

RESEARCH ARTICLE

Thickness-Resolved Plasmonic Windows in Graded Gold Nanoisland Films for Raman, Vapor, and Ellipsometric Sensing

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Abstract

Gold nanoisland sensors are commonly optimized by following the largest optical response, although a single maximum does not define the best film for every readout. This paper determines how nominal Au thickness separates the functional windows of a graded, annealed gold nanoisland film used for Raman enhancement, ethanol transmission sensing, water-induced ellipsometric contrast, and ellipsometric detection of chemisorbed 4-mercaptopbenzoic acid. The common coordinate is deposited Au thickness on fused silica. The decisive morphological transition occurs between 2.0 and 4.4 nm: particle density decreases from 2090 to 88 particles μm^{-2} , while covered area decreases from 0.49 to 0.21. These numbers distinguish a junction-dense low-thickness state, an open-contact intermediate state, an oscillator-dominant high-thickness state, and a continuity-threshold state. 4-MBA Raman detection is strongest near 1.6 nm, where the 1074 and 1586 cm^{-1} bands are most intense under both 532 and 633 nm excitation. Ethanol sensing is strongest around 3.2–3.5 nm, and water-induced ellipsometric contrast occupies the broader 2.0–4.0 nm interval. Bound 4-MBA ellipsometry shifts to 6.8–7.0 nm, where the far-field response is stronger. The practical answer is therefore readout-specific: 1.4–2.2 nm for Raman, 2.8–3.6 nm for volatile or condensable analytes, 6.5–7.1 nm for bound-layer ellipsometry, and 2.0–3.5 nm as the best multimodal compromise.

Keywords: analytical spectroscopy; gold nanoislands; localized surface plasmon resonance; surface-enhanced Raman spectroscopy; spectroscopic ellipsometry; vapor sensing; water adsorption; sensor selection

1. Introduction

Gold nanostructures occupy a central place in analytical chemistry because adsorption, refractive-index change, molecular binding, aggregation, and local electromagnetic coupling can all alter their optical response. The resonance condition of a noble-metal nanoparticle depends on size, shape, dielectric environment, and interparticle spacing; this makes gold and related plasmonic materials useful as label-free optical transducers and as surface-enhanced Raman substrates [1–4]. The same sensitivity creates a practical selection problem: the morphology that maximizes one observable can be unsuitable for another observable measured on the same material.

Localized surface plasmon resonance (LSPR) sensing relies on a

nanostructured metal surface whose extinction, transmission, scattering, or reflectance changes when the surrounding dielectric environment changes. Early and later LSPR biosensing work demonstrated molecular-recognition readouts at the nanoscale, while also showing that refractive-index sensitivity, binding access, and spectral resolution must be considered together [5–8]. Nanostructured plasmonic films are therefore not merely metal coatings; they are measurement components whose morphology must be matched to the chemical event and to the instrument collecting the signal [9–11].

Surface-enhanced Raman spectroscopy imposes a more local requirement. The early observations of intense Raman scattering on roughened noble-metal surfaces and the subsequent SERS literature established that enhancement is governed by electromagnetic concentration

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at high-curvature sites, nanoscale junctions, and molecule–metal proximity, with chemical interactions contributing in some cases [12–16]. Single-molecule and highly sensitive SERS demonstrations further showed that the rare local fields responsible for Raman enhancement are not equivalent to broad far-field extinction maxima [17–19]. A Raman-optimized nanoisland film can therefore require a dense population of small, closely separated islands even when another optical channel favours larger, more isolated particles.

Vapor and liquid-film sensing impose a different constraint. A mobile molecule such as ethanol must reach accessible metal–dielectric boundaries and perturb an optical response that remains strong enough to be measured. High-resolution LSPR gas sensing demonstrates that plasmonic transducers can detect small refractive-index changes, but the analytical surface still needs physically reachable adsorption or diffusion pathways [20]. Water adsorption measured by ellipsometry similarly reflects an interaction between condensable molecules, accessible surface area, and polarization-dependent optical contrast. These channels do not necessarily reward the narrowest inter-island junctions or the highest island count.

