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A Lightweight, Low-Label Framework Towards Autonomous Morphology-Resolved SEM–EDS Analysis in Material Microscopy

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Abstract

High-throughput materials characterization is essential for self-driving laboratories, yet it is often the slowest and least automated step, largely because extracting meaningful structure–property relationships from complex, multimodal data still requires substantial expert interpretation. This is particularly true for nanomaterials, whose properties are highly sensitive to structure, environment, and measurement conditions. Moreover, extracting statistically meaningful particle-level compositional information, such as chemical heterogeneity and impurity distributions, from paired Scanning Electron Microscopy (SEM)–Energy-Dispersive X-ray Spectroscopy (EDS), remains largely manual and expertise-intensive. Here, we establish a lightweight, data-efficient deep learning framework that integrates directly into SEM–EDS workflows to enable rapid, automated, morphology-resolved particle identification and compositional quantification. To overcome the reliance of existing deep learning methods on large annotated datasets and time-intensive manual labeling, our framework operates with minimal human annotation. Starting from only five fully human-labeled SEM images containing 577 annotated objects, we use a canvas-based strategy to generate

a synthetic training dataset comprising approximately 6,000 labeled objects. Training the lightweight model with this augmented dataset yields a mean intersection over union (mIoU) of 0.74, representing a 19.8% relative improvement over training on the experimental images alone. The benefit becomes more pronounced as labeled data grow scarcer: with only two images containing 232 annotated objects, our synthetic augmentation increases mIoU by up to 67%, demonstrating that the pipeline is particularly valuable in the extreme low-data regime. Applied to MXene energy-storage materials, the framework links particle morphology to elemental composition through EDS maps and reveals impurity distributions that remain hidden in conventional field-averaged analyses. The pipeline is designed as a deployable solution that integrates directly into existing SEM–EDS instrumentation without requiring complex architectures or high-performance computing, establishing an accessible route toward autonomous electron microscopy and the broader adoption of deep learning-enabled materials characterization.

1 Introduction

Characterization methods that resolve chemically distinct features across statistically representative sample areas are essential for establishing relationships among microstructure, local composition, and materials properties. This capability is particularly important for heterogeneous and multiphase materials, in which distinct microstructural features and constituents, including particles, phases, grains, interfaces, chemically distinct domains, residual precursors, contaminants, and degradation products, may contribute differently to overall performance. Conventional techniques such as X-ray diffraction, NMR spectroscopy, vibrational spectroscopy, inductively coupled plasma mass spectrometry, and X-ray photoelectron spectroscopy provide indispensable structural and compositional information. However, signals are generally averaged over sample volumes or areas and they cannot therefore readily associate specific chemical signatures with individual microstructural features. Spatially resolved approaches are therefore needed to preserve associations between local structure and composition.

Scanning electron microscopy (SEM) remains a core characterization tool in materials science because it resolves surface features from the microscale to nanoscale and reveals microstructural heterogeneity in complex materials systems¹. When coupled with energy-dispersive X-ray spectroscopy (EDS), SEM enables the elemental composition of selected features to be assessed at individual points or across mapped regions of the surface of a material, correlating morphology with local elemental information. EDS detects characteristic X-rays emitted by elements in the sample following electron-beam irradiation, generating spectra of X-ray counts as a function of energy, with characteristic peaks serving as elemental fingerprints (Figure 1a)^{2, 3}. Elemental maps provide the spatial distributions of detectable elements, while quantitative analysis estimates their atomic and mass fractions within the electron-beam interaction volume of the analyzed region. Integrated SEM–EDS analysis therefore supports compositional classification, approximate stoichiometric assessment, candidate phase assignment, and the identification of foreign inclusions or contaminant populations^{3, 4}. These capabilities are essential for linking nanoscale heterogeneity to performance across diverse material systems, and for investigating degradation, interphase formation, and failure in battery electrode materials^{5, 6}. They are also broadly relevant to high-value manufacturing sectors, including semiconductor⁷ and pharmaceutical manufacturing, where compositional uniformity and microstructural control influence process yield⁸, product performance, quality assurance, and regulatory compliance.

Despite these strengths, conventional SEM–EDS analysis remains heavily reliant on manual region selection, feature identification, and expert interpretation⁹ (Figure 1a). Analysts typically inspect a limited number of fields of view, select representative particles or regions of interest, and interpret associated EDS spectra and maps. This process is time- and resource-intensive and can introduce sampling bias and analyst-dependent selection of regions and features. Only a small fraction of the available microstructural features may be examined, limiting throughput, reproducibility, and statistical representativeness, particularly when large numbers of features must be evaluated across multiple sample regions⁹. This limitation is especially

consequential for battery materials, whose performance depends on chemical and microstructural heterogeneity spanning length scales from individual particles to complete electrodes.

As microscopy datasets increase in volume and complexity, materials characterization increasingly requires high-throughput workflows with reduced manual intervention, often supported by machine learning and correlative imaging strategies^{10, 11}. Deep-learning methods have enabled automated microstructure segmentation and feature extraction from microscopy images across diverse modalities¹²⁻¹⁸. However, supervised segmentation commonly requires large manually annotated training datasets, in some cases comprising tens of thousands of segmented objects. The cost of producing these annotations limits the practicality of such methods and increases the risk of overfitting when only small experimental datasets are available¹⁹. Few-shot, human-in-the-loop, foundation-model-based, self-supervised, and zero-shot approaches can reduce the annotation burden and improve robustness in data-scarce settings²⁰⁻²². Nevertheless, most automated microscopy workflows have focused on segmentation or feature extraction within individual image channels. The automated use of SEM-derived morphological masks to quantify signals from co-registered EDS maps at the level of individual features remains underdeveloped. A bibliometric survey of this landscape (Supplementary Figure S1) reveals that automated SEM segmentation, generative synthetic-data methods, and domain adaptation have largely developed in diverse research communities, while remains limited connectivity to co-acquired EDS analysis. Consequently, a scalable framework for preserving and quantifying morphology–composition relationships across large SEM–EDS datasets are still needed.

The development of advanced electrode materials for energy-storage technologies has created a growing need for characterization methods that can resolve compositional heterogeneity across complex microstructures. MXenes provide a demanding proof-of-concept system for automated, morphology-resolved SEM–EDS analysis because their synthesis can produce morphologically and compositionally distinct particle populations, residual precursor phases, and contaminants²³. MXenes are a rapidly expanding class of two-dimensional transition-metal carbides, nitrides, and carbonitrides whose

compositional diversity, controllable surface terminations, and solution processability make them promising materials for energy storage and other functional applications^{23, 24}. $\text{Ti}_3\text{C}_2\text{T}_x$ is the most widely studied MXene, where T_x denotes surface terminations, such as $-\text{O}$, $-\text{OH}$, and $-\text{F}$, that influence electronic structure, interfacial chemistry, and electrochemical behavior²⁵⁻²⁷. Its high electrical conductivity, hydrophilic and chemically tailorable surface, layered morphology, and tunable interlayer structure have motivated efforts to develop scalable production routes with controlled surface chemistry^{26, 28}.

MXene synthesis nevertheless remains a central obstacle to translating laboratory-scale materials into practical devices^{23, 24, 29}. Although advances such as molten-salt and low-temperature molten-salt etching have increased the feasibility of scalable and surface-chemistry-controlled production, variations in etching, delamination, flake size, defects, and termination chemistry can substantially affect the final material properties^{24, 27}. Synthesized samples may contain target MXene flakes together with residual MAX precursor phases, oxides, and etching byproducts. For example, aluminum fluoride trihydrate ($\text{AlF}_3 \cdot 3\text{H}_2\text{O}$) can be present as a byproduct of the fluoride-based etching of the Ti_3AlC_2 MAX precursor. Such residual species may impede ion transport, block active sites, and promote parasitic interfacial reactions, thereby compromising energy density, cycling stability, and device durability^{4, 8, 10, 30-32}. Rigorous quality control therefore requires the abundance and spatial distribution of target MXene particles, residual precursor phases, and contaminant populations to be determined across representative sample areas (Figure 1b)¹⁰.

