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Fabry-Perot interference pattern scattered by a sub-monolayer array of nanoparticles

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Abstract

Understanding scattering of visible and infrared photons from nanomaterials and nanostructured materials is increasingly important for imaging, thermal management, and detection, and has implications for other parts of the electromagnetic spectrum (e.g., x-ray scattering and radar). New, interesting reports of photon scattering as a diagnostic probe, from inelastic x-ray scattering and interference to ‘nano-FTIR’ microscopy using infrared photons, have been published and are under active investigation in laboratories around the world. Here, we report, for the first time to our best knowledge, the experimental discovery of a Fabry–Perot interference pattern that is scattered by the sub-monolayer array of plasmonic Ag nanoparticles, and confirm it analytically and with rigorous numerical FDTD simulations.

1. Introduction

Almost 100 years ago, Compton scattering of high-energy photons from atomic electrons was observed, which enabled better understanding of the photonic nature of light. X-rays, with wavelengths comparable to inter-atomic distances, are now routinely used in scattering mode to determine basic properties of materials, and recently interference effects have been observed and explained in resonant inelastic x-ray scattering to better understand excited electronic states, analogously to elastic scattering’s uncovering information about the ground state [1].

In contrast, scattering of visible and infrared photons has generally been under-researched in spectroscopy, compared to specular reflectivity, direct transmission, and absorption, because it is challenging to determine the scattering profile of a flux of visible or infrared photons incident on a scattering surface, especially if the features of the surface are sub-wavelength in dimension. Nevertheless, scattering of visible and infrared photons by nanostructured materials is an important optical effect that reveals critical information about optoelectronic properties of the materials and surface. New interest in visible and infrared photon scattering has arisen, connected with the expanding field of plasmonics. For example, the interference of plasmonic and photonic modes of an L-shaped nanoslit have been observed using Mach–Zehnder interferometry in the visible regime [2]. In the visible and infrared regimes, where photons interact with materials strongly, scattering effects can be complex and depend strongly on the dimensions and features of the scattering surfaces. Nevertheless, scattering of photons is important for many important reasons: increasing the absorption of thin films, enhancing color through scattering instead of absorption, nanoscopic FTIR [3], infrared imaging, painted objects like vehicles and buildings where angle-dependent color is important to maintain, cloaking in the diffusive limit [4], and inelastic Raman scattering to do chemical analysis.
2. Background

In surface-enhanced Raman scattering (SERS), employed to identify the presence of unknown molecules, the energy of the incident photon is shifted most efficiently when there is a nanoscopic electromagnetic ‘field enhancement’ in the vicinity of the molecule. The Raman scattering response goes as the 4th power of the incident high-frequency electric field, and is greatly amplified by the field enhancement that accompanies scattering resonances, especially plasmonic resonances. Raman scattering has obtained large enhancements from roughened plasmonic surfaces, and from arrays of plasmonic nanoparticles [5].

Field enhancement is illustrated in figure 1, which displays the scattering versus absorption cross-sections in the visible and near-infrared regime, as a function of wavelength, for Ag nanospheres with three different diameters (50 nm, 120 nm, and 300 nm). The smallest nanosphere is well into the Rayleigh regime, since the size factor $2 \pi r \lambda^{-1}$ ($n$ and $\lambda$ are the index of refraction, respectively, and $r$ is the radius) is generally less than unity for visible wavelengths of 300 nm to 1000 nm [6]. However, plasmonic resonances around 350 nm are still noticeable, due to the plasmonic properties of the Ag metal. The larger nanospheres, especially the 300 nm nanosphere, are in the mixed Mie-to-Rayleigh crossover regime, with size factor of unity or larger.

The Mie regime has much higher scattering, even in the presence of larger absorption. Plasmonic effects strengthen the scattering and absorption even further, and are present (especially absorption) even in the weaker-scattering Rayleigh regime. It is interesting to notice how the scattering cross-section dominates the absorption cross-section for the larger nanoparticles, which approach and enter the Mie regime.