Spectroscopic ellipsometry adds another measurement axis because it probes changes in polarization state rather than only scalar intensity. Classical and modern ellipsometry texts show that the technique is sensitive to thin films, interfaces, and anisotropic optical changes when the optical model and measurement geometry are suitable [21–23]. For plasmonic nanoisland films, a chemisorbed molecular layer can be easier to detect when the underlying gold islands have strong oscillator strength, even when that surface is not the best SERS substrate.

Thermally formed or annealed gold nanoislands are especially suitable for thickness-resolved selection because a nominally simple fabrication variable can generate large changes in density, coverage, diameter, spacing, and partial coalescence. Studies of partially embedded nanoparticles and thermally generated Au nanoislands have shown that LSPR and SERS performance depend strongly on morphology and on how the islands contact the supporting substrate [24–26]. Reviews and substrate-engineering studies further support morphology-led interpretation of gold nanoisland sensing rather than interpretation by a single response maximum [27–30].

The research question is therefore specific: which nominal Au thickness intervals in a graded gold nanoisland film correspond to Raman enhancement, ethanol transmission response, water-induced ellipsometric contrast, bound-molecule ellipsometry, and multimodal use? The answer requires the thickness coordinate, the measured particle population, the covered-area fraction, the optical response, and the chemical readout to be considered together. The goal is not to rank all signals on one scale, but to identify which surface morphology supports each analytical task.

2. Materials and Methods

2.1. Thickness-coded measurement matrix

Graded gold nanoparticles were prepared by graded-layer magnetron sputtering followed by annealing on fused silica [31]. Nominal deposited Au thickness was used as the common coordinate because the same lateral strip carries the morphology sequence and the response sequence. The measurements used here include microscopy-derived particle density and covered-area fraction, optical response, ethanol transmission behaviour, water-induced ellipsometric contrast, ellipsometric response to covalently bound 4-mercaptobenzoic acid (4-MBA), and 4-MBA Raman bands collected under 532 and 633 nm excitation. Each thickness point was classified by the physical event it captures. The 2.0 nm point defines the dense particle population; the 2.8–3.6 nm interval defines the intermediate open-contact region; the 4.4 nm point marks the sparse particle population after density collapse; the 6.8–7.0 nm region identifies a strong oscillator condition for ellip-

sometry; and the 7.6 nm region marks the onset of coalescence-prone or plasmon-cancellation behaviour. This organization keeps the numerical measurements in the text rather than reducing them to an undifferentiated optimization score.

The numerical entries in Table 1 separate response maxima from usable analytical windows. A response maximum can be narrow, whereas a practical window must also tolerate analyte access, local field formation, instrument geometry, and modest thickness variation. The critical separation is between the low-thickness Raman region and the higher-thickness ellipsometric molecular-layer region; they reflect different optical mechanisms rather than competing values for the same optimum.

2.2. Thickness-state assignment

Four surface states were assigned from the measured morphology and response sequence. The junction-dense state contains high particle population, high metal coverage, and short interparticle separations. It favours SERS because local field concentration and reporter-molecule placement near metallic junctions control the Raman signal. The open-contact state has reduced crowding while preserving optical activity; it favours ethanol and water because mobile or condensable molecules require reachable metal–dielectric boundaries. The oscillator-dominant state has stronger far-field optical response and favours ellipsometric detection of bound 4-MBA. The continuity-threshold state is excluded from preferred sensing use because partial film continuity weakens localized-plasmon reliability.

A thickness interval was accepted for a readout only when three conditions coincided: the measured response appeared in that region, the morphology could physically support the transduction mechanism, and the selected surface remained accessible to the relevant analyte. This direct assignment avoids an unsupported numerical score. The evidence consists of specific thickness intervals, particle-density and covered-area values, optical-response positions, and analyte-dependent readouts, not replicate calibration curves for every analyte concentration.