Elemental mapping is particularly useful in this context because it can relate the morphology of individual particle populations to their local elemental distributions. Measurements performed before and after electrochemical testing can additionally reveal compositional changes associated with degradation and interphase formation^{33, 34}. In $\text{Ti}_3\text{C}_2\text{T}_x$ samples, SEM resolves the morphologies of the different particle populations, whereas co-registered EDS maps provide information on the distributions of elements such as carbon, oxygen, fluorine, aluminum, and titanium (Figure 1a). However, manually inspecting and quantifying these maps across sufficiently large particle populations is impractical. MXene quality control therefore illustrates the broader need for an automated approach that can segment morphological features

in SEM images and quantify their associated EDS signals without discarding feature-level spatial information.

Here, we introduce SEMEX.Lab, a lightweight and data-efficient framework that combines deep-learning-based segmentation of SEM images with mask-guided analysis of co-registered EDS maps (Figure 1c). Morphological masks extracted from the SEM channel are transferred to the elemental maps, enabling automated, morphology-resolved quantification of elemental signals at the level of individual particles and features. This approach preserves associations between morphology and composition that are lost when EDS signals are averaged over entire images or manually selected regions. It also enables large feature populations to be evaluated systematically, reducing analyst-dependent sampling and supporting statistically representative compositional analysis.

To address the scarcity and cost of annotated experimental micrographs, we further develop a canvas-based strategy for generating realistic synthetic SEM training data. The method constructs new micrographs by combining mosaic backgrounds assembled from image sub-patches with object instances drawn from a curated morphology pool. Unlike conventional copy-paste augmentation³⁵, which places objects onto existing training images, the canvas-based strategy produces entirely synthetic training images in which object densities and class proportions can be controlled independently. It does not require the separate training and optimization of generative adversarial networks (GANs)^{36, 37} or diffusion models^{38, 39} and avoids the need to construct explicit three-dimensional particle models for physics-based image rendering⁴⁰⁻⁴². The strategy thereby expands training diversity while maintaining low annotation and computational requirements.

Using heterogeneous $\text{Ti}_3\text{C}_2\text{T}_x$ samples as a proof of concept, we demonstrate automated segmentation and morphology-resolved elemental analysis of target MXene particles, residual precursor phases, and contaminant populations using limited manually labelled data. The segmentation model comprises 15 million parameters and processes each image in approximately 1.3 s on a standard CPU, enabling direct

deployment on existing SEM workstations without dedicated GPU hardware or external inference servers. Benchmarking against representative recent segmentation approaches places the method in a low-data, low-computational-cost regime, consistent with its intended use as an accessible and deployable microscopy analysis framework (Figure 2).

Although demonstrated here using MXene SEM-EDS datasets, the analytical principle is not specific to MXenes. The framework uses a structure- or morphology-bearing image channel and spatially co-registered signal maps; it can therefore be adapted to other heterogeneous materials examined by SEM-EDS. In principle, the same strategy could also be extended to other multimodal microscopy methods, including multichannel atomic force microscopy, in which signals from co-registered channels must be quantified according to feature morphology at defined spatial scales.

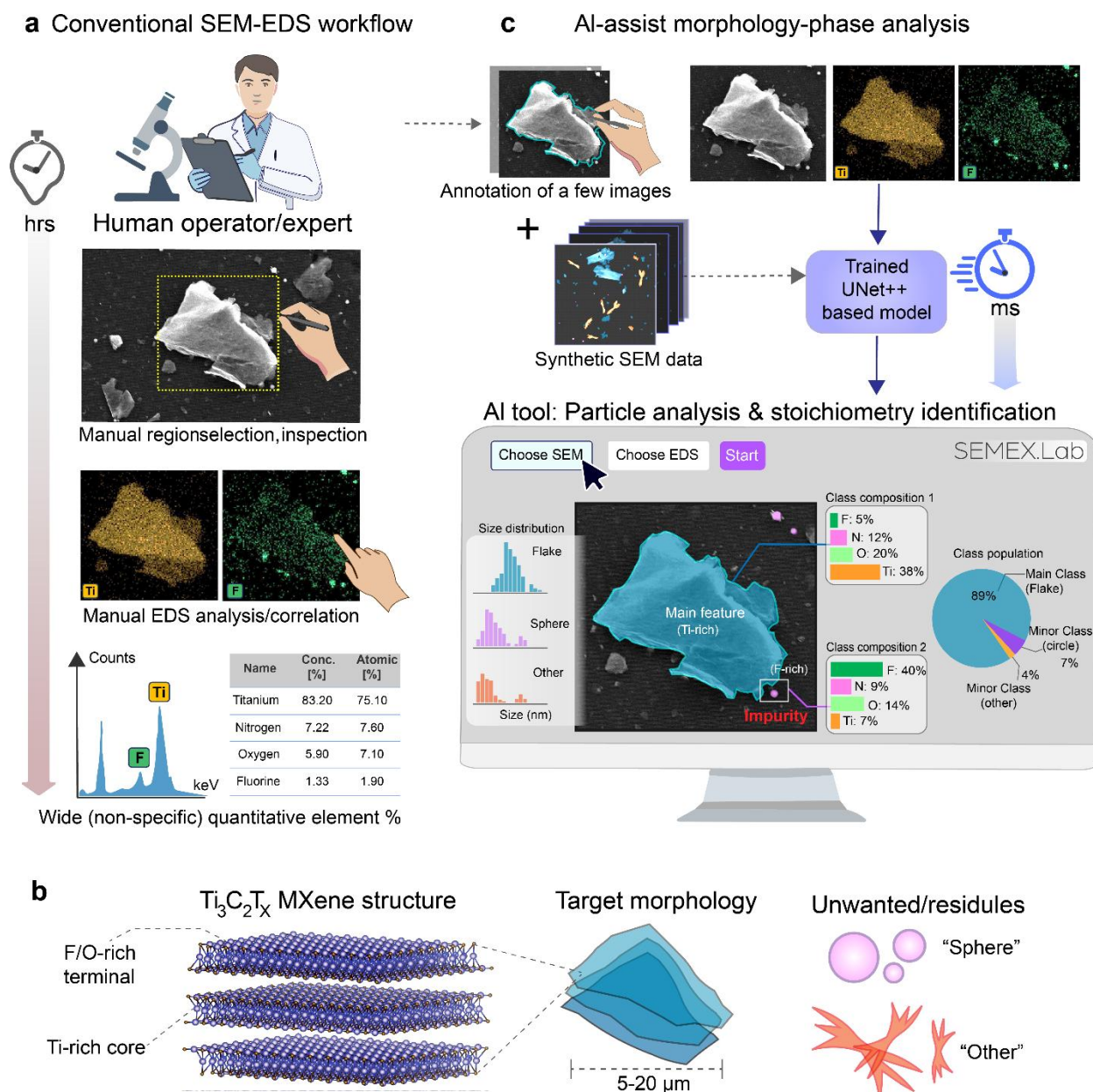


Figure 1. Conventional SEM–EDS workflow versus the proposed AI-driven automated morphology–phase analysis. (a) $\text{Ti}_3\text{C}_2\text{T}_x$ MXene layered structure with target flake morphology and unwanted residues present as spheres and other shapes. (b) *In the conventional workflow*, an expert manually selects regions of interest, inspects features, and correlates SEM morphology with EDS elemental maps (e.g., Ti, F). The output is a single field-averaged elemental composition, with no breakdown by particle morphology. The analysis typically takes hours per sample. (c) *In the proposed workflow*, a segmentation model, trained on

a small set of annotated SEM images together with synthetic data, takes SEM images and co-acquired EDS maps as input and performs inferences in ~ 1.3 s on a standard CPU or 34 ms on a GPU. Implemented in SEMEX.Lab, the model segments morphological classes (Flake, Sphere, Other) and reports stoichiometry and population statistics for each class.

