

Geometry-dependent localization of surface plasmons on random gold nanoparticle assemblies

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Assemblies of plasmonic nanoparticles (NPs) support hybridized modes of localized surface plasmons (LSPs), which delocalize in geometrically well-ordered arrangements. Here, the hybridization behavior of LSPs in geometrically completely disordered two-dimensional arrangements of Au NPs fabricated by an e-beam synthesis method is studied. Employing electron energy loss spectroscopy in a scanning transmission electron microscope and numerical simulations, the disorder-driven spatial and spectral localization of the coupled LSP modes that depends on the NP thickness is revealed. Below a NP thickness of 0.4 nm, localization increases toward higher hybridized LSP mode energies. In comparison, above 10 nm thickness, a decrease of localization toward higher mode energies is observed. In the intermediate thickness regime, a transition of the energy dependence of the localization between the two limiting cases, exhibiting a mode energy with minimal localization, is observed. It is shown that this behavior is mainly driven by the energy and thickness dependence of the polarizability of the individual NPs.

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I. INTRODUCTION

Strongly disordered micro- and nanostructures of (noble) metals, such as random assemblies of gold nanoparticles (NPs) or thin random gold networks, are currently used for various applications such as electromechanical gas and vapor sensors [1,2] or flexible electrodes, e.g., for neural implants [3] or monitoring of blood vessels [4]. The latter exploits the presence or absence of strongly localized surface plasmon (LSP) resonances in a particular frequency band depending on the composition and geometry of the nanostructure. Localization contrasts with the formation of delocalized modes in periodic structures (such as lattice resonances and quasicontinuous plasmon bands), which exhibit high quality factors, strong dispersion, and ballistic transport, among other characteristics [5–8]. Both phenomena originate from the coupling and hybridization of LSP resonances via electromagnetic interaction [6]. Localization of surface plasmons in strongly coupling random networks and NP assemblies has been intensely studied by various groups [9–12], establishing the fundamental role of the disorder in the observed

spatial localization and spectral distribution. Recently, we have demonstrated the localization of surface plasmons in ultrathin random gold networks, which increased significantly in an energy interval from 0.3 to 1.6 eV, and the absence of hybridized modes beyond [9]. In order to model the intriguing localization behavior, we described the random network as a random assembly of strongly oblate Au NPs (i.e., the inverted system in the sense that holes of the network correspond to NPs) by exploiting Babinet's principle [13–15], which was then amenable to numerical solutions. Notwithstanding obtaining good agreement between experiment and simulation, the question of how the thickness and other geometric parameters affected the localization behavior remained open.

In this work, we study the direct system, i.e., a random two-dimensional (2D) assembly of Au NPs fabricated by a recently developed synthesis route utilizing the electron beam within a scanning electron microscope (SEM) [16]. This fabrication method has the advantage of synthesizing highly disordered assemblies of NPs of a large size distribution ranging from 2 to 100 nm. We probe the spectral properties as well as spatial localization by spatially resolved electron energy loss spectroscopy (EELS) in the scanning transmission electron microscope (STEM) (see Fig. 1 for a scheme). Numerical simulations have been performed using a self-consistent dipole model. We find a localization behavior that decreases toward higher energies in contrast to the ultrathin Au networks, where it increased at higher energies [9]. The numerical simulations based on a discrete dipole approximation show that this qualitatively different behavior can be traced back to the difference in average thickness.