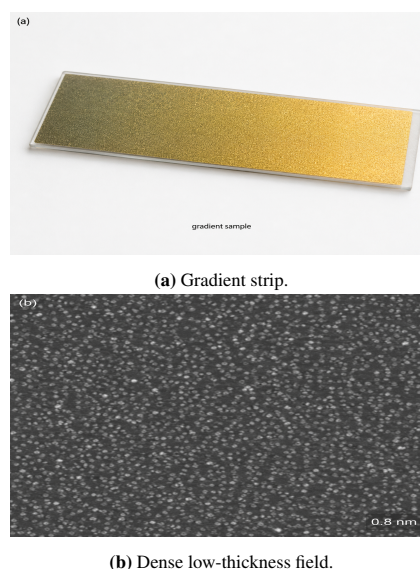
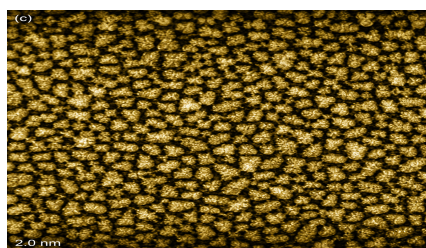


Figure 1: Thickness-gradient morphology.

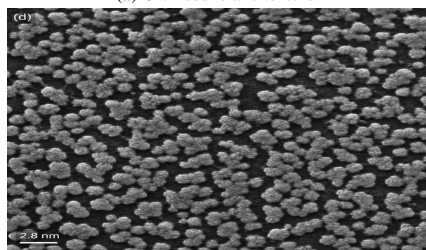
The morphology atlas in Figures 1, 2 and 3 show why the film cannot be interpreted as a single type of plasmonic substrate. The low-thickness panels contain a dense, highly populated island field, whereas the intermediate and high-thickness panels show progressively wider separations and more connected textures. This visual progression supports thickness as a surface-state coordinate rather than a simple monotonic

Table 1: Thickness-coded measurements.

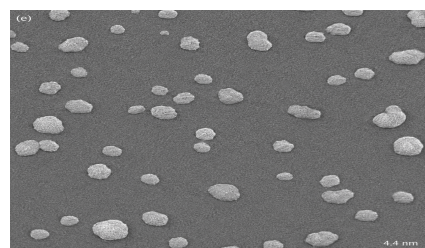
Measurement class	Thickness coordinate	Specific observation	Analytical interpretation
Dense morphology	2.0 nm	2090 particles μm^{-2} ; covered-area fraction 0.49	Junction-rich state for 4-MBA Raman enhancement
Intermediate morphology	2.8–3.6 nm	Lower crowding with measurable optical activity	Open-contact state for mobile and condensable adsorbates
Sparse morphology	4.4 nm	88 particles μm^{-2} ; covered-area fraction 0.21	Post-collapse particle population with reduced hotspot probability
Reflectance response	near 6.8 nm	Strong reflectance resonance	Oscillator-dominant state for ellipsometric molecular-layer response
Continuity boundary	near 7.6 nm	Coalescence-prone or plasmon-cancellation behaviour	Excessive thickness for robust localized-plasmon sensing
Raman channel	near 1.6 nm	Strong 4-MBA bands at 1074 and 1586 cm^{-1} under 532 and 633 nm excitation	Low-thickness SERS window
Ethanol channel	about 3.2–3.5 nm	Strong dynamic transmission change near 580 nm	Volatile-molecule sensing window
Water channel	about 2.0–4.0 nm	Ellipsometric contrast in the intermediate region	Condensable-adsorbate window
Bound 4-MBA channel	about 6.8–7.0 nm	Strong ellipsometric molecular-layer response	Chemisorbed-layer ellipsometry window



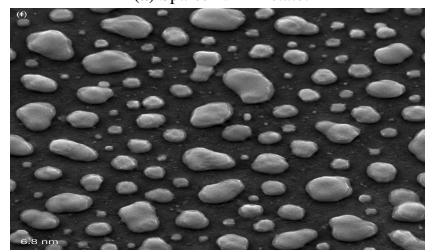
(a) Crowded island texture.



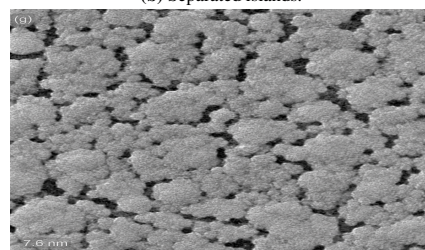
(b) Narrow-junction texture.

Figure 2: Thickness-gradient morphology.

(a) Sparse 4.4 nm state.



(b) Separated islands.