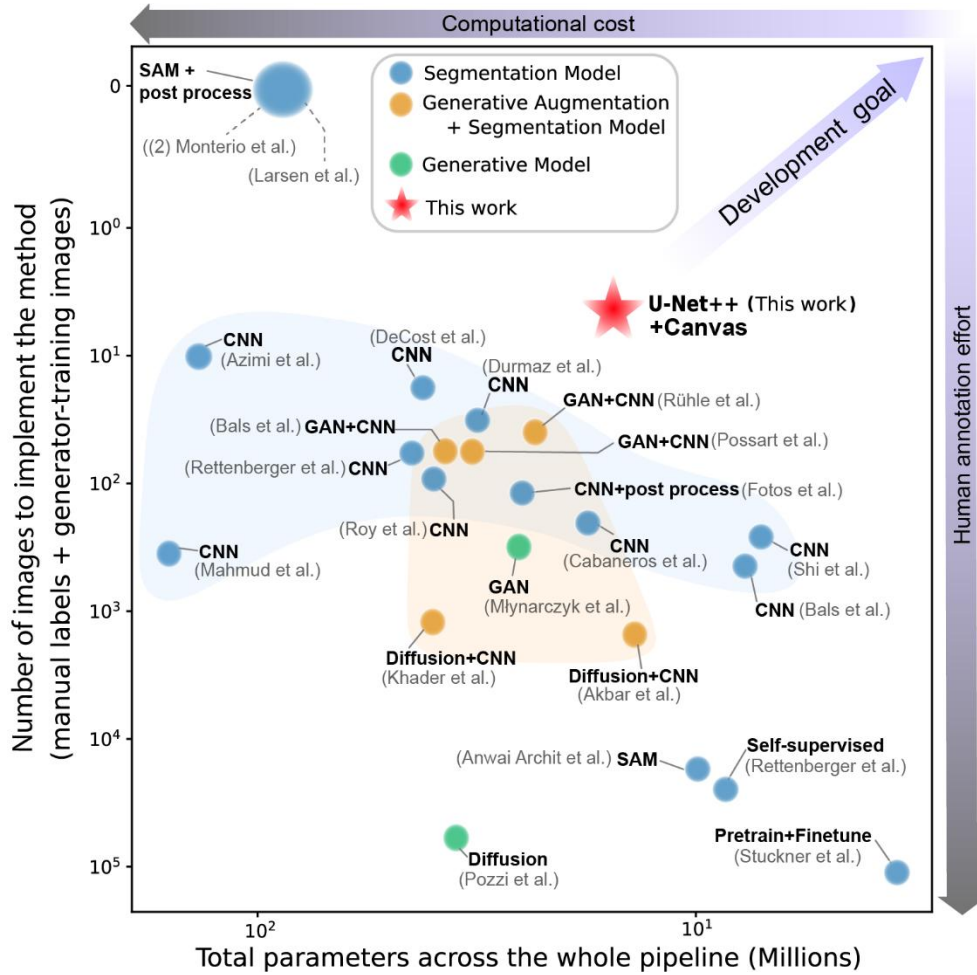


Figure 2. Positioning our work within the broader research landscape. Map positioning our method against representative recent approaches along two axes: the number of images required to prepare the method (manual labels plus generator-training images; vertical axis) and the total number of parameters across the whole pipeline (horizontal axis). A lower parameter count indicates lower computational cost, and a smaller number of required images indicates less human effort. Our method occupies the low-computational-cost, low-human-effort region: the direction we identify as the development goal. Each

plotted method is labeled with its corresponding reference^{12, 14-16, 19, 20, 36-39, 43-55}; the underlying parameter counts and image requirements for all methods are listed in Supplementary Table S1.

2 Results

We present a SEM–EDS analysis framework that takes unlabeled SEM micrographs and co-acquired EDS maps as inputs and produces morphology-resolved compositional statistics. A canvas-based strategy expands a handful of labeled images into a synthetic training dataset, a lightweight segmentation model trained on this set assigns each pixel to a morphology class, and the predicted masks are then intersected with EDS maps to recover morphology-specific elemental compositions. The framework was developed using an SEM dataset of 2D energy-storage materials acquired under consistent imaging conditions, with co-acquired EDS elemental maps collected for selected regions of interest. A fixed set of 70 manually annotated images, containing around 11,220 morphological objects, was held out for validation and testing. For training, we deliberately varied the number of manually annotated SEM images from zero to ten to evaluate the framework's performance in the low-data regime.

To enable training in this low-data regime, we generated a synthetic dataset (D') using the canvas-based strategy to augment the real annotated images D (Section 2.1). We first evaluated the segmentation performance by comparing models trained on real data alone (D) against those trained on real and synthetic data ($D \cup D'$) (Section 2.2). We then assessed model robustness under varying training conditions: initial real image count, synthetic image count, objects per synthetic image, and class ratio (Section 2.3). Finally, we performed qualitative and quantitative morphology-resolved EDS analysis using the segmented SEM masks to link morphological classes to their elemental composition (Section 2.4).

2.1 Segmentation pipeline with synthetic training data

Training semantic segmentation models typically requires hundreds or thousands of manually annotated images, a significant barrier for materials-science applications. Our pipeline reduces this requirement to as few as 5 labeled images, combined with synthetic data generated by the canvas-based strategy.

Our segmentation pipeline consists of three stages, summarized in Figure 3. First, high-throughput SEM acquisition produces a large pool of unlabeled micrographs of the target energy-material samples. An expert annotator selects a small subset, between zero and ten images in the experiments reported below and provides pixel-level labels. Each object is assigned to one of three morphology classes: Flake, Sphere or Other, forming the labeled dataset D (Figure 3a). Second, we extract the labeled objects from D using its segmentation mask, organizing them into a class-specific morphology pool. For each synthetic image, a new background is constructed by tiling random crops of 10×10 pixels drawn from the background regions of D in a mosaic pattern until a canvas of 1024×1024 pixels is filled. Objects are then sampled from the morphology pool and pasted onto this background at random positions, producing a set of fully labeled synthetic images D' (Figure 3b). When objects overlap, they are layered in a fixed order, Flake at the bottom, Sphere in the middle, Other on top, so that rarer morphologies remain visible above more common ones. Third, a segmentation model is trained on the union of the labeled images D and the synthetic set D' (Figure 3c). Figure 4 compares representative synthetic images with real SEM micrographs, showing close visual similarity across the three morphology classes. At inference, the trained model segments unlabeled SEM images in ~ 1.3 s per image on a standard CPU and ~ 34 ms per image on an RTX 3090 GPU. The resulting masks feed into the downstream morphology-resolved EDS analysis described in *Section 2.4* (Figure 7). Full implementation details for each stage are given in Methods (*Section 4.3*).