Plasmonic nanoparticles exhibit scattering and absorption that can be designed by choice of nanoparticle geometry, size, shape, and material. Measuring the light scattering from just one particle is difficult and not also relevant for applications. Instead, we study a metasurface of these nanoparticles, in a sub-monolayer array, defining a ‘monolayer’ as a close-packed array of nanoparticles; at most, one nanoparticle thick. Plasmonic Ag nanoparticles were cast into sub-monolayer arrays and coated onto smooth, well-characterized thin films of amorphous silicon (a-Si), deposited on IR-transparent BK7 glass substrates, in order to investigate the effect of

![Figure 1. Scattering and absorption cross-sections (Q) of Ag nanospheres, having diameters 50 nm, 120 nm, and 300 nm (clockwise starting on the upper left). Q is the cross-section divided by the physical cross-section $\pi r^2$.](image)
the nanoparticles on a-Si thin film absorption [7]. Recent studies have shown the advantages of engineered sub-monolayer arrays of plasmonic particles, and even three-dimensional nanoparticle supercrystals, for SERS detection [8, 9].

These smooth thin films showed expected Fabry–Perot resonances, with reflectivity minima (due to a phase shift arising from the high-to-low index of refraction transition at the bottom of the thin film) given by

\[ n_f \lambda = 2nd \]

where \( n \) = index of refraction and \( n_f \) is an integer representing the excited Fabry–Perot resonance (1, 2, 3…). The Fabry–Perot thin film cavity’s reflectivity maxima are given by

\[ \frac{n_f \lambda}{\lambda - n_f \lambda} = 2nd \]

The Fabry–Perot transmission maxima are given by

\[ \cos \left( \frac{n_f \lambda}{2d} \right) = \frac{1}{n_f} \]

The perpendicular wavevectors (wavevectors in the z-direction) for maximum specular reflectivity are given by

\[ k_z = \frac{\lambda}{2(n_Si - 1/2)d} \]

This can be seen by the fact that the z-wavevector within the a-Si \( k_{z,m} = nk_z \), when multiplied by twice the film thickness \( d \), must equal a half-integer for maximum reflectivity (since there is an additional phase shift of \( \pi \) at the a-Si/glass interface). Because the index of the aSi is larger than that of the glass substrate, when \( nd = \frac{m_f \lambda}{4} \), there is high reflectivity, not an antireflective coating, which is the case for when the indices increase monotonically with progressively deeper layers into the substrate. These trends are illustrated in figure 2.

### 3. Experimental

Silver (Ag) is one of the best plasmonic metals, because of its low loss. Ag nanoparticles were synthesized in the laboratory for this investigation. There were two types: near-spherical icosahedral single-crystal, surfactant-free Ag nanoparticles (SNPs), with diameters of 50 nm, 120 nm, and 300 nm, obtained from Clemson University (described in [10]; a representative scanning electron microscopy (SEM) image is shown in figure 3), and silver nanoprisms (‘NPs’), synthesized following a procedure by Yang *et al* [11] to form ‘large’ and ‘small’ nanoprisms. The ‘large’ nanoprisms were more irregularly shaped flat particles with the same thickness as the smaller particles with much larger particle sizes of \( >500 \) nm (right inset of figure 4). These irregular shapes were also observed by Yang [11]. Two different size fractions of nanoprisms, labeled ‘small’ and ‘large’, were separated and measured by UV–vis-NIR extinction spectra, as shown in figure 4. The suspensions of small particles were purplish red in color, while the large particles were grey and appeared milky, indicating the presence of stronger photon scattering in the latter suspension. The ‘small’ nanoprisms were mainly flat and disk-shaped, with diameters of less than 100 nm and a thickness of 35 nm, although there were some larger triangular shaped nanoprisms with edge lengths of approximately 150 nm (left inset of figure 4).

The SNPs and NPs were formed into sub-monolayers, following the techniques in [10], which affixed the nanoparticles to the substrates, took them through a process of sequential mixing steps to replace the water with
polymethyl methacrylate (PMMA), while keeping the nanoparticles, adhered to the substrate and wet in order to prevent aggregation. Finally, the substrates were placed on a spincoater and spun to obtain a nanoparticle-bearing PMMA coating, whose final thickness was roughly equal to the 60 nm in the case of the 120 nm diameter SNPs. Because the NPs were flatter, they were completely covered by a thickness of roughly 120 nm PMMA. Following the techniques described in [10], reported in more detail in [12] with additional information on fabrication, resulting samples had sub-monolayer arrays of Ag nanoparticles with a rough average quasi-periodicity (see figure 3) on thin films of amorphous silicon ('a-Si') 100 nm, 200 nm, 300 nm, 400 nm, and 500 nm in thickness on IR-transparent glass. While these sub-monolayer arrays were not perfectly periodic, they could be simply fabricated over areas larger than a few square centimeters in size. Recently, periodic arrays have been fabricated to ∼0.5 cm² in area [8].

