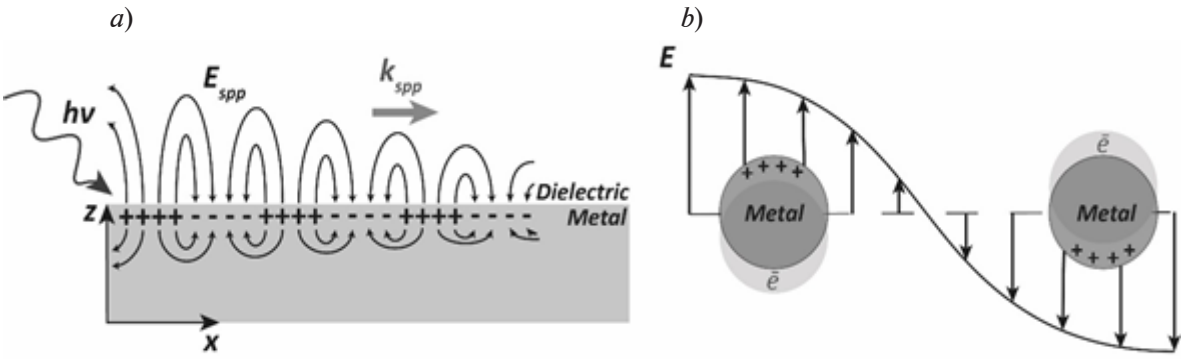


Fig. 1. Schematic of two types of plasmon resonance:
surface plasmon polariton (SPP) propagating along a metal–dielectric interface (*a*);
localized surface plasmon resonance (LSPR) in nanoparticles excited
by an incident electromagnetic wave (*b*)

LSPR occurs when the metal nanostructure is much smaller than the wavelength of incident light. Optimal nanoparticle sizes for the optical range are from 10 to 15 nm [9]. The interaction with an external electromagnetic wave excites collective non-propagating oscillations of the surface electrons in the metal nanostructure. The plasmon oscillation frequency depends on the nanoparticle morphology and the wave polarization. For an ellipsoidal nanoparticle, the LSPR frequency ω_α along the axis α is given by the following formula [10]:

$$\omega_\alpha = \omega_p \Big/ \sqrt{1+\varepsilon_d\left(1/L_\alpha - 1\right)}, \qquad (1)$$

where ω_p is the Drude plasma frequency, ε_d is the relative permittivity of the surrounding medium, L_α is the geometric factor for the given axis ($L_\alpha = 1/3$ for a sphere).

As follows from Eq. (1), the resonance frequency depends on the dielectric permittivity of the surrounding medium; this dependence is the basis for plasmonic sensors. Moreover, plasmon resonance concentrates the electromagnetic field strongly around the nanostructure. Molecules in the near field of the nanoparticle participate in radiative and non-radiative processes, whose efficiency is increased by enhanced local fields. This effect, often called the nanoantenna effect [11], underlies surface-enhanced fluorescence [12] and surface-enhanced Raman scattering [13]. Both phenomena can be exploited in sensing applications.

In contrast to LSPR, SPPs involve electron oscillations along metal–dielectric interfaces, in particular, on thin metal films. These waves are polarized in the direction normal to the surface (TM-polarization) and can exist only at interfaces between materials whose real parts of the dielectric permittivity have opposite signs (the real part of the dielectric permittivity in metals is negative at optical frequencies). At optical frequencies, waves propagating along a metal–dielectric interface take the form of electromagnetic fields coupled to surface plasma oscillations, i.e., oscillations of conduction electrons in the metal [14]. This is essentially a two-dimensional excitation whose electromagnetic field decays exponentially with distance from the interface [14].

The SPP wavevector Q at frequency ω can be written as [10]:

$$Q = \frac{\omega}{c}\sqrt{\frac{\varepsilon_m \varepsilon_d}{\varepsilon_m + \varepsilon_d}}, \tag{2}$$

where ε_m is the dielectric function of the metal.

Because the wavevector of incident light does not match that of the propagating SPP (the SPP wavevector is always larger), SPPs cannot be excited by radiation in free space. Excitation requires momentum matching, which can be achieved, for instance, via attenuated total reflection or by using a periodic structure.

The classic setup for SPP excitation is the Kretschmann configuration [15], where a metal film (typically a noble metal, most often gold) deposited on a prism reflects the incident light almost completely, except at a specific angle called the SPP excitation angle. As follows from Eq. (2), this angle depends on the refractive index of the medium, a property that sensors exploit.

Propagating SPPs or a mixed SPP/LSPR regime can be used to detect changes in the dielectric permittivity of the surrounding medium. When analyte molecules in an aqueous solution bind to the plasmonic metal film, the SPP excitation angle shifts, and the SPP wavelength increases because the molecules have a higher refractive index [16, 17].

SPP-based optical fiber sensors were also fabricated [18, 19]. These sensors rely on single-mode tapered fibers coated with a thin metal layer on one side. The optical mode propagating in the fiber can excite various hybrid surface plasmon modes in the metal layer. Consequently, the transmission spectrum of the metal-coated fiber exhibits several SPP peaks whose positions depend on the system parameters and the refractive index of the surrounding medium [18]. SPP-based fiber-optic sensors offer several advantages, including light weight, capabilities for remote sensing and multiplexing.

Sensors based on plasmons in nanoparticles

Noble-metal nanoparticles, such as gold, silver and copper, exhibit LSPR in the optical range and serve as the basis for plasmonic sensors [20]. According to Eq. (1), the LSPR position depends on the shape of the nanoparticle and its dielectric environment: the resonance wavelength shifts when analyte molecules bind to the nanoparticle surface (sometimes prior functionalization of the nanoparticle is required to achieve this), changing the local refractive index (Fig. 2).