(c) Connected high-thickness region.

Figure 3: Thickness-gradient morphology.

tuning variable.

The paired density and coverage records in Figures 4, 5 and 6 provide the numerical basis for the state boundaries. The collapse from 2090 to 88 particles μm^{-2} is large enough to change the dominant sensing mechanism, not merely the signal amplitude. A film below this collapse is a hotspot-rich Raman candidate, whereas a film inside the transition interval exposes more surface for mobile analytes while preserving measurable plasmonic contrast.

3. Results and Discussion

3.1. Analytical surface states along the gradient

The graded film divides into four analytically meaningful surface states. The junction-dense state occupies the low-thickness region where particle population and covered area are high. This state is not simply the smallest-thickness region; it is the region where near-field junctions are most probable. The open-contact state occupies the intermediate region where the film has lost some crowding but has not yet become optically weak or fully sparse. The oscillator-dominant state appears near the strongest reflectance response, where a molecular overlayer can produce a larger polarization-dependent perturbation. The continuity-threshold state lies near the high-thickness boundary where localized plasmonic behaviour becomes less dependable. This segmentation changes the interpretation of the thickness-resolved measurements. Signals are no longer placed on one ranked thickness list; each signal is connected to the surface state that can physically generate it. The Raman maximum near 1.6 nm is therefore not in

conflict with ethanol and water behaviour around 3.2–3.5 nm, and neither is in conflict with 4-MBA ellipsometry near 6.8–7.0 nm. The different response positions show that the same gold film supports multiple transduction regimes.

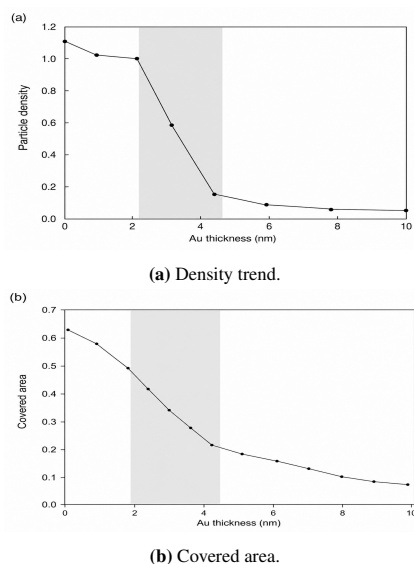
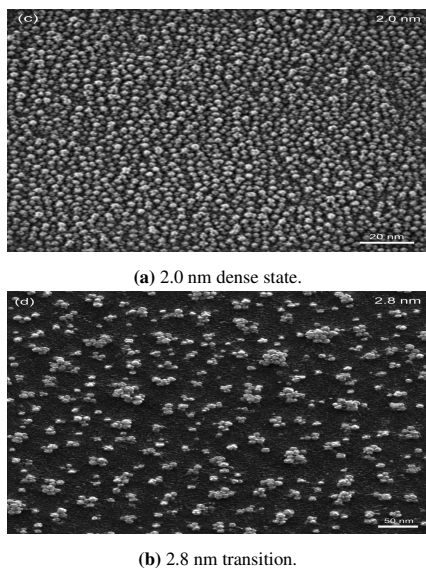
The assignments in Table 2 keep the measured thickness intervals, morphology change, and readout mechanism visible at the same time. The table does not collapse the material into a single score. It separates the film into operational states that can be selected according to the chemical task: molecular reporter Raman scattering, volatile-molecule transmission, water adsorption, or bound-layer ellipsometry.

3.2. Raman response belongs to the junction-dense state

The strongest 4-MBA Raman response belongs to the low-thickness state centred near 1.6 nm. This position agrees with the dense 2.0 nm morphology, where the surface still carries 2090 particles μm^{-2} and a covered-area fraction of 0.49. The 1074 and 1586 cm^{-1} reporter bands are strongest under both 532 and 633 nm excitation, so the preferred Raman region is not explained by a single excitation wavelength. It is