The optical properties of the resulting sub-monolayer arrays of Ag nanoparticles on thin a-Si films were completely characterized spectrophotometrically [12]. Fabry–Perot maxima in total reflectivity were observed as described in the Background section, at wavelengths approximately equal to 4nd/(mz − ½) ∼ 7000 nm (too long a wavelength for the spectrophotometer), 2333 nm, 1400 nm, 1000 nm, 778 nm, and 636 nm, corresponding to mz = 1, 2, 3, 4, 5, 6, respectively. These are shown as dashed lines in figure 5, for three different samples.

We observed roughly 5 peaks in the diffuse reflectance that matched (same wavelengths) the 5 Fabry–Perot peaks in the specular reflectivity of 500 nm aSi (and other thicknesses), in samples without nanoparticles. These
5 peaks were immediately recognizable as the Fabry–Perot cavity (thin film) resonances described in the paragraph above. In samples with nanoparticles (NPs and SNPs), we noticed an interesting effect: the 5 measured peaks in the integrated backscattering (diffuse reflectivity)’s Fabry–Perot interference pattern were shifted, towards shorter wavelengths, with respect to the spectrum of the simple 500 nm thick aSi film. The backscattering spectrum can be thought of as a ’scattered interference spectrum’, which is shifted with respect to the specular interference spectrum due to an average in-plane k-vector, imparted by the nanoparticles on top of the thin film.

4. Analysis and discussion

The spectral shift in the scattered Fabry–Perot interference pattern towards shorter wavelengths (‘blue-shift’), makes intuitive sense as a combination of single-particle scattering and a diffraction effect from a quasi-periodic array. Together, these two effects impart momenta to the outgoing photon, producing a diffuse and non-specular reflectivity. If the array were truly periodic, there would be in-plane diffraction orders resulting in narrow, discrete resonances, qualitatively similar to the backscattering spectrum in figure 5(Sb).

The measured Fabry–Perot interference pattern in the diffuse reflectance can be thought of as being scattered from the sub-monolayer arrays of plasmonic Ag nanoparticles. In order to maintain the same perpendicular wavevector component within the a-Si that gives a maximum reflectivity from the Fabry–Perot cavity, $\bar{k}_{\text{perp}}$, the photon energy must be higher, and therefore the scattered (not specular) interference pattern is blue-shifted to higher photon energies.

If the samples behaved like a uniform medium, say with the same effective index of refraction as the ’rule of mixtures’ averaged sum of indices of Ag nanoparticles and PMMA host, there would be no diffuse reflectance, so explanations for the shift in the scattered interference pattern based solely on an ’effective medium’ theory fail to explain the observed effect.

Because the sample was not perfectly periodic, characterized instead by a mean spacing between particles (e.g. ’semi-periodic’), there was quantitative variation from sample to sample, although the spectral dependence averaged to be the same as in figure 5. The 120 nm SNPs showed similar results to the scattered Fabry–Perot interference pattern Sb in figure 5. As shown in [12], 50 nm SNPs were too small to scatter substantially and produced results similar to the control samples, while 300 nm SNPs scattered considerably, but also showed similarly a shift in the scattered interference pattern, from the specular interference pattern.

We developed a brief analytical model, to explain more quantitatively the shift in the scattered Fabry–Perot interference pattern, for normal incidence experiments. It explains how the blue-shift arises from the conservation of photon energy. Fabry–Perot peak wavelengths, given by $(m_{n}-1/2)\lambda = 2nd$ as remarked earlier, must be at smaller $\lambda$, because unlike the specularly reflected photons, the scattered photons have a component of the photon momentum lying in the plane of the sample (along the x-direction, with no loss in generality due to
the square array). The blue-shift is due to the rise of an in-plane wavevector \( \vec{k}_z \) from diffraction from a sub-monolayer of particles with mean spacing \( a \) (‘d-spacing’), which for the case of normal incidence is given by

\[
\vec{k}_z = \bar{q}_z = \frac{m_z}{a_z^2}
\]

(1)

From conservation of photon energy (elastic scattering):

\[
\vec{k}_{in,z} = \frac{n^2}{\lambda^2} - \vec{k}_x - \vec{k}_y
\]

(2)

In other words, the \( z \) component of momentum inside the material is given by the photon energy, subtracting the energy component due to in-plane momentum. We therefore obtain a formula for the wavelengths having the maximum reflectivity:

\[
\lambda = \frac{nd}{\sqrt{\left((m_z - 1/2)^2 + (m_z + m_x^2) d^2\right)/4 - \vec{k}_{in,z}^2}}
\]

(3)

where we have used \( \vec{k}_{in,z} = (m_z - 1/2) / (2d) \).