A change in the refractive index n of the surrounding medium by Δn shifts the position of the LSPR peak λ_{res} (in wavelength units) or E_{res} (in energy units). The corresponding proportionality constant, or sensitivity, can be expressed in wavelength (S_λ) or energy (S_E) units [21]:

$$S_\lambda = d\lambda_{res}/dn,\ S_E = dE_{res}/dn = -1.240\ S_\lambda/\lambda^2\ (\text{nm/eV}).$$

The relatively large linewidth Γ complicates analysis, because this parameter can vary by more than an order of magnitude among nanostructures with different geometries. Clearly, it is easier to detect a resonance shift for narrow lines, so the ratio of the resonance shift to the linewidth

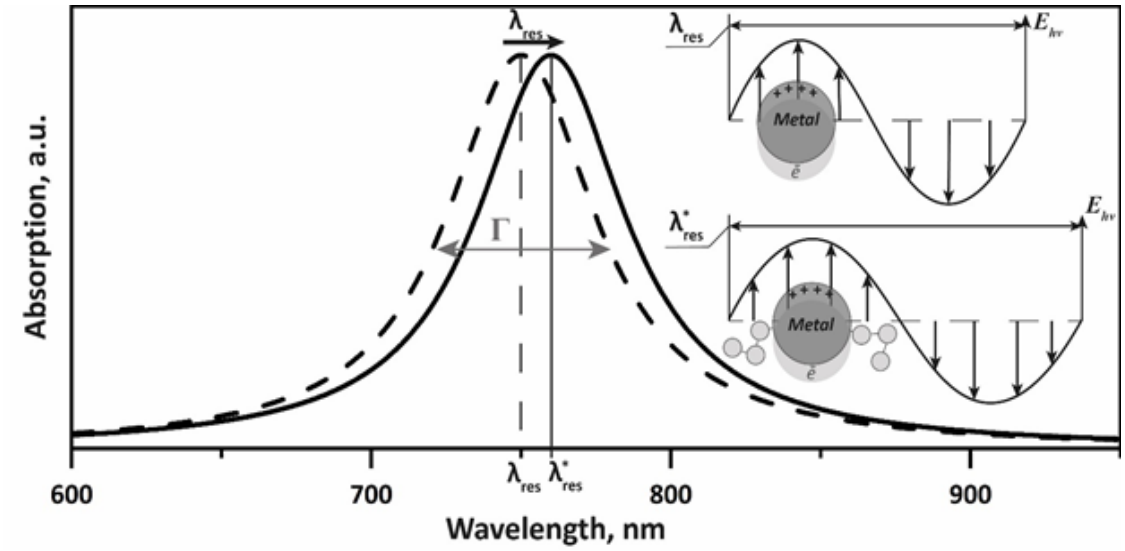


Fig. 2. Shift of LSPR peak in response to changes in the surrounding medium; key parameters are resonance linewidth Γ (full width at half maximum) and peak position λ_{res} on the wavelength axis

serves as a more accurate measure of a plasmon-based sensor's performance. This dimensionless quantity is the figure of merit (FOM) of the resonance structure [21]:

$$\text{FOM} = S/\Gamma.$$

FOM can be easily determined experimentally; it provides a convenient basis for comparing the plasmonic characteristics of different structures. Earlier studies found [22] that the FOM for small metal nanoparticles depends only on the nature of the metal itself. It reaches its highest value when damping is low (i.e., when the imaginary part of the dielectric function is small) and at frequencies where the real part of the dielectric function exhibits the strongest dependence on frequency. The resonance frequency and in turn the FOM of plasmonic sensors can be fine-tuned by adjusting the nanoparticle geometry. Among all noble-metal sensors, those based on silver nanoparticles exhibit the highest FOM, owing to silver's low losses in the optical frequency range.

Colorimetric plasmonic sensors, relying on shifts of the LSPR peak in colloidal nanoparticle solutions, offer a simple and convenient approach to detecting analytes in solution, often allowing direct visualization of analyte concentration. For example, functionalized gold nanoparticles were used for colorimetric detection of heavy metals, small biological molecules and biomacromolecules [23–25].

A new colorimetric assay was proposed in [26] for ultrasensitive visual glucose detection based on gold bipyramids. This glucose assay achieved a detection limit of 0.02 mM. Moreover, the proposed sensor can be used to estimate glucose levels by the naked eye from the color change, thus distinguishing between healthy individuals and diabetic patients without any sophisticated instrumentation.

Enhancement of the electromagnetic field near nanoparticle surfaces due to LSPR can be used in plasmon-enhanced fluorescence spectroscopy. LSPR-based sensors can achieve enhanced fluorescence by increasing either the excitation efficiency of the analyte or its emission efficiency [27]. In both cases, stronger local electromagnetic fields lead to greater enhancement. According to Eq. (1), field enhancement near sharp features is higher because of the geometric factor (often called the 'lightning rod' or 'tip' effect). Thus nanorods, elongated ellipsoids, and dendrites exhibit stronger plasmon-enhanced fluorescence than nanospheres. The enhancement factor for plasmonic fluorescence typically lies in the range of 10–100, though optimized nanostructures can yield values as high as 10^3 in some cases [28].

In particular, Xie et al. used arrays of silver nanotriangles to enhance the fluorescence of near-infrared dyes [29], finding that the low quantum yield (4%) of the dye's NIR fluorescence could be increased by two orders of magnitude if a silver nanotriangle array was used.