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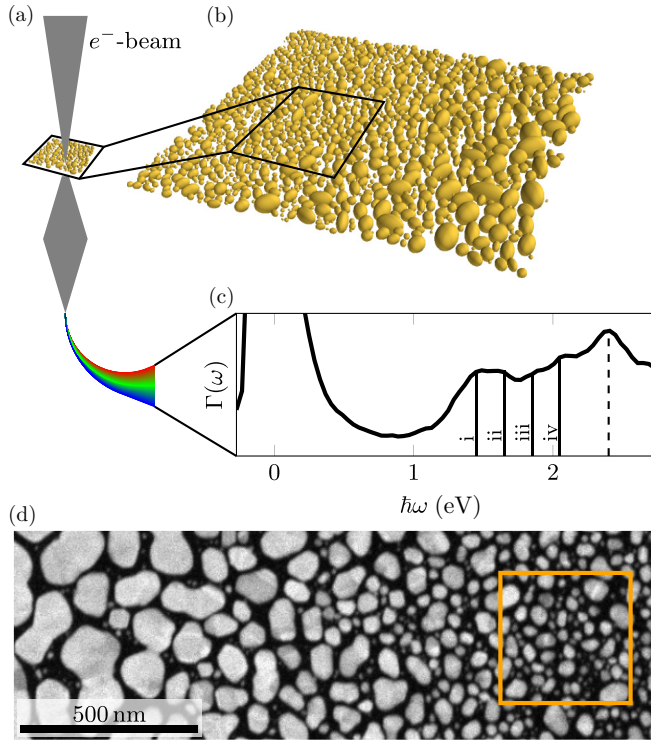


FIG. 1. Plasmon mapping using STEM-EELS of randomly distributed gold NPs. (a) Scheme of a focused electron beam (gray) that is scanned over the sample (b), where the gold NPs are simplified as ellipsoids. (c) An EEL spectrum is collected at each scanning point within the black box region in panel (b). (d) HAADF STEM image of gold NP assembly depicted in panel (b) containing the orange indicated region that has been mapped by STEM-EELS yielding the average spectrum shown in panel (c). The four spectral positions indicated by i–iv correspond to different plasmon modes of the system [see Fig. 5(a)]. The dashed line indicates the spectral position of nonhybridized plasmon modes.

II. EXPERIMENT

A large number of gold NPs of a wide size distribution were fabricated using a recently developed synthesis approach described by Weinell *et al.* [16]. This approach involves the partial sublimation of a gold precursor particle of micrometer size induced by irradiation with an intense electron beam within an SEM. The vaporized gold redeposits on an amorphous silicon oxide substrate as gold NPs with decreasing size at increasing distances to the precursor's position (see Fig. 1 for a representative example). The shapes of the NPs are rather irregular, exhibiting crystallographic facets and twin boundaries, at large NP sizes above 100 nm, while approaching ellipsoidal shapes when they are smaller, i.e., the size regime studied in this work [see Fig. 1(d)]. For this thickness regime, electron holography measurements revealed that the thickness of the NPs is approximately 0.75 times smaller than their short in-plane dimension (details will be discussed further below). In order to study the plasmon localization, STEM-EELS datasets were recorded [17] at spatial subsets of the Au NP distribution with particle sizes [see indicated region in Fig. 1(d)] similar to the hole sizes in the Au networks of our previous measurements reported in Ref. [9]. Considering that

the thickness of these NPs is on the order of 10 nm, these measurements reveal the localization of hybridized plasmons at a thickness regime that is one order of magnitude larger than that of the thin Au networks studied previously, hence revealing the decisive impact of the thickness on localization.

The STEM-EELS datasets were recorded using an FEI Titan³ transmission electron microscope (TEM). During the scanning of a focused beam of electrons with 80 keV kinetic energy, a high-angle annular dark-field (HAADF) image was acquired. At the same time, EEL spectra were recorded at different probe positions on the sample. Here, the signal corresponds to the probability Γ of the beam electrons at probe position (x, y) to suffer from a specific energy loss ω , i.e., to excite a plasmon mode with a specific energy. This signal is called loss probability and corresponds to a projection of the plasmons' electric field component parallel to the electron beam: $\Gamma(x, y, \omega) \propto \int_{-\infty}^{\infty} \text{Re}\{e^{-i\omega z/v_z} \tilde{E}_z(\omega, x, y, z)\} dz$. The combination of EELS (spectral resolution) and STEM (spatial resolution) allows to study (1) the spectral position and width of different plasmon modes depending on the probe position, i.e., $\Gamma(\omega)$ at fixed (x, y) [see Fig. 1(c)] and (2) the spatial localization of plasmon modes from so-called loss probability maps [$\Gamma(x, y)$ at fixed ω ; see Fig. 5(a)].