These formulae are analogous to equation (2) in [13], which rewritten in our notation is:

\[
\vec{k}_{in,||} = \frac{n^2}{\lambda^2} - \vec{k}_{in,z}
\]

where for [13], \( \vec{k}_{in,z} \) would be \( \frac{m_z}{a_z^2} \), and \( L \) is the thickness of the GaN layer described in [13].

In the simple analytic model, the periodicity \( a \) of the square lattice defines the cutoff wavelength of the scattering, since backscattering is cut off when the photon, having wavelength \( \lambda \), receives maximum \( x \)-momentum transfer \( \vec{k}_x = 1/\lambda \), corresponding to the photon momentum’s being entirely redirected parallel to the interface (grazing refraction upon exiting the material).

The expected blue-shift in the scattered interference pattern, at \( m_x = 2 \), is only \( \sim 60 \) nm for a period of \( 2900 \) nm \((a = 2900 \) nm\). For smaller periods \((a)\), the scattered \( m_x = 2 \) mode is forbidden, once the shifted \( m_x = 2 \) peak occurs at a longer wavelength than \( a \), because in this simple analytical model, there is no scattering for \( \lambda > a \). Smaller values of \( a \) force \( k_z \) to be too small, to support \( m_x = 2 \). Higher order Fabry–Perot modes, occurring at \( \lambda < a \), are present. However, clearly this simple analytical model cannot fully explain the observed experimental scattered Fabry–Perot interference pattern; in particular, there is no firm cutoff of the scattering. We resorted to modeling the system numerically using finite-difference time domain (FDTD) techniques.

FDTD modeling showed that the blue-shift in the interference pattern of the scattered light, including the \( m_x = 2 \) and \( m_z = 3 \) peaks that the analytic model cannot fully explain, is due in first order to the scattering from the Ag nanoparticle, which itself (e.g., at any lattice spacing) imparts a range of \( x \)-momenta and \( y \)-momenta to the scattered photons. Different nanoparticles produce slightly different in-plane momentum shifts, so the blue-shift is slightly dependent on the particle. The average spacing of the particles imparts an average momentum component as well, but it is a second-order effect, primarily manifesting itself as additional scattering amplitude that appears at key wavelengths that diffract more orders (see paragraph below).

FDTD simulations of the integrated backscattering from periodic arrays are shown in figure 6. The Ag nanoparticle, atop the 500 nm silicon film, causes the interference pattern to shift to the blue part of the spectrum. In the simulations, the periodicity \( a \) of the square lattice defines the cutoff wavelength of the backscattering, and also determines the back-diffracted orders: 1 (just specular \((0, 0)\)) for \( \lambda > a \), 5 at \( \lambda = a \) \((0, 0)\) and 4 1st order diffraction peaks, 9 at \( \lambda = \frac{a}{\sqrt{2}} \) \((+/−1, +/−1)\) orders appear in the backscattered spectrum), 13 at \( \lambda = \frac{a}{2} \) \((2, 0)\) orders appear in the backscattered spectrum), etc. However, these effects are 2nd order, while to first order the individual nanoparticle dominates the backscattering spectra, according to the FDTD simulations. A series of simulations, varying both \( a \) (the periodicity) and the nanoparticle shape and size, was carried out. As described above, the FDTD simulation’s periodicity played a 2nd order role in defining the peak spectrum; changing \( a \) did not produce as large a change to Sb as did the changing the nanoparticle itself or the thickness of the underlying Si thin film. The silicon film thickness was also varied by a small amount in the simulations. Clustering of nanoparticles was treated by simulating two paired nanoparticles that were ‘nearest neighbors’ having separations less than 100 nm; clustering was also expected to reduce the mean separation of the array of nanoparticles, and would therefore be represented by smaller values of \( a \) in the simulations. In the future, an array of samples with different periodicities could be analyzed experimentally and quantitatively, in order to verify that the individual nanoparticle is the most important component to the scattered interference pattern from these sub-monolayer arrays.
Generally, nano-cylinders with larger aspect ratios backscatter in a more broadband way, than spheres, and were simulated to represent the NPs. The diameters of the nano-cylinders were varied from 300 nm to 600 nm. The simulated scattering can be observed using cross-sectional pictures of the electric field around the NP and inside the a-Si thin film; see figure 7. Larger diameter (600 nm) cylinders, in particular, show a clear backscattered lobe at higher frequencies, in simulations. When reflectivity is maximized, especially in the near infrared, there is generally little backscattering, in part since the exciting field does not reach the nanoparticle. At higher frequencies, in the mid-visible range, there is much more overlap between scattered modes and Fabry–Perot reflectivity maxima (see figure 8 for the latter), and the situation is complicated by increased silicon absorption in that range. A conformal mesh and 500 frequency points were used in the FDTD simulations.