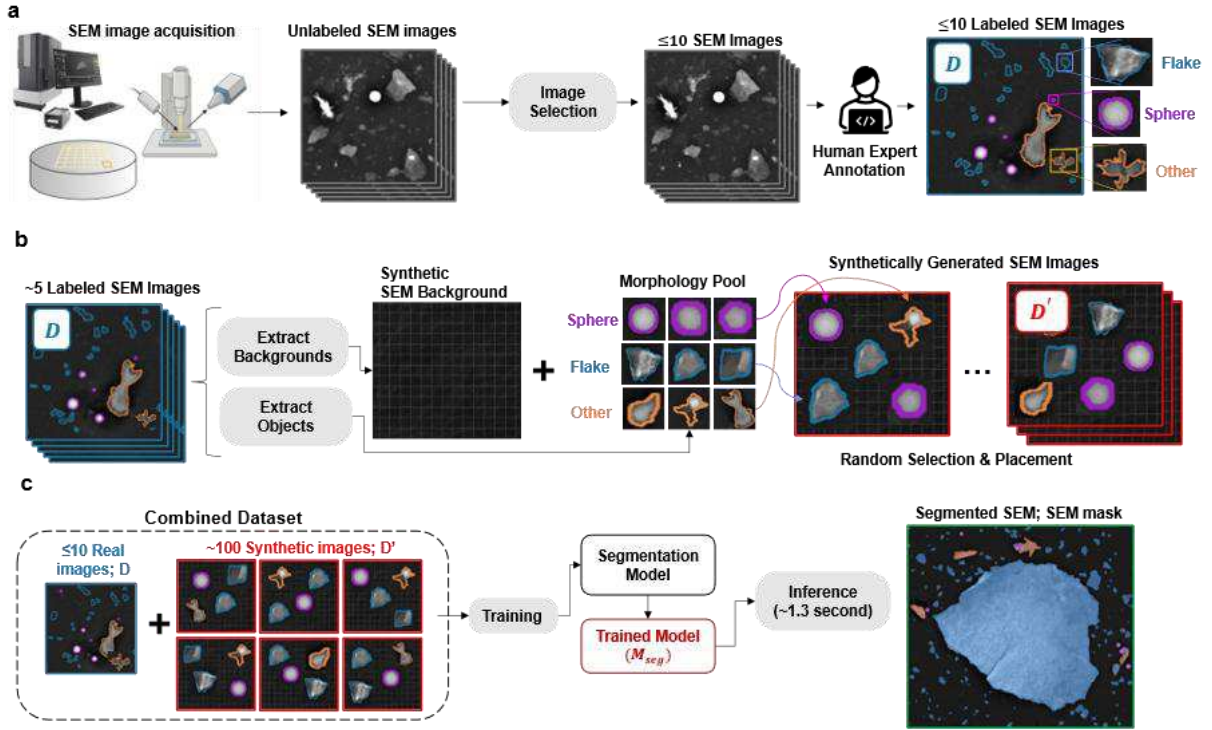


Figure 3. Overview of the automated, morphology-resolved SEM characterization pipeline. (a) *Data acquisition and annotation.* SEM images are acquired under consistent imaging conditions (see Section 4); a small representative subset (~ 5 images) is selected and pixel-level annotated by a human expert into three morphology classes — Flake (blue), Sphere (purple), and Other (orange) — yielding the labeled dataset D . (b) *Synthetic data generation.* Object instances are extracted from D to build a class-specific morphology pool. In parallel, 10×10 -pixel patches are sampled from the background regions of D and tiled in a mosaic pattern to construct a 1024×1024 -pixel canvas. Objects from the morphology pool are randomly placed onto the canvas, producing the synthetic dataset D' . (c) *Model training and inference.* The combined dataset ($D \cup D'$) is used to train the segmentation model M_{seg} , producing pixel-wise semantic masks at inference (~ 34 ms per image on a consumer GPU; ~ 1.3 s on a standard CPU).

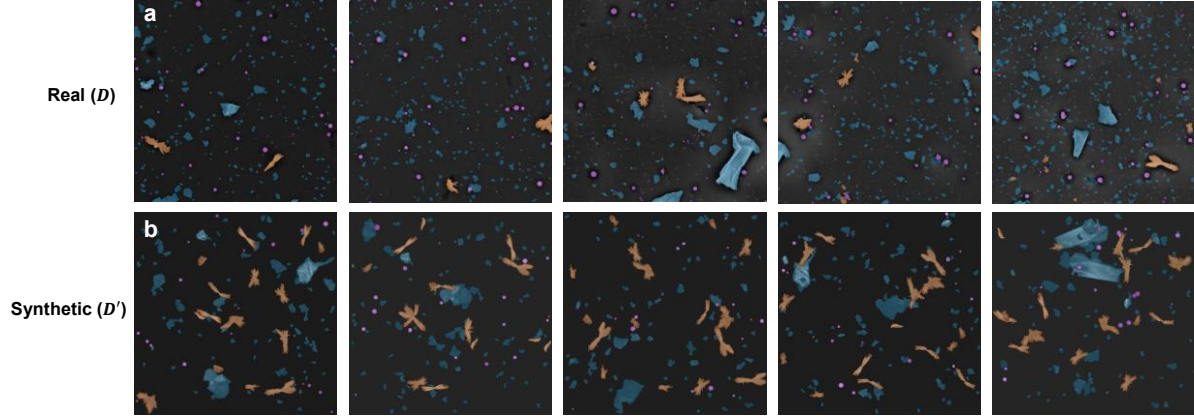


Figure 4. Comparison of real and synthetic SEM training images. (a) Representative real SEM images from the labeled dataset D , with overlaid morphology classes: Flake (blue), Other (orange), and Sphere (purple). (b) Representative synthetic images generated by the canvas-based strategy (D') using five real annotated SEM images, shown with the same overlay scheme. Additional examples are provided in Supplementary Figure S2.

2.2 Synthetic data improves segmentation with minimal labeled examples

We first evaluated whether training with synthetic data D' alongside real labeled D data improves segmentation performance. We trained a U-Net++ model⁵⁶ on $D \cup D'$ and compared it with the same architecture trained on D alone. The choice of the number of real labeled images ($|D| = 5$), synthetic images ($|D'| = 100$), and objects per synthetic image ($k = 60$) is justified by the ablation study in *Section 2.3*. Here, we use these settings to first establish the overall benefit of synthetic data. To ensure a fair comparison, we performed three independent runs with different random seeds, covering synthetic dataset generation, model training, and inference, while keeping the validation and test sets fixed across all runs. The model trained with $D \cup D'$ achieved a mean Intersection-over-Union (mIoU) of 0.743 ± 0.003 compared with 0.62 ± 0.04 for the baseline trained on D alone, and a mean Dice coefficient of 0.845 ± 0.002 compared with

0.73 ± 0.06 , an relative improvement of approximately improvement 19.8 %, and 15.8 % in mIoU and Dice coefficient, respectively (Table 1).

The benefit of synthetic data is highly non-uniform across morphology classes. The Flake class gains only 4 % improvement in IoU and 2 % improvement in Dice. This modest improvement reflects the dominance of flakes in the source data. The instance pool extracted from D contains 473 Flakes compared to 89 Spheres and 15 Other particles – a roughly 32:6:1 ratio. Even five real images therefore expose the model to abundant flake instances and flake boundary pixels, placing the baseline trained on D alone close to the achievable performance ceiling for this class and leaving little headroom for synthetic data to contribute. In contrast, the Sphere class gains 28 % in IoU and the Other class gains 97 % with parallel trends in Dice (17 % and 85 %, respectively).

Figure 5 shows qualitative comparisons between the model trained with D and with $D \cup D'$ on three representative test images. The model trained with D frequently misclassifies small spherical particles and irregular fragments, particularly in dense regions (Figure 5, red dashed boxes), while the model trained with $D \cup D'$ correctly recovers these structures (Figure 5, solid green boxes). The model trained with $D \cup D'$ is also markedly more reproducible across runs: the standard deviation of the Other-class IoU drops by over an order of magnitude, from 0.2 to 0.01, and the standard deviation of mIoU drops from 0.04 to 0.003. Such variance reduction is critical for practical deployment, where users need confidence that a model trained on only a handful of labeled images will behave consistently across runs. Having established that synthetic data substantially improves both accuracy and uncertainty at $|D| = 5$, we next examine how this improvement depends on the choice of synthetic data generation parameters.

To confirm that these gains stem from the canvas-based approach rather than from synthetic data generation in general, we also compared the canvas-based strategy against a standard augmentation-only baseline, in which geometric and photometric augmentations (Table S2) were applied to the same five

real images. The canvas-based strategy outperformed this baseline by 32 % in mIoU (Supplementary Note 1; Figure S3).