Surface-enhanced Raman scattering (SERS) is one of the most powerful and promising analytical techniques for biological and chemical sensing, offering several key advantages [30–33]:

high selectivity, owing to the unique spectral fingerprints of analytes;

high sensitivity, down to single-molecule detection;

operational simplicity;

capability to detect multicomponent mixtures with a single excitation laser, thanks to narrow spectral bands.

Raman scattering (RS) by itself has low efficiency because of its small scattering cross-section (on the order of 10^{-28}–10^{-30} cm^2/molecule). However, the enhanced local electromagnetic field near plasmonic nanostructures can boost the Raman signal by factors of 10^4 to 10^8 [34]. The magnitude of this local field enhancement near the plasmonic nanostructures determines the performance of SERS-based sensors; consequently, considerable research effort has been focused on developing effective SERS substrates [35–37].

An effective technique for fabricating plasmonic structures, notable for its relative simplicity and low cost, is ion exchange in glass, which can produce plasmonic structures both in the bulk and on the surface [38–41]. Combining ion exchange with glass polarization and etching makes it possible to create microchannels with plasmonic nanoparticles inside the glass, which holds promise for microfluidics applications [42, 43].

Plasmonic structures formed by ion exchange in glasses

Glasses are unique materials with properties critical to many technologies. They are transparent and hard at room temperature, mechanically strong, and highly chemically resistant. In 1752, the great Russian scientist Mikhail Lomonosov wrote in a letter to Ivan Shuvalov, the curator of Moscow University [44]:

They're wrong about it, Shuvalov: wrong to class
The gaudy look of minerals higher than our glass.
. . . . . .
When I sit before you, and to ecstasy will pass,
It's not the precious stones I praise, nor gold, but glass[1].

Ion exchange from molten salts is a well-established technology for modifying the physicochemical properties of glass. Craftsmen used this method during the Renaissance to produce ceramics coated with a thin glass film containing silver and copper nanoparticles of various sizes, formed through ion exchange between metal ions and alkali ions in the glass, followed by reduction of the metal ions and their aggregation into nanoparticles [45].

Optical glass is typically an amorphous material composed of several types of oxides [46]:

network formers (SiO_2, GeO_2 or P_2O_5);

intermediate network formers (Al_2O_3, TiO_2), which cannot form glass by themselves but can be combined with network formers;

network modifiers (Na_2O, K_2O, CaO, or BaO), which are less strongly bound to the glass network.

Ion exchange is essentially a chemical reaction where mobile ions in a solid are exchanged for ions of similar charge from a solution. Because alkali modifier ions are weakly bound to the glass matrix, they are the most suitable for ion exchange. For optical and photonic applications, the vast majority of cases involve exchanging Na^+ or K^+ ions in the glass for Ag^+, K^+, or Tl^+ ions from a molten salt bath (Fig. 3).

[1] M. V. Lomonosov, "A Letter on the Uses of Glass" (excerpt), trans. by Colin John Holcombe, Ocaso Press, accessed 20.06.2026, https://www.ocasopress.com/lomonosov-use-of-glass.html.

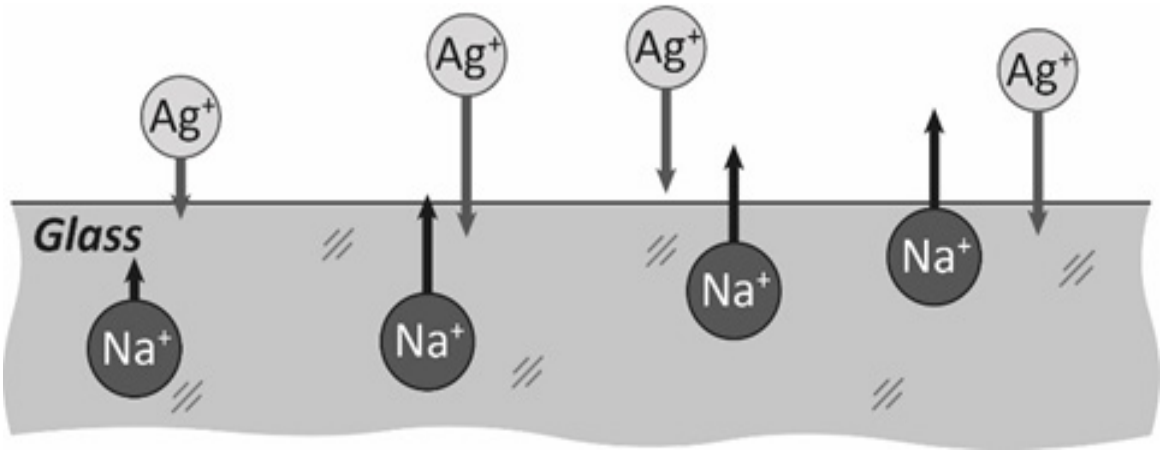


Fig. 3. Schematic representation of ion exchange in glass

Ion exchange technology is successfully used for glass strengthening [47], fabricating optical waveguides and active optical devices [48, 49], and forming metal nanostructures in glass [50]. Let us focus in more detail on the last of these applications.

Formation of metal nanoparticles in the bulk and on the glass surface. Among the many types of metal nanoparticles, silver nanoparticles appear particularly interesting for synthesis and study because silver exhibits the highest electrical and thermal conductivity of all metals [51]. The electronic and optical properties of silver nanoparticles can be controlled by choosing appropriate synthesis conditions.

Common methods for producing metal nanoparticles in glasses include rapid synthesis by quenching from the melt [52], the sol-gel technique [53], ion implantation [54], and ion exchange [55].