Figure 6. Experimental integrated backscattering $S_b$, specular reflectivity $R_{std}$, and FDTD simulation of backscattering $S_b$ (blue-colored curve) from two 600 nm diameter nano-cylinders that are 35 nm tall, and separated (edge-to-edge distance) by 30 nm. These 600 nm diameter nano-cylinders represented the 'large' NPs, arranged in a square array with $a = 2.7 \, \mu m$. The amplitude of $S_b$ was scaled by a factor of 10 to fit it on the same graph. A simulation of a single 600 nm nano-cylinder with the same dimensions gave a very similar spectrum, except that the peaks were narrower, and the amplitude of $S_b$ was lower (less particles). Note that the backscattering Fabry–Perot interference spectrum has shifted in the same way as the experimental $S_b$ spectrum with respect to the $R_{std}$ spectrum. Small kinks in the simulated total backscattering at 1.35 $\mu m$ and 1.91 $\mu m$ are from the contribution of additional orders for smaller wavelengths than those cutoff dimensions. In these simulations, the polarization lay along the axis of the paired nano-cylinders; the orthogonal polarization gave a similar scattered Fabry–Perot interference pattern. The simulated Si film thickness was 500 nm.

Figure 7. (left). Electric field profile from a 600 nm diameter, 90 nm high (35 nm high is similar) scattering nano-cylinder (at $z = 0$, extending from $x = -0.3$ to $x = 0.3$) excited by 1220 nm wavelength, a peak in the diffracted intensity and having relatively low specular reflectivity. Backwards and forward scattering are apparent. The striped blue-colored region represents the thin a-Si film, extending from $z = 0$ to $z = -0.5$, with glass underneath it.
As shown in Figure 3 for the case of the SNPs, some of the nanoparticles cluster, resulting in a pair of ‘nearest neighbors’, and those pairs of nearest neighbors were modeled using FDTD as well, with separations as little as 30 nm, separated by the lattice spacing $a$. These simulations resulted in larger scattering amplitudes (larger Sb), but little shift in the backscattering spectrum from that of a single particle with periodicity $a$, so that the paired clustering appeared to add incoherently to the total scattered amplitude. Paired clustering (with inter-particle separations much less than the mean), while not shifting the scattered Fabry–Perot spectrum (Sb), broadened the peaks of Sb beyond what was simulated for a single nano-cylinder with a fixed periodicity. Given that paired clustering of some fraction of NPs occurred, the periodicity $a$ can be thought of as representing the mean separations of all the NPs, including both the small near-neighbor separation of clustered pairs and larger separations for sparsely distributed NPs. Figure 6 represented the best balance between representing the average periodicity and matching experiment. Given that the simulations treat a periodic array, and the experiment represents an average over a mean periodicity, the agreement between experiment and simulations in Figures 6 and 8 is good.

5. Conclusions

We have explored the optical properties of a sub-monolayer array of plasmonic Ag nanoparticles atop an a-Si thin film. We have observed and reported, for the first time in our best knowledge, the experimental discovery of an interesting shift of the visible/near-infrared Fabry–Perot interference pattern, with respect to the specular Fabry–Perot reflectivity spectrum, scattered by the sub-monolayer of Ag nanoparticles. Our analytical model provides quantitative clues on how the scattered interference spectrum is shifted with respect to the specular reflectivity, and numerical simulations confirm the blue-shift of the scattered interference pattern. Understanding visible and infrared scattering from nanomaterials and nanostructured materials is increasingly important, and will help guide scattering experiments in other parts of the electromagnetic spectrum such as x-ray diffraction and radar, as well as applications such as nano-FTIR nanoscopic light scattering probes.

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Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

Ethical statement

The authors declare no known conflicts of interest.

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