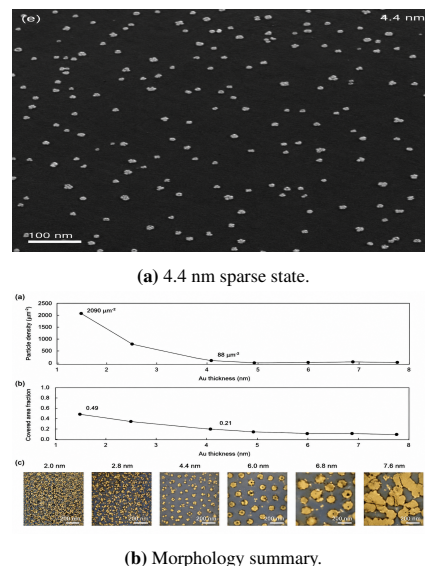
Table 2: Thickness-state assignments.

Surface state	Dominant physical condition	Best-matched channel	Reason for assignment
Junction-dense	High particle count, high covered area, short interparticle separations	4-MBA SERS	Raman enhancement requires intense local fields and reporter placement near metallic junctions
Open-contact	Reduced crowding with preserved optical response	Ethanol and water sensing	Mobile and condensable adsorbates require accessible boundaries as well as plasmonic sensitivity
Oscillator-dominant	Strong far-field resonance and larger optical perturbation	Bound 4-MBA ellipsometry	A chemisorbed molecular layer can be measured through a polarization-sensitive response
Continuity-threshold	Partial coalescence or plasmon cancellation	Avoided for selection	Localized plasmonic behaviour becomes less reproducible and less mode-specific

**Figure 4:** Density and coverage transition.**Figure 5:** Density and coverage transition.

a morphology-controlled near-field condition.

This assignment is important for analytical spectroscopy. A far-field reflectance maximum is not required for the highest SERS signal because Raman enhancement is dominated by local field concentration. Increasing nominal gold thickness beyond the density-collapse interval can create larger optical oscillators, but it reduces the population of narrow junctions where the reporter molecule is likely to experience strong enhancement. The Raman result therefore answers one part of the research question clearly: a dedicated 4-MBA SERS substrate

**Figure 6:** Density and coverage transition.

belongs in the low-thickness junction-dense region, not in the strongest far-field reflectance region.

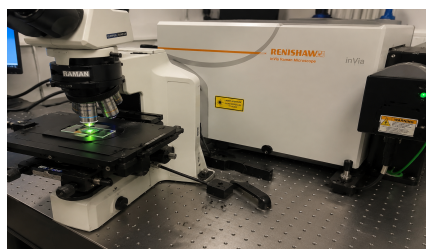
The Raman instrumentation views in Figures 7 and 8 show why spatial selection along the gradient matters. Spot-wise collection interrogates a local surface state rather than an averaged entire strip. For this reason, the low-thickness Raman window is a positional selection on the graded substrate; routine use would require careful positioning or uniform films prepared at the selected thickness.

3.3. Ethanol and water responses identify an open-contact interval

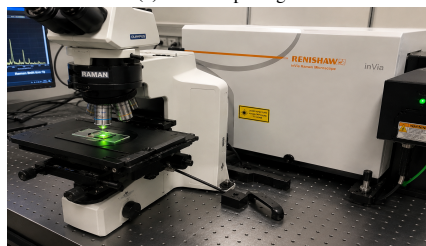
Ethanol and water responses occupy the intermediate region rather than the dense Raman region. Ethanol transmission sensing reaches its strongest behaviour around 3.2–3.5 nm, with a dynamic component near 3.2 nm and an intensity-drop descriptor near 3.5 nm. Water-induced ellipsometric contrast spans a broader 2.0–4.0 nm region. The overlap is analytically meaningful because both analytes depend on accessible surface contact while still needing the gold nanoislands to preserve plasmonic sensitivity.

The open-contact interval reconciles analyte access with optical response. Very dense islands provide many possible near-field sites, but they can also make the response spatially heterogeneous and dependent on a small population of junctions. Very sparse islands improve exposure of individual particles but reduce the number of active interaction regions. The 2.8–3.6 nm interval sits between these extremes. It is therefore the strongest choice for volatile or condensable analytes when the analytical objective is a reproducible transmission or ellipsometric response rather than maximum Raman enhancement.