Ion exchange offers several advantages:

it can introduce relatively high concentrations of metal ions into the glass;

the glass remains undamaged after the exchange (unlike ion implantation, which introduces substantial defects into the glass matrix);

it allows producing large volumes of glass with metal nanoparticles.

After silver ions are introduced into the glass matrix by ion exchange, the next step is to induce phase decomposition of the supersaturated glass–metal solid solution and bring about precipitation of the second phase, metal nanoparticles. This is achieved either by annealing (thermal treatment) the ion-exchanged glass in air or in a hydrogen atmosphere, or by ionizing radiation, with or without subsequent thermal treatment. Alternatively, nanoparticles can be obtained by ion exchange in glass containing reducing agents, that is, variable-valency ions.

The main limitation of the method is that it is difficult to produce monodisperse nanoparticle ensembles with a narrow size distribution. This limitation stems from the nature of the process itself, which consists of two stages: ion diffusion and subsequent aggregation. Ion exchange produces polydisperse nanoparticles due to stochastic nucleation and diffusion-controlled growth [56]. Even after the process parameters are optimized, the size distribution remains wide [57]. The depth at which the nanoparticles are located ranges from hundreds of nanometers to several micrometers, depending on the process duration and temperature, as well as on the charge of the diffusing ion. The penetration depth is significantly smaller for multiply charged ions (e.g., Cu^{2+}) than for singly charged ions (Ag^{+}, Na^{+}) [58]. Thermal treatment at temperatures above the glass transition temperature can induce the formation of core–shell structures [59].

The glass composition critically affects ion diffusion and reduction. Adding ions of variable valence, e.g., cerium, iron, or antimony, promotes the reduction of silver during ion exchange and the growth of photoluminescent molecular silver clusters and plasmonic silver nanoparticles [60].

Subjecting glasses doped with halides (such as chlorine or bromine) to $Na^+ \leftrightarrow Ag^+$ ion exchange and thermal treatment produces photo- and thermochromic AgCl or AgBr nanocrystals [61]. The method for forming nanoparticles by ion exchange is reproducible, with only minor deviations in glass composition or process parameters.

When thermal treatment is carried out in air at temperatures below a certain threshold, molecular clusters (smaller than 1 nm) form in the glass; these likely represent silver dimers [62]. The processes occurring can be written as follows [62]:

$$2(\equiv Si - O - Ag^+) \leftrightarrow \equiv Si - O - Si\equiv + O^- + (Ag_2)^+,$$

$$O^- + (Ag_2)^+ \rightarrow 2\,Ag^0 + (1/2)\,O_2.$$

Increasing the annealing time at temperatures below the threshold increases only the number of clusters, not their size. Annealing above the threshold temperature causes the silver nanoparticles to grow in size [62]. The electronic structure of molecular clusters differs substantially from that of metal nanoparticles; in particular, molecular clusters exhibit luminescence in the visible range under UV excitation, making them useful for various sensing applications [63, 64]. However, because of their small size, molecular clusters lack the collective electron oscillation, i.e., the plasmon resonance, that characterizes larger nanoparticles.

An alternative thermal treatment method for forming metal nanoparticles in ion-exchanged glass is annealing of the sample in a reducing hydrogen (H_2) atmosphere. This approach produces a glass-metal nanocomposite inside the glass and a metal nanoisland film on the glass surface, driven by diffusion of metal atoms reduced due to hydrogen penetration into the glass [56]. The reduction of metal ions is described by the reaction [65]:

$$\equiv Si - O - Ag^+ + (1/2)\,H_2 \rightarrow \equiv Si - O - H^+ + Ag^0.$$

Under certain conditions, annealing of ion-exchanged glass in hydrogen can favor nanoparticle formation either on the surface or in the bulk of the glass [66], and can create ordered ensembles of metal nanoparticles on the surface [67].

Ionizing radiation, such as gamma rays, electrons, heavy ions, X-rays, or laser beams, can be used in combination with thermal treatment, or as a substitute for it, to produce silver nanoparticles in ion-exchanged glass [68–70]. Ionizing radiation creates excitons in the dielectric glass matrix, leading to stable defects and changes in the valence state of dopant and impurity ions in the glass.

Gamma irradiation of the dielectric glass matrix produces electron–hole pairs in the matrix. Some of the electrons reduce silver ions to the neutral state [71]:

$$\text{glass} \xrightarrow{\gamma\text{-rays}} h^+ + e^-,$$

$$Ag^+ + e^- \rightarrow Ag^0$$

Subsequent formation of nanoparticles occurs when thermal treatment activates the diffusion of neutral silver atoms in the glass matrix by the following reaction [71] (Fig. 4):

$$nAg^0 \rightarrow Ag_n.$$

The formation of silver nanoparticles manifests as peaks at plasmon resonance wavelengths of 400–420 nm in the optical absorption spectra, and the initially transparent glass turns pale yellow or intense yellow, depending on the volume fraction of nanoparticles. The formation of silver nanoparticles depends on the irradiation dose; gamma irradiation accelerates this rate compared to thermal annealing because it raises the diffusion coefficient of silver in the glass by several orders of magnitude [71, 72].