Table 1. Adding synthetic data improves segmentation, with the largest gains for under-represented classes. Performance of U-Net++ trained on (i) real annotated SEM images only (D , $|D|=5$) versus (ii) real images combined with synthetic images ($D \cup D'$), $|D'| = 100$, $k = 60$ objects per image, Flake:Sphere:Other = 1:1:1; parameter choices justified in *Section 2.3*, evaluated on a held-out test set of 35 images (4,865 annotated objects). (a) Specific class and mean Intersection-over-Union. (b) Specific class and mean Dice coefficient. mIoU and mDice are averaged over the four classes (Flake, Sphere, Other, Background). Values are mean $\pm$ s.d. across three independent training runs. Denote M_D and $M_{D \cup D'}$ as the metric (mIoU or Dice) of the model trained on D and on $D \cup D'$, respectively. The relative improvement is: $\frac{M_{D \cup D'} - M_D}{M_D} \times 100$

(a) Intersection-over-Union					
Training regime	mIoU	IoU Flake	IoU Sphere	IoU Other	IoU Background
Real only (D)	0.62 ± 0.04	0.714 ± 0.003	0.53 ± 0.05	0.3 ± 0.2^1	0.956 ± 0.001
Real + Synthetic ($D \cup D'$)	0.743 ± 0.003	0.741 ± 0.006	0.680 ± 0.007	0.59 ± 0.01	0.961 ± 0.001
Relative improvement (%)	+19.8	+3.78	+28.3	+96.7	+0.52
(b) Dice coefficient					
Training regime	mDice	Dice Flake	Dice Sphere	Dice Other	Dice Background
Real only (D)	0.73 ± 0.06	0.833 ± 0.002	0.69 ± 0.04	0.4 ± 0.3	0.977 ± 0.001
Real + Synthetic ($D \cup D'$)	0.845 ± 0.002	0.851 ± 0.004	0.810 ± 0.005	0.74 ± 0.01	0.980 ± 0.001
Relative improvement (%)	+15.8	+2.16	+17.4	+85	+0.31

¹ the Other class has few samples, so its baseline is highly uncertain.

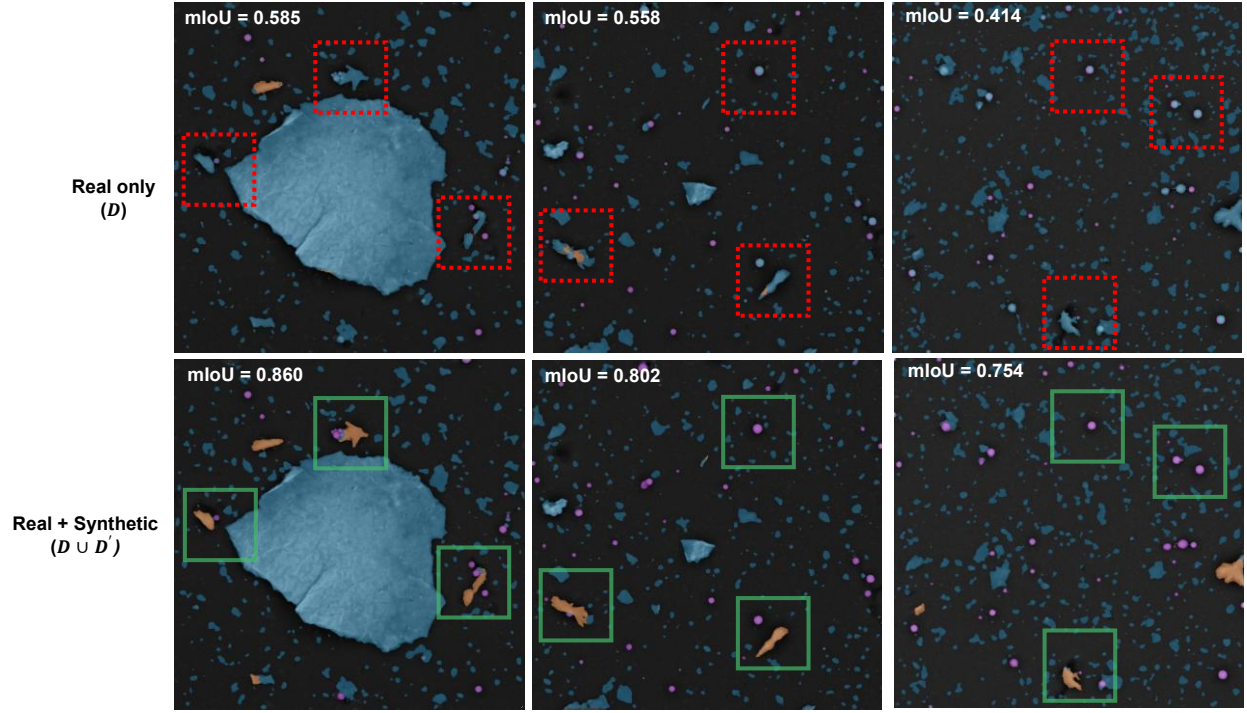


Figure 5. Qualitative comparison of segmentation results. Each column shows one representative test image. The top row shows predictions from the model trained on real data only (D); the bottom row shows predictions from the model trained on real data combined with synthetic data ($D \cup D'$). Red dashed boxes mark objects misclassified by the D model; solid green boxes mark the same regions correctly segmented by the $D \cup D'$ model. Per-image mIoU is reported in the top-left corner of each panel. Segmentation masks are color-coded by morphology class: Flake (blue), Sphere (purple), and Other (orange).

2.3 Ablation study of the synthetic data generation pipeline

To identify the conditions under which the synthetic data pipeline is most effective, we systematically varied four generation parameters: (Q1) the number of real labeled images $|D|$, (Q2) the number of synthetic images $|D'|$, (Q3) the number of objects packed into each synthetic image (k), and (Q4) the class ratio among Flakes, Spheres, and Others. Default values and tested ranges for all generation parameters are summarized in Table S3; full numerical results for each ablation are listed in Tables S4–S8. The quantitative answers reported below are specific to our SEM imaging conditions and target morphologies. While we

expect the qualitative trends to transfer to other particle-like SEM datasets, this remains to be verified. All experiments used three independent random seeds and were evaluated on the same held-out test set. The effects of background canvas size and 2D rotation augmentation are analyzed in Supplementary Note 2 and 3 (Figure S4 and S5, respectively).

(Q1) *How many real labeled images are needed?* We varied $|D|$ from 1 to 10 and trained the segmentation model on the combined dataset $|D \cup D'|$ (Figure 6a, Table S4). Performance rises sharply between 1 and 5 real labeled images, from an mIoU of 0.48 ± 0.01 to 0.743 ± 0.003 , and plateaus thereafter in the range 0.743–0.768 up to $|D| = 10$. The gain is largest at $|D| = 2$ (67 % improvement in mIoU), confirming that the pipeline extracts useful diversity even from minimal labeled input. Together, these results indicate that the morphology pool saturates quickly and that a small number of well-chosen real images capture most of the morphological variability of the sample. We therefore fix $|D| = 5$ for all subsequent experiments. The per-class breakdown (Table S5) shows this saturation is driven mainly by the harder classes: rare morphologies such as Sphere and Other benefit most from the added synthetic data, whereas Flake and Background are already well captured at small $|D|$. It is the smallest value at which performance saturates and corresponds to approximately 2.5 h of expert annotation, a labeling budget compatible with routine SEM workflows.