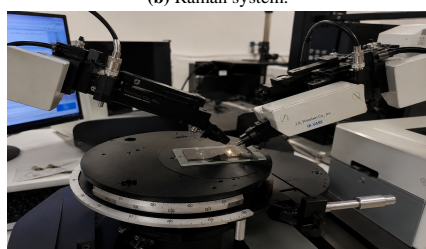
The optical arrangement in Figures 9 and 10 link the lateral gradient to a measurable transmission channel. The geometry makes the 3.2–3.5 nm interval useful because the selected region can be interrogated



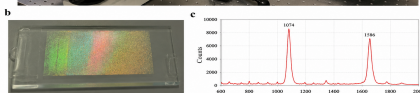
(a) Microscope stage.



(b) Raman system.



(c) Stage alignment.

Figure 7: Raman readout.

(a) Reporter response.



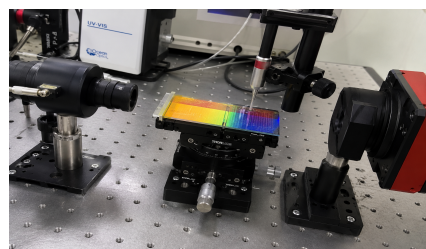
(b) Probe spot.

Figure 8: Raman readout.

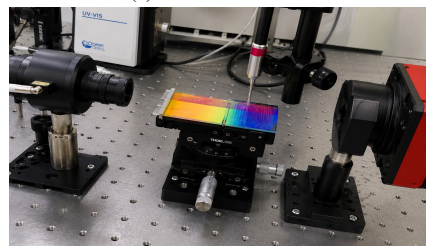
without changing substrate chemistry. In practical instrumentation, the same fabrication route can be used to prepare a vapor-sensitive film when final thickness is targeted to the open-contact state instead of to the Raman state.

3.4. Bound-molecule ellipsometry shifts to the oscillator-dominant state

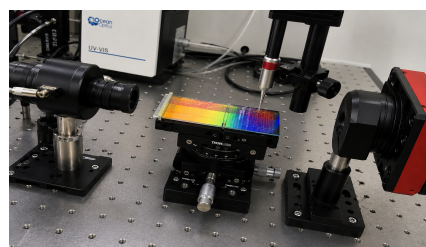
Ellipsometric detection of covalently bound 4-MBA occupies a higher-thickness region near 6.8–7.0 nm. This position follows the measurement principle. A stable thiol-bound molecular layer perturbs the



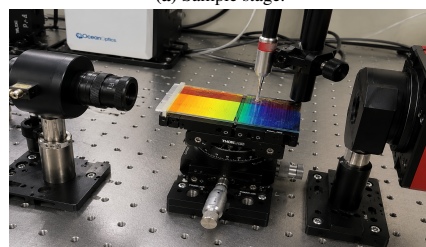
(a) Transmission bench.



(b) Gradient alignment.

Figure 9: Optical interrogation.

(a) Sample stage.



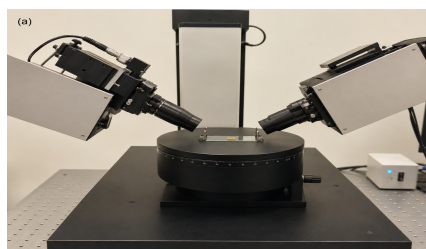
(b) Spectral readout.

Figure 10: Optical interrogation.

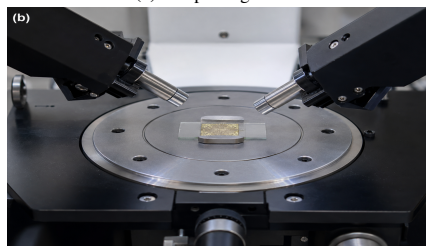
polarization-dependent response most strongly when the underlying gold nanoisland film has sufficient oscillator strength. The same region coincides with the strongest reflectance resonance, so bound-molecule ellipsometry is assigned to the oscillator-dominant state.

The high-thickness region requires narrow process control because it lies close to the continuity-threshold region near 7.6 nm. A small shift in deposited thickness, annealing outcome, or position on the gradient can move the film toward coalescence-prone behaviour. That boundary matters more for routine instrumentation than for a single laboratory maximum. The useful ellipsometric interval is approximately 6.5–7.1 nm, where the high-oscillator advantage is kept below the region of reduced localized-plasmon reliability.