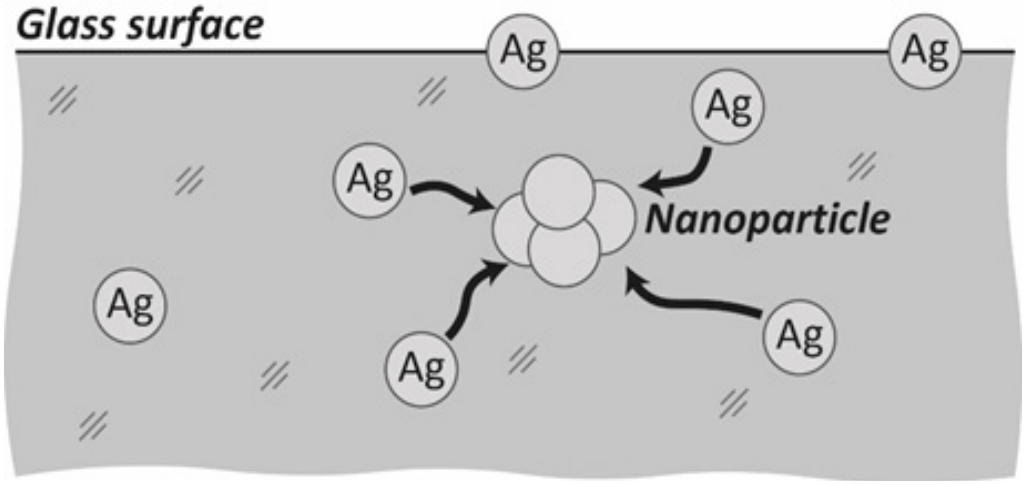


Fig. 4. Schematic of silver nanoparticle formation in glass

Nanoparticles can also be formed in the glass by laser irradiation without additional thermal annealing. Laser irradiation heats the irradiated region of the glass and softens it [73]; in ion-exchanged glass, it breaks the bonds between silver and non-bridging oxygen, after which the silver ions are reduced by electrons from negatively charged non-bridging oxygen atoms [66]. Additionally, laser irradiation at 355 nm produces free electrons in the glass through multiphoton absorption [74]. This in turn leads to reduction of silver ions to the metallic state through trapping of electrons. Supersaturation of the near-surface region of the glass with silver atoms drives phase decomposition and precipitation of metal nanoparticles [75].

Dendritic metal structures in glass. Dendritic (or fractal) nanostructures have attracted considerable interest because of their branched morphology and large surface area, making them attractive for many practical applications, particularly SERS and catalysis [76, 77]. Studies investigated the production of silver dendrites using various methods:

photoreduction in the presence of polyvinyl alcohol [78];

reduction with ascorbic acid in the presence of sodium polyacrylate [79];

galvanic displacement [80] using pulsed sonolectrochemical techniques [81], and others.

However, most methods of wet chemistry for producing dendritic nanostructures rely on surfactants or polymer templates, which complicates the synthesis procedure and the sample purification. In some cases, contamination of the final product is difficult to avoid because undesirable side products form in the reaction medium, rendering the dendrites unsuitable for further use. Therefore, developing template-free methods for synthesizing dendritic nanostructures remains a major challenge.

A promising method for synthesizing silver dendrites, requiring no complex equipment and no wet chemistry, is ion exchange in glass combined with DC poling applied to opposite sides [82] or to the same side [40] of the glass substrate. Fig. 5 shows the structures obtained by this technique.

The mechanism of dendrite formation with a voltage applied to ion-exchanged glass containing metal ions can be described as follows [82].

In the first stage, silver ions drift in an electric field and are reduced at the cathode, forming nuclei of the metallic phase on the glass surface. In the second stage, these nuclei grow by diffusion and form metal islands in contact with the cathode; some of these islands act as secondary cathodes and thus become sources of dendrite growth. Because the silver ions are concentrated in the near-surface region of the glass after ion exchange, dendrite growth occurs mainly in the plane parallel to the sample surface, within the glass. Subsequent etching of the glass in a dilute hydrofluoric acid/ammonium fluoride (HF/NH_4F) solution causes needle-like structures with heights up to ~100 nm and finer dendrite branches to emerge on the glass surface [40]. The resulting metal nanostructures hold promise for applications in catalysis and SERS sensors.

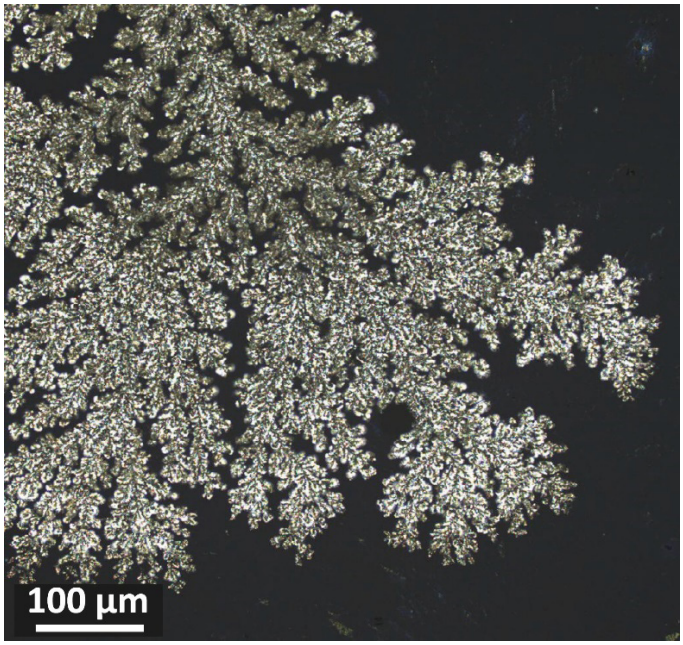


Fig. 5. Optical image of silver dendrites in ion-exchanged glass

Plasmonic nanostructures as substrates for SERS sensors

The interaction between light and noble metal nanostructures is the key to one of the most effective optical detection methods, SERS. Metal nanostructures act as 'hot spots' for the electric field of the incident light wave, due to LSPR [83]. When an analyte molecule lies close to one of these hot spots, its Raman signal increases by several orders of magnitude due to enhancement of the electric field acting on the molecule. This effect gives LSPR-based sensors ultralow detection limits combined with the high selectivity inherent in Raman spectroscopy [84].