(Q2) *How many synthetic images are needed?* Having fixed $|D| = 5$, we next asked whether the synthetic set D' itself saturates. We varied $|D'| \in \{50, 100, 150, 200, 300, 400, 500\}$ and trained the segmentation model on $D \cup D'$. Increasing $|D'|$ from 50 to 100 raised the mean mIoU from 0.70 ± 0.04 to 0.743 ± 0.003 and reduced the run-to-run standard deviation by over an order of magnitude (Figure 6b, Table S6), indicating that $\lesssim 100$ synthetic images do not cover the variation needed for stable training. Beyond this point, the mean mIoU stabilized in the range 0.72–0.75 up to $|D'| = 500$. The largest single-step gain therefore occurs between 50 and 100 synthetic images: 6.1 % improvement in mIoU and $\sim 14\times$ variance reduction). We therefore fix $|D'| = 100$ as the default in all subsequent experiments.

The plateau in mIoU beyond $|D'| = 100$ reflects a diversity ceiling inherited from D . Both the morphology pool and the mosaic backgrounds are derived from D . The pool is extracted from labeled instances and the backgrounds are tiled from 10×10 -pixel patches of unannotated regions so that the effective diversity of D' is bound by the content of D . Once $|D'|$ is large enough to span the combinatorial space of instance placements and background crops, further synthetic images only reshuffle the same source content and contribute no new morphological information. The relevant axis for further scaling is therefore the diversity of D , not the size of D' .

(Q3) *How densely should objects be packed into each synthetic image?* Holding all other generation parameters fixed, we varied the objects-per-image count $k \in \{10, 30, 60, 120, 300\}$. As shown in Figure 6c, performance rises sharply from $\text{mIoU} = 0.39 \pm 0.09$ at $k = 10$ to 0.743 ± 0.003 at $k = 60$, and plateaus thereafter up to $k = 300$ (Table S7).

The steep gain in segmentation performance between $k = 10$ and 30 reflects the scarcity of object instances and boundary pixels per image, which provides the model too few examples per synthetic image. Once enough objects are present per image to provide a sufficient training signal, further increases in k no longer improve performance. Although $k = 60$ is already sufficient to saturate segmentation performance, we adopt $k = 120$ ($\text{mIoU} = 0.751 \pm 0.009$) as the final default for all subsequent experiments and the deployed pipeline, because it matches the mean object density of our real SEM micrographs (~ 120 objects per 1024×1024 -pixel field).

(Q4) *How should the class ratio be chosen?* Fixing $k = 120$ and holding all other generation parameters at their defaults (defaults; $|D| = 5$, $|D'| = 100$, $k = 120$), we varied the Flake:Sphere:Other ratio across seven settings: 1:1:1, 3:1:1, 5:1:1, 1:3:1, 1:5:1, 1:1:3, and 1:1:5. Three models were trained per setting from independent random seeds.

Segmentation performance was largely insensitive to moderate Flake/Sphere imbalance: mIoU varied by less than 0.03 across the five settings 1:1:1, 3:1:1, 5:1:1, 1:3:1, and 1:5:1 (Figure 6d, Table S8). In contrast,

raising the Other ratio to 1:1:3 and 1:1:5 degraded mIoU from 0.751 (1:1:1 ratio) to 0.74 ± 0.02 and 0.72 ± 0.01 , respectively. We attribute this to the layering order described in *Section 2.1*: Other morphologies are pasted on top and therefore occlude any underlying Flake or Sphere pixels. At fixed k , increasing the Other fraction progressively covers the Flake and Sphere boundaries that carry informative learning signal, leaving less for the model to learn from.

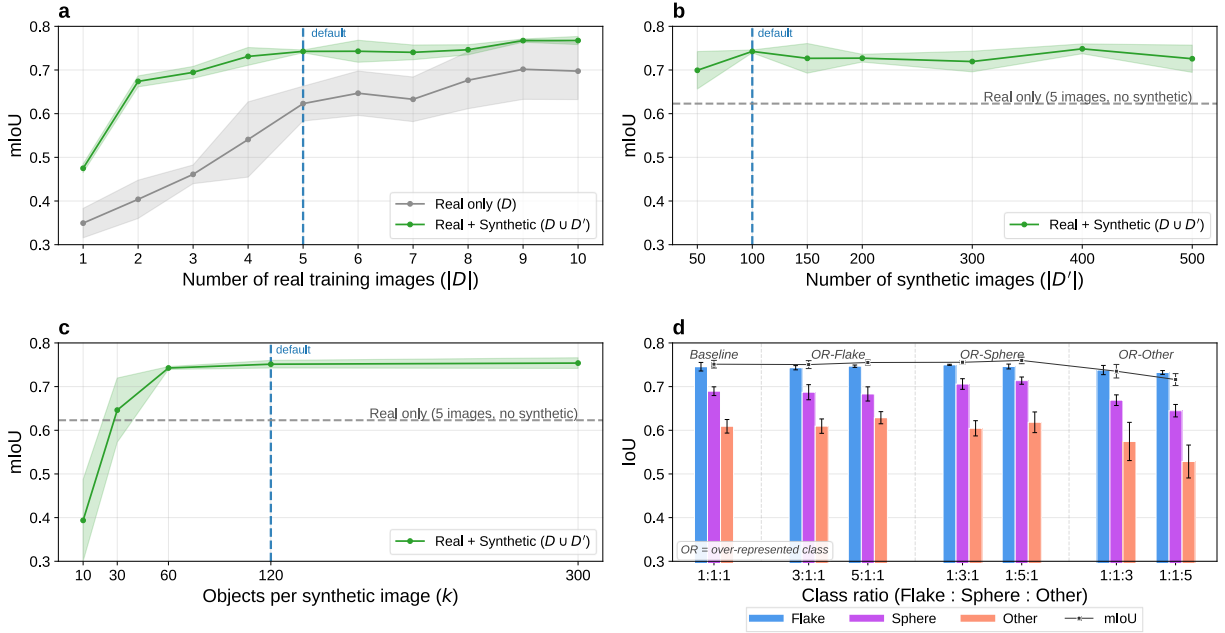


Figure 6. Ablation of synthetic data generation parameters. (a) Dependence of mIoU on the number of real training images ($|D|$). Comparison of models trained on real data only (D) and on real data combined with synthetic data ($D \cup D'$). (b) Dependence of mIoU on the synthetic set size ($|D'|$), with $|D| = 5$. (c) Dependence of mIoU on the number of objects per synthetic image (k), with $|D| = 5$ and $|D'| = 100$. (d) Class specific IoU and mIoU across class ratios (Flake:Sphere:Other), grouped by over-represented (OR) class (*Baseline*, *OR-Flake*, *OR-Sphere*, *OR-Other*). Vertical dashed lines in (a–c) mark default settings; horizontal dashed lines in (b) and (c) mark the model trained on real data only (D). Shaded regions and error bars indicate the standard deviation across three independent runs.

2.4 Morphology-resolved elemental analysis

By integrating the segmentation mask with EDS elemental maps, the framework enables the extraction of particle-level compositional statistics that are inaccessible through conventional analysis in such a time-efficient manner. These quantitative analyses are demonstrated through SEMEX.Lab, a user interface (UI) showcase built on top of the segmentation pipeline that accepts raw SEM and co-acquired EDS images as inputs and returns morphology-resolved compositional statistics without requiring any programming expertise. Rather than functioning merely as a statistical data extraction tool, SEMEX.Lab serves as an interpretive platform that enables a physics-grounded understanding of material systems, including heterogeneous samples in which morphologically distinct particle populations may share the same elemental identity, or, conversely, in which particles of similar shape may belong to entirely different phases.