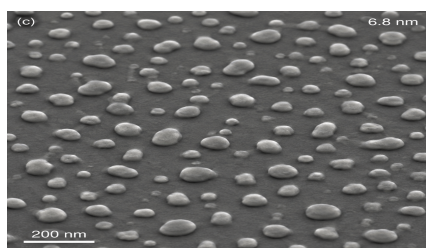
The ellipsometric panels in Figures 11 and 12 emphasize that high optical contrast and robust localized-plasmon sensing are not identical. The 6.8 nm region offers a favourable oscillator condition for detecting a bound molecular layer, whereas the 7.6 nm region illustrates why additional thickness is not automatically beneficial. The analytical decision is therefore to use the high-thickness region only for bound-layer ellipsometry and not as a general-purpose substrate.



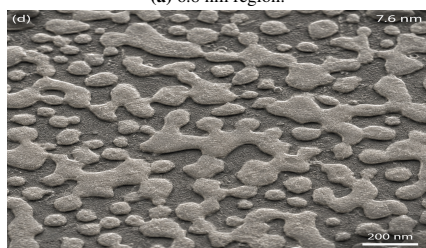
(a) Sample alignment.



(b) Probe geometry.

Figure 11: Ellipsometric boundary.

(a) 6.8 nm region.



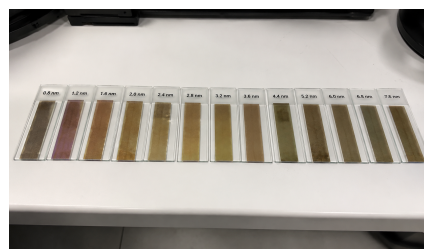
(b) 7.6 nm boundary.

Figure 12: Ellipsometric boundary.

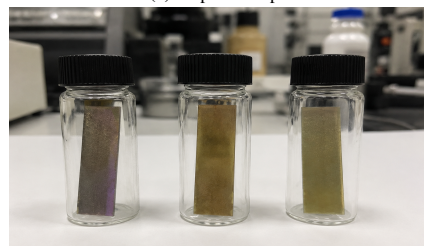
3.5. Multimodal selection favours a compromise, not a universal maximum

A general substrate intended to support more than one readout should be selected from 2.0–3.5 nm. This interval begins at the maximum-density morphology and extends through the open-contact region where ethanol and water responses are strongest. It cannot provide the strongest ellipsometric response to bound 4-MBA because that channel shifts toward 6.8–7.0 nm. Its value is different: it preserves enough near-field coupling for Raman-relevant operation, supports mobile-adsorbate sensing, and remains far from the continuity-threshold state. This finding prevents overinterpretation of the graded film. The optimum is not a single thickness shared by all modalities. Instead, the film behaves as a sequence of usable states. A Raman-only device should be prepared near the low-thickness junction-dense state. A vapor or water sensor should use the open-contact interval. A bound-layer ellipsometric sensor should use the oscillator-dominant region with strict thickness control. A multimodal test substrate should accept a moderate compromise and use 2.0–3.5 nm.

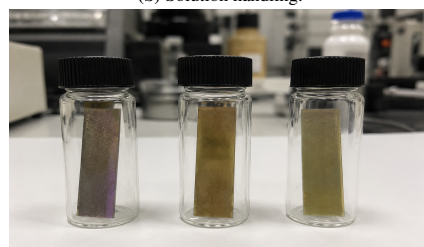
The handling sequence in Figure 13 adds a practical constraint to the



(a) Prepared strips.



(b) Solution handling.



(c) Replicate exposure.

Figure 13: Analytical handling.

optical interpretation. A selected thickness must remain usable during preparation, exposure, rinsing or solution handling, and repeated measurement. The multimodal compromise interval is attractive not because it gives the largest response in every channel, but because it avoids the most fragile extremes of the gradient.

The selection table answers the research question in operational terms. The measurements support several readout-specific windows, but they do not support a universal gold thickness. The most defensible multimodal choice is 2.0–3.5 nm, whereas the best single-channel choices separate according to whether the measurement depends primarily on hotspot density, mobile-analyte access, or far-field ellipsometric oscillator strength.