III. SELF-CONSISTENT DIPOLE MODEL

To describe hybridized surface plasmon modes in large random assemblies of gold NPs in a numerically tractable way, we consider the interaction of dipolar plasmonic oscillators (corresponding to the individual NPs) exposed to an external electric field (i.e., the evanescent field of the beam electrons). This assumption neglects the impact of the irregular NP shapes as well as higher-order multipole couplings, which both lead to a slight modification of nearest neighbor coupling as well as the small spectral shifts (see Sec. III in the Supplemental Material [18]), which, however, does not modify the localization behavior of the surface plasmons, considered below, in a qualitative way. The coupled system of N dipoles under the influence of an external electric field can be described by the following set of $i = \{1, \dots, N\}$ equations:

$$\mathbf{P}_i(\omega) - \alpha_i(\omega) \sum_{j=1, j \neq i}^N \mathbf{G}_{ij}(\omega) \mathbf{P}_j(\omega) = \alpha_i(\omega) \mathbf{E}_i^{\text{ext}}(\omega), \quad (1)$$

which needs to be solved for the individual dipole moments $\mathbf{P}_i$ of the NPs. Here, α_i is the polarizability tensor of the individual NP i , $\mathbf{E}^{\text{ext}}$ is related to the field of the beam electrons, and $\mathbf{G}_{ij}$ is the retarded dipole interaction kernel that reads

$$\mathbf{G}_{ij}(\omega) = \frac{e^{ikr_{ij}} k^2}{r_{ij}} \cdot (\mathbf{I}_3 - \mathbf{e}_{ij} \otimes \mathbf{e}_{ij}) + e^{ikr_{ij}} (1 - ikr_{ij}) \left(\frac{3\mathbf{e}_{ij} \otimes \mathbf{e}_{ij} - \mathbf{I}_3}{r_{ij}^3} \right), \quad (2)$$

where $k = \omega/c$ denotes the wave number, r_{ij} the distance between NP i and NP j , $\mathbf{I}_3$ the 3×3 unit matrix, and $\mathbf{e}_{ij}$ the unit vector pointing from NP i to NP j .

Experimentally observable resonances of such a system of coupled dipoles occur at real frequencies ω , when the coefficient matrix on the left-hand side of Eq. (1) multiplied

by α_i^{-1} has a determinant close to zero, i.e., $\det(\alpha^{-1} - \mathbf{G}) < \delta \ll 1$. Here, α and $\mathbf{G}$ denote the composite matrices of all α_i or $\mathbf{G}_{ij}$, e.g.,

$$\alpha = \begin{pmatrix} \alpha_1 & \mathbf{0} & \cdots & \mathbf{0} \\ \mathbf{0} & \alpha_2 & \ddots & \mathbf{0} \\ \vdots & \ddots & \ddots & \vdots \\ \mathbf{0} & \mathbf{0} & \cdots & \alpha_n \end{pmatrix}. \quad (3)$$

Alternatively, resonances can be identified by solving a corresponding eigenvalue problem (see Sec. I in the Supplemental Material [18]), which is computationally less expensive. Transformation into an eigenvalue problem also allows us to consider the impact of the NP thickness on the position of the resonance frequencies in a semianalytical manner (see Sec. II in the Supplemental Material [18]). Employing a Drude model (plasma frequency ω_p and damping parameter γ) for the dielectric function of gold and well-known expressions for the polarizability of ellipsoidal NPs, we obtain the following resonance conditions:

$$\left| \lambda_n(\omega) + 3 \frac{\omega(\omega + i\gamma)}{\omega_p^2} - 1 \right| \leq \delta \quad (4)$$

and

$$\left| \lambda_{\perp,n}(\omega) + 3 \frac{\omega(\omega + i\gamma)}{\omega_p^2} \right| \leq \delta \quad (5)$$

for the eigenmodes (eigenvalues λ_n) of coupled dipole modes on a system of round NPs of random size and spatial distribution and extremely oblate NP (thin particle limit), respectively (see again Secs. I and II in the Supplemental Material [18] for details). Considering the order of magnitude of the eigenvalues $O(\|\lambda_n\|) = k^3 \prod_m a_m \ll 1$ (with $a_{1,\dots,3}$ denoting the semiaxes of the NPs), resonances in the 2D system are therefore shifted toward small frequencies with respect to the round NP limit.