Optimization of the substrates is required to develop high-performance SERS sensors. Ideal SERS substrates should provide high signal enhancement, reproducible response, reusability, stable metal nanoparticles, low cost, and a simple fabrication method [85].

Comparison of ion exchange with other fabrication methods for SERS

The main parameters characterizing plasmonic structures used as SERS substrates are the nanoparticle size distribution, the density of hot spots, and the enhancement factor of the Raman signal. Below we briefly compare methods for fabricating metal nanostructures for SERS.

Ion exchange in glass. Nanoparticles obtained by ion exchange in glass have a rather wide polydisperse size distribution, which results from the diffusion-controlled mechanism of their formation [86]. The particle size can be varied from 2 to 250 nm by controlling the ion exchange times and annealing conditions [87, 88]. The RMS deviation in size can reach 30–40% [86]. The hot-spot density is moderate, but it can be varied, for example, by combining ion exchange with thermal poling of the glass [66]. Combining these two methods also makes it possible to form hot spots in a predetermined pattern. The SERS enhancement factor is moderate (up to 10^7 [89]).

Femtosecond laser writing with reduction. In this approach, intense femtosecond laser pulses are focused into the bulk of glass or a precursor solution. Multiphoton absorption triggers local reduction of metal ions (e.g., Ag^+ or Au^{3+}) to the neutral state, producing nanoparticles in the focal region. This method makes it possible to obtain particles of controllable size (typically 20–200 nm) by adjusting the laser power and scanning speed, and forming structures

with designed geometries [91]. The size dispersion is about 10% [92]. The hot-spot density is high. Arrays of dimers or particle chains with gaps below 10 nm can be created, including 3D structures inside the material [92]. The high SERS enhancement factor (up to 10^{10} [93]) is achieved by precisely controlling nanoparticle size and by creating ensembles with tailored characteristics [91, 94].

Nanoimprint lithography with metal deposition. This technique involves applying a polymer resist to the substrate and embossing a pattern (holes or pillars) into it with a mechanical stamp. A metal layer (Ag, Au) is then deposited by thermal or electron-beam evaporation; after the resist is removed, highly ordered metal structures remain on the substrate [95, 96]. The resulting size distribution is extremely narrow (virtually monodisperse). This method also provides the best reproducibility of shape and size (from 10 nm to several micrometers), matching the stamp pattern [97]. Nanoimprint lithography offers the highest hot-spot density and makes it possible to design arrays with field enhancement in precisely defined narrow regions (e.g., in gaps between nanodisks) [97]. This yields high SERS enhancement factors (up to 10^7 [97]) for structures with sub-nanometer gaps.

Chemical etching with metallization. In this approach, a surface (e.g., silicon, copper, or aluminum foil) is treated with a chemical etchant to create roughness. Next, a noble metal layer is deposited on this rough surface chemically or galvanically [98, 99]. The process is difficult to control and often produces dendritic structures. The resulting size distribution is wide and fractal, with sizes ranging from nano- to microscale [100]; the hot-spot density is rather high, providing a sufficiently high SERS enhancement factor (up to 10^8 for metallized mesoporous silicon [101]).

The table compares the methods described above in terms of these three key characteristics.

Table

Comparison of plasmonic structure parameters for different fabrication methods

Method	Size distribution	Hot-spot density	SERS enhancement factor
Ion exchange in glass	Wide (polydisperse) [86]	Medium/low (random distribution) [88]	10^3–10^7 [89, 90]
Femtosecond laser writing + reduction	Narrow (controlled) [91, 92]	High [92]	10^8–10^{10} [93, 102]
Nanoimprint lithography + metal deposition	Very narrow (monodisperse) [10]	Very high (deterministic placement) [96]	10^6–10^7 [97]
Chemical etching + metallization	Wide (random, dendritic) [100]	Very high (sub-nanometer gaps) [14]	10^7–10^8 [101]

Despite the relatively low hot-spot density and moderate SERS enhancement factor compared to other techniques, ion exchange offers several unique advantages making it attractive for SERS substrates:

1. Technological simplicity and scalability. The process is simple and inexpensive, suitable for large-scale production of SERS-active substrates. It requires neither complex lithographic equipment nor high vacuum.

2. Low cost of starting materials. The substrate is multicomponent glass, which is inexpensive and technologically convenient. The ion exchange procedure itself also requires no costly equipment or consumables.

3. Nanoparticles are protected from the environment. Unlike many other methods where nanoparticles lie directly on the surface and are susceptible to oxidation and sulfidation, a thin glass layer covers the metal nanoparticles within the bulk of ion-exchanged glass. This ensures durability and preservation of the substrate without loss of properties, and allows multiple uses by successive etching, which exposes fresh layers with intact nanoparticles for reuse.

4. A high enhancement factor (up to 10^5-10^6) is sufficient for many analytical applications, including biomedical ones.

5. Flexibility and controllability. The process allows varying both composition and structure of the nanoparticles. For example, sequentially introducing different metal ions (copper and silver) and controlling the annealing temperature makes it possible to form core–shell nanostructures and nanostructures of mixed composition whose plasmon resonance position depends on the metal concentration ratio. Combining ion exchange with laser irradiation also allows writing the structures of a required shape.

6. Integration into optical circuits. Forming nanoparticles in glass is ideal for developing integrated sensor chips that combine optical waveguides and microfluidics.