3.6. Implications and validation needs

The main implication for analytical chemistry is that graded gold nanoisland films should be described with both morphology and use case. Reporting only the strongest spectral feature can obscure whether the substrate was optimized for Raman enhancement, vapor sensing, water adsorption, or bound-layer ellipsometry. A thickness label is meaningful only when connected to the surface state generated at that thickness and to the readout mechanism being used.

The main implication for instrumentation is that the selected thickness should be tied to the measurement path. A Raman microscope demands positional control of the laser spot over a junction-dense region. A transmission vapor sensor requires stable optical alignment over the open-contact interval. An ellipsometer needs a high-oscillator region but must remain below the continuity threshold. These requirements are not interchangeable, even though all arise from the same gold nanoisland material family.

The limitations are also specific. The measurements support relative thickness selection, but they do not establish limits of detection, adsorption isotherms, chemical selectivity against interferents, long-term

Table 3: Readout-specific thickness choices.

Analytical use	Preferred thickness	Accepted interval	Main justification
4-MBA SERS	near 1.6 nm	1.4–2.2 nm	Junction-dense morphology gives the highest probability of reporter molecules occupying high-field regions
Ethanol transmission sensing	3.2–3.5 nm	2.8–3.6 nm	Accessible surface contact and preserved plasmonic response coincide near the dynamic transmission maximum
Water-induced ellipsometric contrast	intermediate region	2.0–4.0 nm, with 2.8–3.6 nm preferred for shared use	Condensable adsorbate response follows the open-contact part of the gradient
Bound 4-MBA ellipsometry	6.8–7.0 nm	6.5–7.1 nm	Strong oscillator response improves polarization-sensitive molecular-layer detection
Multimodal substrate	around 3.2 nm	2.0–3.5 nm	Raman relevance, vapor/water response, and distance from continuity-threshold behaviour are jointly maintained
Avoidance region	near 7.6 nm and above	not recommended	Partial continuity or plasmon cancellation reduces localized-plasmon reliability

drift, recovery time, or batch-to-batch variation. Those quantities require concentration-series experiments and independent repeat fabrication. The thickness intervals identified here should therefore guide substrate selection for analytical design, not replace full calibration of a finished sensor.

4. Conclusion

A single graded gold nanoisland film does not produce one universal sensing thickness. It produces separate functional windows governed by the relation between island morphology and readout physics. The 2.0 to 4.4 nm morphology transition, where particle density decreases from 2090 to 88 particles μm^{-2} and covered area falls from 0.49 to 0.21, is the central evidence. This transition separates the high-junction surface required for 4-MBA SERS from the more accessible intermediate surface required for ethanol and water sensing, and from the stronger optical-oscillator region required for bound-layer ellipsometry.

The specific answer is readout-dependent. 4-MBA SERS belongs to the low-thickness junction-dense region centred near 1.6 nm because Raman enhancement requires high local-field concentration and because the 1074 and 1586 cm^{-1} reporter bands are strongest there under both 532 and 633 nm excitation. Ethanol and water sensing belong to the open-contact region around 3.2–3.5 nm because mobile or condensable molecules require accessible metal–dielectric boundaries while still benefiting from plasmonic response. Bound 4-MBA ellipsometry belongs to the oscillator-dominant region near 6.8–7.0 nm because a chemisorbed layer is most effectively detected when the polarization-sensitive optical response is strong. The region near 7.6 nm should not be treated as a preferred window because coalescence-prone or plasmon-cancellation behaviour weakens localized-plasmon reliability. For analytical deployment, a dedicated Raman film should be prepared in the 1.4–2.2 nm interval, a volatile or water-responsive film in the 2.8–3.6 nm interval, and a bound-layer ellipsometric film in the 6.5–7.1 nm interval with tight control against over-thickening. When a single gold nanoisland substrate must support several readouts, the most defensible compromise is 2.0–3.5 nm because it retains Raman-relevant coupling, supports vapor and water response, and stays well below the continuity-threshold state. The conclusion therefore answers the research question directly: thickness selection must be matched to the analytical mode, and the intermediate 2.0–3.5 nm region is the only defensible compromise for multimodal use.

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