To mimic the experimental situation [see Fig. 1(d)] as close as possible by the dipole model, the following procedure was applied. The HAADF STEM image contrast from the whole quadratic region [shown in Fig. 1(d)] has been thresholded (binarized) in the first step to distinguish between gold particles and substrate. Subsequently, a particle finding algorithm that identifies particles in the first step and fits 2D ellipses to each identified particle in the second step has been applied to the binarized image, yielding Au NP coverage of 53% and mean semiaxis size of 37 nm. The positions of the ellipses, as well as the size and orientation of the principal axis of the ellipses, are used as input for the coupled dipole model. The remaining free parameter, the thickness of the NPs, has been varied in the simulations to study the dependency of the resonant plasmonic modes on the thickness (e.g., with respect to the shift of resonance frequency predicted above).

Additionally, we measured the thickness and hence the relation between thickness and in-plane dimensions of a set of exemplary NPs by off-axis electron holography [19] (for details see Sec. IV in the Supplemental Material [18]), in order to determine the full three-dimensional shape of the NPs. The electron holograms were acquired at a different sample region, containing NPs of similar sizes to those in Fig. 1(d), that was

close to a hole in the substrate providing sufficient space for an undisturbed reference wave. Off-axis electron holography measures the thickness of the NPs by reconstructing the electrostatic phase shift imposed on the electron wave subjected to the accelerating projected electrostatic potential of the NPs. From these, an average ratio of 0.75 between the thickness and the in-plane width (i.e., short semiaxis of elliptical NP shape) of the NPs could be derived, corresponding to slightly oblate NP shapes in average. Thus, the average thickness of the NPs in the analyzed region is $t_{av} = 0.75 \times 37 \text{ nm} \approx 28 \text{ nm}$.

In order to quantify the localization of the resonant modes in the simulation, the inverse participation number has been evaluated [20]. The participation number quantifies how many particles take part in resonant modes at a certain frequency, and hence how delocalized a resonance is. It is defined through

$$p_{\text{sim}}(\omega) = \left\langle \sum_{i=1}^N \frac{1}{|\mathbf{P}_{i,n}(\omega)|^2} \right\rangle_n \quad (6)$$

averaged over all resonant eigenmodes n at a certain frequency ω . This participation number definition is similar to the experimentally measurable inverse normalized second moment of the loss probability that is therefore used to measure the plasmon localization in the experiment [20,21]:

$$p_{\text{exp}}(\omega) = \left(S \int_S \frac{\Gamma^2(\mathbf{r}_{\perp}, \omega)}{(\int_S \Gamma(\mathbf{r}_{\perp}, \omega) d^2 r_{\perp})^2} d^2 r_{\perp} \right)^{-1}. \quad (7)$$

Here, S denotes the integration area (i.e., the field of view of the measurement). We note that p_{exp} slightly deviates from p_{sim} in that it rather quantifies the scanning points of the STEM-EELS measurement participating in a resonance rather than the number of particles, which is why it is rescaled further below when comparing simulations with the experiment.

IV. RESULTS

A. Simulated thickness dependence of localization

In order to investigate the influence of the thickness of the NPs, several simulations were carried out (each with a homogeneous thickness of the NPs), with the thickness being changed gradually in a range between 0.2 and 20 nm. By inspecting the spectral dependence of their localization behavior, revealed by the polarization maps (see Fig. 2) and quantified by the inverse participation number (see Fig. 3), we can distinguish three distinct localization regimes prevailing at different NP thicknesses: (1) a thin sample regime for NP thickness below 0.4 nm, (2) a transition region between 0.4 and 10 nm thickness of the NPs, and (3) a thick sample regime for NP thickness above 10 nm.