7. Well-studied and amenable to modeling. The processes of ion exchange and subsequent nanoparticle formation are well understood and can be mathematically modeled, allowing prediction of the properties of the final nanostructures.

Thus, while the method has numerous advantages, the most important are the unique combination of simplicity and cost-effectiveness, and the potential for improved durability of SERS substrates due to natural protection of the nanoparticles by the glass matrix. This makes the technology particularly attractive for practical and stable sensors suitable even for field conditions. Although the method has some limitations in controlling the nanoparticle size dispersion, these do not pose a serious obstacle to its application.

Silver nanoparticles provide the best SERS enhancement among all noble metal nanostructures, although they are prone to oxidation and sulfidation.

Given the parameters of substrates fabricated by different methods (see above), SERS substrates based on ion-exchanged glasses with silver nanostructures appear the most promising for the following reasons:

silver nanostructures provide high SERS signal enhancement;

silver nanoisland films can be removed from the surface after use, and new nanoparticles can be formed on the surface by simply heating the glass substrate with silver ions; in other words, the substrate is reusable [38];

when silver nanostructures lie in the bulk of the glass, they remain undamaged; they can be brought to the surface directly before use by etching the glass [40];

the method is simple, low-cost, and the results are highly reproducible.

Substance detection with SERS on substrates with silver nanostructures formed by ion exchange

Ref. [39] reports on SERS enhancement provided by silver nanoisland films formed on the glass surface by ion exchange and thermal annealing, as well as the effect from the arrangement of hot spots in ensembles of self-assembled silver nanoislands. Raman spectra were measured for an aqueous solution of 1,2-di(4-pyridyl)ethylene (BPE) at a concentration of 10^{-3} M under non-resonant excitation. Comparison of SERS characteristics of nanoisland films at different growth stages showed that enhancement dominated at the nanoisland coalescence stage due to increased electric field in closely spaced groups

of nanoislands. A linear dependence between the SERS signal and the surface density of nanoislands was observed at earlier growth stages. The maximum enhancement occurred at the onset of coalescence.

Silver nanoisland films formed on the surface of ion-exchanged glass by thermal annealing are stable in biocompatible buffers, which opens new opportunities for applications in SERS diagnostics of biological objects dissolved in buffers. It was found in [41] that silver nanoisland films are stable in phosphate-buffered saline (PBS), one of the most common buffers in biological research, in PBS diluted 1:100 with water, in PBS-based buffer where sodium chloride (NaCl) is replaced by the same amount of sodium perchlorate ($NaClO_4$), and in acidic phosphate buffer. The nanoisland films provided a good SERS signal from a BPE monolayer (concentration of 10^{-4} M) after exposure to PBS, making them promising for biosensor applications.

SERS has been successfully applied to diagnose amino acids and proteins. SERS spectra of D-alanine and DL-serine were observed in [103] using substrates prepared from silver nanoisland films immersed in standard PBS solutions with millimolar concentrations of these amino acids. The resulting spectra differ significantly in peak intensity and position from the Raman spectra of bulk solutions and from SERS spectra of crystallites of the corresponding amino acids on substrates not immersed in buffer, as well as from Raman spectra on pure glass. This effect is presumably due to electrostatic orientation of zwitterionic amino acids by the silver nanoparticles, which acquire a negative charge due to desorption of Ag(I) chloride in the solution.

Thus, despite sufficient signal enhancement from metal nanoislands, the interaction of the amino acids with the metallic substrate should be taken into account in the detection of amino acids in various biocompatible buffers.

A comparative study [104] evaluated SERS and enzyme-linked immunosorbent assay (ELISA) for detecting SARS-CoV-2 virus nucleoproteins. SERS substrates were silver nanoisland films on the surface of ion-exchanged glass. The SERS spectra can be used to distinguish between the features of antigens, antibodies, and the features indicating the binding between them. The SERS method exhibited a much lower detection limit (1 pg/mL) compared to ELISA (approximately 50–100 pg/mL), and significantly reduced the analysis time: ELISA requires about two hours, whereas SERS measurements take only a few seconds.

The branched structure of dendrites makes them highly attractive for sensing applications. Several studies explored the use of dendrites for SERS diagnostics [105–107]. For example, dendritic nanostructures formed by electrolysis in silver-enriched glass for SERS detection of low concentrations of hazardous substances were analyzed in [108]. The detection limit for the thiram pesticide corresponded to a surface concentration of $4.6 \cdot 10^{-10}$ g/mm^2 (a monolayer). The Raman enhancement factor provided by the dendrites was $4 \cdot 10^5$.

Silver is well known for its antibacterial properties, making it valuable for medicine and pharmacology [109]. However, the toxic effects of silver on living cells can hinder the use of silver nanoparticle substrates for SERS analysis of biological objects. Nanoparticles of other metals, particularly gold, can be used in such cases. Bimetallic nanoparticles (e.g., silver-gold) are also promising: they could combine the advantages of each metal, i.e., the high LSPR figure of merit provided by silver, and the chemical and biological inertness of gold for biological diagnostics.

Ion exchange and back-diffusion were used in [110] to produce ensembles of biocompatible Au–Ag alloy nanoparticles with varying silver contents, suitable for use as SERS substrates. The bimetallic Au–Ag nanoparticles were grown by silver-to-sodium ion exchange in silicate glass, followed by deposition of a depercolated gold film on the sample surface and subsequent heat treatment. Varying the heat treatment regime made it possible to control the fraction of silver in the nanoparticles and adjust the spectral position of their LSPR between the resonances of gold and silver nanoparticles.