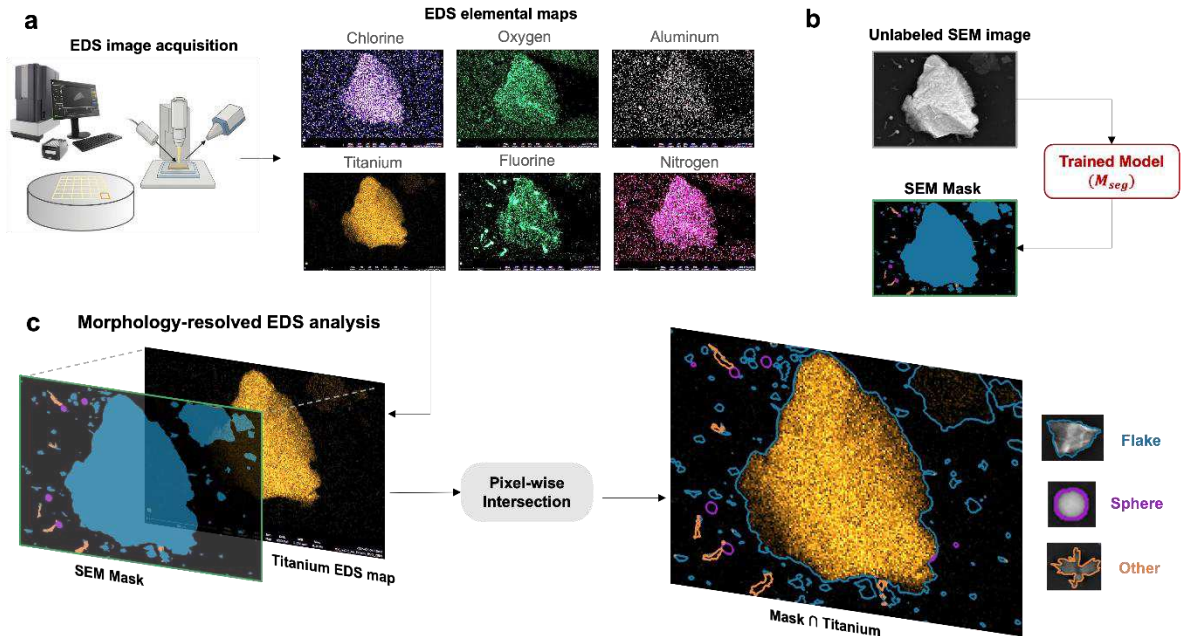


Figure 7. Workflow for morphology-resolved EDS analysis. (a) SEM-EDS acquisition produces an SEM image and co-acquired EDS elemental maps for six elements (Cl, O, Al, Ti, F, N). (b) The unlabeled

SEM image is passed through the trained segmentation model M_{seg} (Section 2.2), which assigns every pixel to one of three morphology classes, Flake (blue), Sphere (purple), or Other (orange), yielding a semantic mask. (c) Morphology-resolved EDS analysis is performed by the pixel-wise intersection of each class mask with each elemental map, yielding the elemental signal attributable to each morphology class. The figure example illustrates the titanium case ($\text{Mask} \cap \text{Ti}$).

To demonstrate the practical application of our framework, we applied our trained segmentation model to 9 SEM images with corresponding EDS elemental maps associated with Ti, O, Al, Cl, N, and F elements (Figure 7). The trained model rapidly segmented each image in less than one second, producing morphology-specific masks (Flake, Sphere, and Other) that were then intersected pixel-wise with each EDS elemental map (according to Equation 5, section 4.6). Segmentation mask alignment with elemental signal boundaries (as shown in Figure 8) indicates that the predicted masks precisely align with the spatial extent of each morphological class for downstream compositional analysis.

Across the 9 test images, the pipeline identified a total of 718 particles, including 469 Flakes (86.4% of the total segmented areas), 72 Spheres (9.83%), and 177 Other particles (3.76%), as reported in Table 2. This particle-count breakdown is itself a meaningful result. In a typical materials synthesis process targeting MXene flakes, other morphologies (i.e., Sphere and Other) represent non-target products or impurities, such as unreacted precursors, oxidized phases, or side-reaction products. Our framework measures these populations at the individual-particle level, allowing for direct estimation of the synthesis yield, which cannot be determined by conventional field-averaged EDS or bulk characterization techniques. This meaningful information provides an overall image of the synthesis quality that is directly relevant to material or device performance^{4, 8}.

The *within-morphology elemental composition* further reveals the chemical identities of the different morphology classes (Table 2 and Figure 9a). Within the Flake class, Ti is the dominant element at 37.8%,

followed by O at 20.2% and N at 11.6%, consistent with the titanium carbide/nitride-based chemistry expected for MXene flakes³. In contrast, the Sphere-class is enriched in O, Al, and F, suggesting an aluminum-rich oxide phase, which is likely residual Al₂O₃ or Al(OH)₃ from incomplete MXene precursor phase (MAX-phase) etching during synthesis⁵⁷. The Other class exhibits an intermediate and more fluorine-rich content (F: 18.9%, O: 28.2%, Al: 16.6%), possibly from unreacted LiF precursor or residues such as AlF₃^{30,33} and NaAlF₆³. *Bulk quantification* (Figure 9b) confirms the dominance of the Flake class populations and its primary Ti element (up to 98.6% of total Ti signal in the sample). In contrast, the Sphere class is the minority, with about 10% of the total Al or F found to be dominant in the Sphere class. This level of quantification is impossible by conventional field-averaged EDS. The atomic-percent counterpart is shown in Supplementary Figure S6, with full atomic stoichiometries referenced to the least-abundant element in Supplementary Tables S9–S11.

Importantly, our framework goes beyond this first-level compositional identification. Typically, in a heterogeneous sample, the different morphologies often share a similar stoichiometry. They can present a similar chemical composition but have adopted different shapes due to different growth kinetics, decomposition, or aggregation during synthesis⁵⁸. Our SEMEX.Lab platform captures this meaningful information explicitly by pairwise elemental similarity across morphology classes (see Figure 10 and Supplementary Video 1). Finally, SEMEX.Lab can point out cases in which distinct morphologies are compositionally comparable, efficiently guiding the user towards deeper interpretation of materials that may co-exist within a multi-phase system rather than distinct impurities. Critically, this similarity-based grouping represents a qualitative interpretive level beyond what statistical EDS analysis alone provides.