In the low thickness regime (1), the localization increases with increasing energy (see green box in Fig. 2). Even though the dielectric function of gold allows for plasmon excitation up to $\approx 2.3 \text{ eV}$, the thin systems exhibit resonant hybridized modes only in a narrow frequency band (see plotted spectral range of the green curves in Fig. 3). That explains the previously found high transparency of very thin gold networks at optical frequencies [9]. In contrast, thick samples from regime (3) show a decrease in localization as a function of thickness

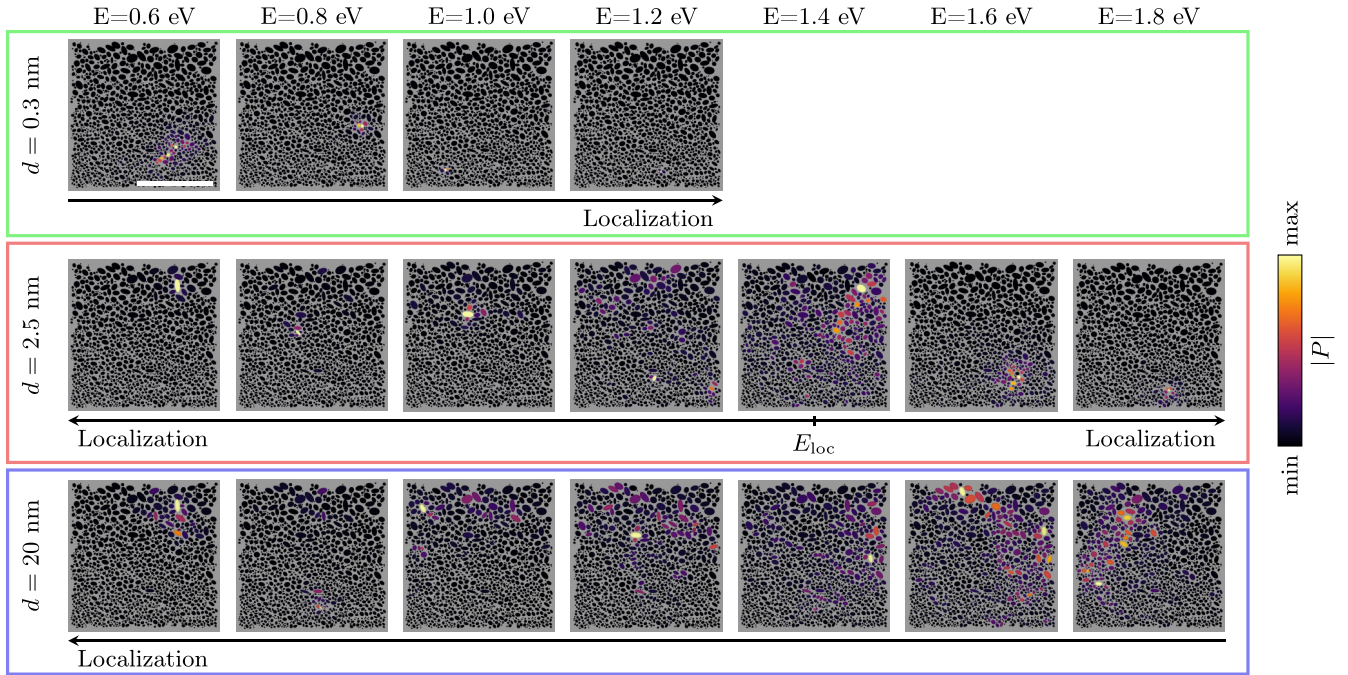


FIG. 2. Simulated polarization $|P|$ maps of selected eigenmodes with different energy and sample thickness. For thin samples ($d = 0.3$ nm, upper row/green box), localization increases the higher the energy until no resonant modes are present for energies above 1.2 eV. Thick samples ($d = 20$ nm, lower row/blue box), on the other hand, show decreasing localization with increasing energy. The maps of samples with intermediate thickness ($d = 2.5$ nm, lower row/red box) reveal the frequency of minimal localization. Above this frequency, they behave like the thin samples, and below, they behave like the thick ones. The scale bar shown in the map with $d = 0.3$ nm and $E = 0.6$ eV corresponds to 200 nm.