Cell viability assays on pure glass and on glass substrates with nanoparticles of different compositions showed that cells grown on control slides, on slides with gold nanoparticles, and on slides with nanoparticles containing 40–50 wt.% silver exhibited similar viability within the reference limits. In contrast, substrates with pure silver nanoparticles showed a noticeable decrease in viability and approximately 100% loss of functionality/adhesion [110].

Future directions

The versatility of ion exchange in glass opens new prospects for its application in plasmonics and photonics; some of these are already under way, while others are just beginning to be explored.

Integrated photonic platform. Ion exchange in glass is one of the primary technological platforms for producing low-cost, high-performance planar waveguides [111]. The process, which typically involves exchanging K^+ or Ag^+ ions for Na^+ ions in the glass, induces a local increase in the refractive index, forming the basis for light guiding. This technology has evolved over the past 30 years, enabling a wide range of devices for telecommunications, optical information processing, and sensing [112]. Waveguide structures include both passive and active components. Passive devices, such as directional couplers, gratings, and optical splitters, have been successfully constructed many times [113]. Active devices, such as integrated optical amplifiers and lasers, are fabricated by doping the glass with rare-earth ions (e.g., Nd^{3+}), with subsequent formation of waveguides by ion exchange [114]. One of the most interesting directions is the application of this technology to quantum photonics. In the future, ion-exchanged waveguides could be used to implement quantum circuits for cryptography and quantum processing, integrating them with ion-exchanged glass lasers on a single chip. This positions ion-exchanged glass as a promising platform for quantum integrated optics [115].

Flexible glass-based photonics. The concept of "flexible photonics" is a new stage for ion-exchanged glass devices [116]. This direction is seen as an alternative to polymer-based flexible photonics, which has its drawbacks: a limited range of refractive indices, NIR absorption, and low chemical stability. Although glass is traditionally considered a rigid material, using thin ion-exchanged layers makes it possible to fabricate photonic components on flexible glass substrates. The idea is that the ion exchange technology can be adapted to create flexible photonic structures based on commercial glass, simultaneously retaining low optical losses and chemical stability.

Ultra-thin sheets of commercial glass (e.g., Willow Glass from Corning, less than 100 μm thick [117]) with high surface quality and paper-like flexibility could be used for flexible photonic devices. Ion exchange has been proposed for strengthening these ultra-thin glasses. For alkali-containing glasses, conventional ion exchange (e.g., $Na^+ \leftrightarrow K^+$) generates compressive stress on the surface that counteracts microcracks under bending, so that smaller bending radii can be achieved without fracture. This paves the way for fully glass-based flexible devices for portable electronics.

Photonic devices with noble metal ions/nanoparticles. Another benefit offered by growing metal nanoparticles and clusters via ion exchange lies in creating low-cost photonic systems with improved performance for a wide range of applications. In particular, introducing noble metal ions (Ag, Au) by ion exchange into the bulk or onto the surface of microcavities (microspheres, microdisks) with whispering gallery modes (WGM), made of multicomponent glasses, can impart new properties to these devices. The luminescence efficiency and emission of the rare-earth ions can be enhanced by nucleating and growing noble-metal nanostructures (dimer clusters and/or metal nanoparticles several nanometers in size) inside the gain medium of a resonator made of glass doped with rare-earth ions, after by ion exchange and thermal post-treatment. This enhancement arises from surface plasmon resonance and/or energy transfer mechanisms, as observed for planar substrates [118, 119]. Moreover, signal enhancement at the converted frequency occurs within the same microresonator, which, supporting high optical energy density, allows photons to interact repeatedly with both the rare-earth ions and the metal nanostructures, typically located a few tens/hundreds of nanometers below the resonator surface, within the range of the waveguide modes. If these nanostructures are small plasmonic nanoparticles (e.g., a few nanometers in size), a combination of two resonant phenomena, waveguide modes of the microresonator and the plasmonic response of the nanoparticles, could lead to further significant enhancement of the signal.

The presence of noble-metal nanoparticles embedded in the waveguide cavity in glass could also enhance the nonlinear optical response of the medium due to plasmonic effects. For example, silver nanoparticles, with their high third-order nonlinearity, are prime candidates for developing all-optical signal processing devices, such as efficient optical limiters that require materials with a high nonlinear refractive index $n^{(2)}$ [120].

Quantum and coherent plasmonics. Research efforts in this field are shifting from simple field enhancement by plasmonic nanoparticles to exploiting quantum phenomena such as phase coherence and strong coupling between plasmons and other quasiparticles (excitons, electron-hole pairs). This opens new avenues for creating elements for quantum computers based on graphene plasmons and new types of nanophotonic devices [121]. Quantum plasmonics makes it possible to fabricate single-photon sources, transistors via photon blockade, and thresholdless nanolasers (spasers). Hybrid plasmonic-photonic circuits support on-chip generation, manipulation, and detection of single plasmons [122, 123].

Conclusion

Nanostructures that exhibit plasmon resonances have a wide range of practical applications, from visualizing glucose concentrations in blood to detecting toxic substances and diagnosing viral infections. The performance of plasmon-enhanced sensors depends directly on the geometry of the plasmonic structures and on the methods used to fabricate them. Importantly, the parameters of the nanostructures can be readily adjusted to different applications by choosing the material and the morphology of the structure. The distinct benefits of fabricating plasmonic nanostructures in glasses via ion exchange are their simplicity and low cost. All these factors provide the flexibility necessary for developing various sensors based on plasmonic field enhancement, opening up broad opportunities for using such devices. Further advances in plasmonics will enable even more efficient signal enhancement and imaging of analytes, as well as improved designs for new devices detecting chemical and biological compounds via fluorescence and SERS.

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