(see blue box in Fig. 2). This is reflected by the slope of the inverse participation number (blue curves in Fig. 3). NP assemblies of intermediate thickness from regime (2) (red box in Fig. 2) exhibit a behavior similar to thick samples up to an energy of minimal localization E_{loc} , i.e., a minimum of the inverse participation number (see red curves in Fig. 3). Above

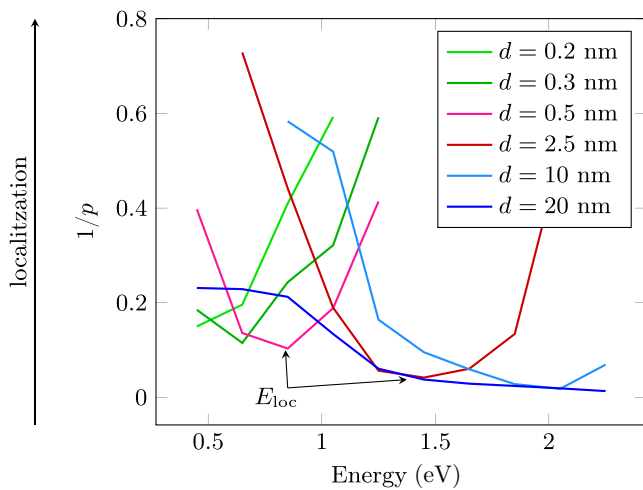


FIG. 3. Inverse participation number for different thicknesses d of the sample. The curves reveal a thin sample domain (green curves), a thick sample domain (blue curves), and a transition region (red curves). The minima of the curves correspond to minimal plasmon localization E_{loc} .

the energy of minimal localization, the spectral localization increases again as observed in thin samples.

From the above observations, we can conclude that the energy of minimal localization of the NPs increases with increasing thickness starting from the thin NP regime [see black curve in Fig. 4(a)]. The curve ends in a plateau slightly above 2 eV for thicknesses up to ≈ 10 nm, which corresponds to the transition to the thick particle limit, where the minimal localization coincides with the maximal surface plasmon frequency of Au.

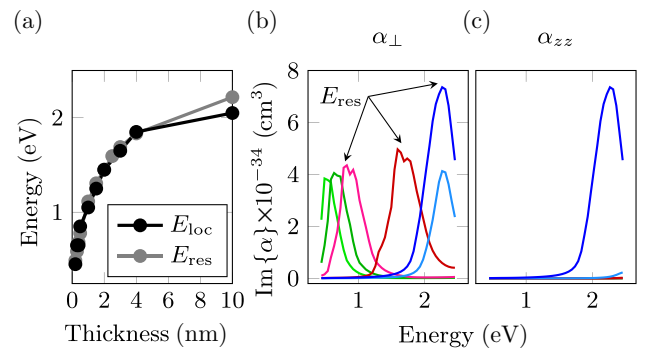


FIG. 4. (a) Energy of minimal localization E_{loc} compared to the mean resonance energy E_{res} of the dipolar mode given by the maxima of the curves in panel (b). (b) Mean in-plane and (c) out-of-plane imaginary parts of the polarizability for different NP thicknesses (light green = 0.2 nm, dark green = 0.3 nm, pink = 0.5 nm, dark red = 2.5 nm, light blue = 10 nm, and dark blue = 20 nm).

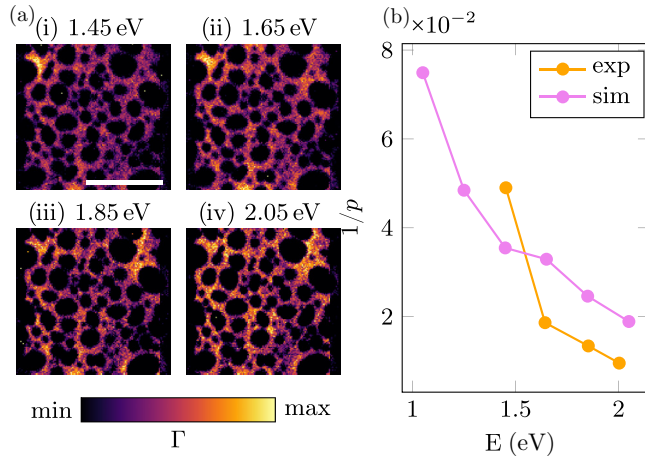


FIG. 5. (a) Experimental loss probability maps for different energies reveal decreasing localization with increasing energy. This behavior is reflected by the frequency dependence of the inverse participation number p_{exp} computed from Eq. (7) [orange curve in panel (b)]. A comparison with the participation number p_{sim} [Eq. (6)] of a simulation considering the actual NP thicknesses shows good qualitative agreement. The scale bar in panel (a) corresponds to 200 nm.

Since the positional arrangement of the NPs was kept constant in the simulations shown in Figs. 2 and 3, the origin of the thickness-dependent change of the localization behavior must be somehow connected to the polarizability of the individual NPs. To analyze the latter as a function of NPs thickness, we compared the mean resonance energy E_{res} of the in-plane dipolar plasmon mode given by the maximum (pole) of the imaginary (real) part of the polarizability ($\alpha_{\perp}$) [see Fig. 4(b)] with the energy of minimal localization [see Fig. 4(a)]. Indeed, almost perfect agreement between E_{loc} and E_{res} is observed for the transition region between 0.4 and 10 nm thickness. For thicknesses above 10 nm, the resonance frequency saturates similar to E_{loc} around 2.3 eV, which corresponds to the upper surface plasmon excitation limit given by the dielectric function of Au. Accordingly, all resonant modes of thick NP assemblies have energies $\leq E_{\text{loc}}$, and hence a decreasing localization with increasing energy is generally observed. In contrast, in systems with thicknesses in the range of 0.4 nm and below, all resonant modes have energies $\geq E_{\text{loc}}$ exhibiting increasing localization with increasing energy.

B. Experimental results

In order to verify the theoretic predictions concerning the thick particle limit (the thin particle limit has been studied already in, e.g., Ref. [9]), we conducted STEM-EEL spectrum imaging on the NP assembly region indicated in Fig. 1. This region contains particles of average in-plane dimensions of ≈ 37 nm. The average thickness of these NPs, as derived from electron holography measurements, is approximately 28 nm, which falls into the thick particle regime. Accordingly, we observe decreasing plasmon localization for increasing energy in both the loss probability maps [Fig. 5(a)] and the inverse

participation number [orange curve in Fig. 5(b)], as predicted by our simulation results (Sec. IV A), in which a constant thickness for all NPs was assumed.

To corroborate the dominating influence of the NP thickness on the observed localization behavior, we additionally simulated the more realistic scenario, where the NP thickness was set to $0.75 \times b$, with b being the short in-plane semiaxis of the NPs. The inverse participation number of this simulation is plotted in Fig. 5(b) (violet curve) and fits remarkably well to the experimental (orange) curve as well as to the localization behavior of the thick particle regime (see Fig. 3).

V. CONCLUSIONS

We can summarize that random assemblies of plasmonic Au NPs (having a mean semiaxis size of 37 nm and a coverage of 53%) exhibit a disorder-driven localization behavior that crucially depends on the thickness of the NPs. Maximal delocalization occurs at energies pertaining to a resonance condition of NPs' average dipole LSP mode because a maximal number of NPs can contribute to a delocalized mode at such energies. This stipulates two main localization regimes: (1) Random arrangements of NPs with thicknesses around and below 0.4 nm (i.e., nanoplatelets) exhibit increasing localization over the whole plasmonically active energy range between ≈ 0.3 and 2.3 eV, and (2) random arrangements of NPs with thicknesses above 10 nm show increasing delocalization in that energy regime. Intermediate thicknesses exhibit a maximal delocalization within the plasmonically active energy interval. These findings also translate to random metallic networks via the Babinet's principle. It is also important to note that the specifics (notably energy of minimal localization) depend on the in-plane size distribution and the NP material (e.g., spectral positions change when employing Ag NPs). The observed thickness dependence of hybridized LSP modes in random 2D plasmonic NP arrangements and plasmonic thin films provides a possible tuning knob for their optical response to be exploited in applications. For instance, the robust localization toward optical frequencies in the thin limit can be exploited to deliberately create highly transparent assemblies/thin films in the optical regime.

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DATA AVAILABILITY

The data that support the findings of this article are openly available [22].

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