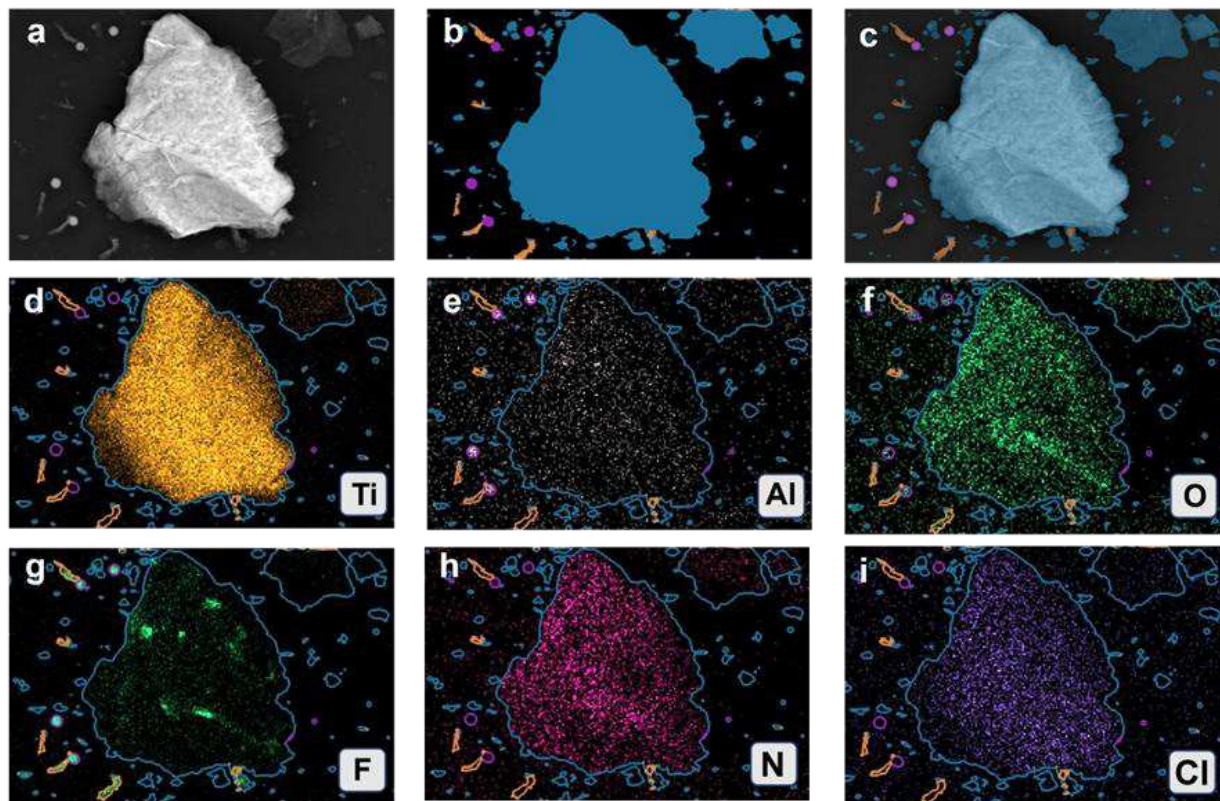


Figure 8. Elemental EDS maps with overlaid morphology contours for a representative SEM example. (a) Original SEM image. (b) Predicted segmentation mask with morphology classes: Flake (blue), Sphere (purple), and Other (orange). (c) Overlay of the segmentation mask on the original SEM image. (d–i) Elemental EDS maps overlaid with segmentation contours for Titanium (d), Aluminum (e), Oxygen (f), Fluorine (g), Nitrogen (h), and Chlorine (i).

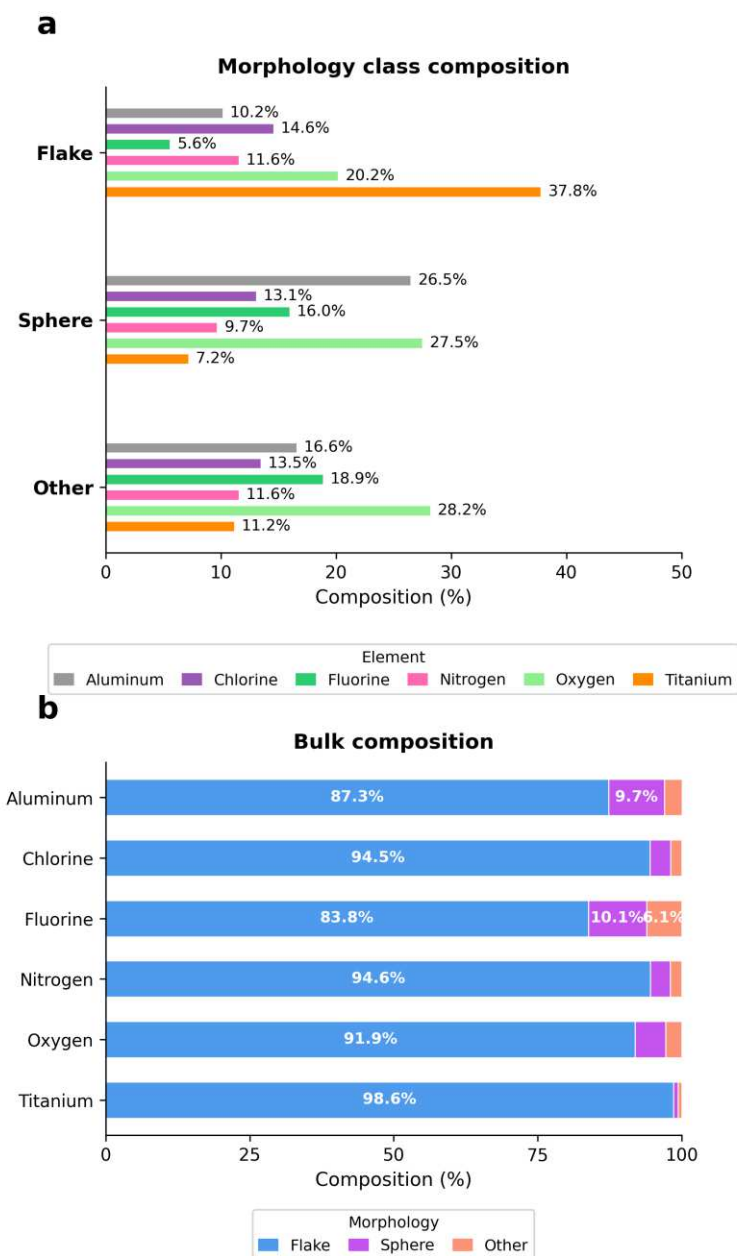


Figure 9. Morphology-resolved elemental analysis across particle classes. Elemental statistics aggregated from 718 segmented particles across 9 SEM micrographs with co-acquired EDS elemental maps. (a) *Morphology class composition*. For each morphology class (Flake, Sphere, Other) the six element bars (Al, Cl, F, N, O, and Ti) add up to 100 %, revealing the stoichiometry of each particle type. (b) *Bulk composition*. For each element, the values indicate the percentage of that element's total signal that

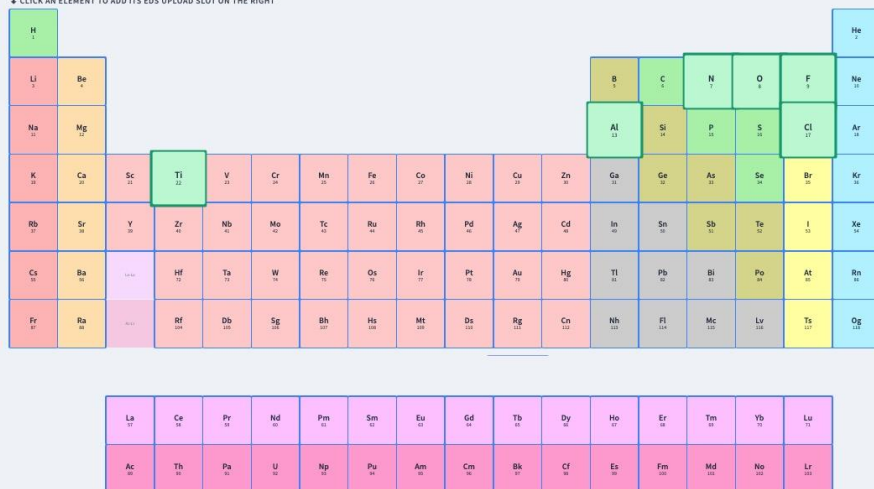
originates from Flakes, Spheres, and Other particles. For each element, the three morphology values add up to 100%. Numerical values are listed in Table 2.

Table 2. Elemental composition resolved by particle morphology. Elemental statistics aggregated over 718 segmented particles from 9 SEM micrographs with co-acquired EDS elemental maps for Ti, O, Al, Cl, N, and F. *Morphology class composition (top)*: for each morphology class (Flake, Sphere, Other), the six element values sum to 100% and represent the relative elemental composition of that particle type across the six mapped elements. *Bulk composition (bottom)*: for each element, the values show what percentage of that element's total signal originates from Flakes, Spheres, and Other particles. Rows sum to 100% for each element. *N objects* = number of segmented particles per class; *Area fraction (%)* = fraction of total segmented particle area occupied by each class.

Morphology	N objects	Area fraction (%)	Ti	O	Al	Cl	N	F
Morphology class composition (%)								
Flake	469	86.4	37.8	20.2	10.2	14.6	11.6	5.6
Sphere	72	9.83	7.2	27.5	26.5	13.1	9.7	16.0
Other	177	3.76	11.2	28.2	16.6	13.5	11.6	18.9
Total	718	100.0	—	—	—	—	—	—
Bulk composition (%)								
Flake	—	—	98.6	91.9	87.3	94.5	94.6	83.8
Sphere	—	—	0.8	5.3	9.7	3.6	3.4	10.1
Other	—	—	0.6	2.8	3.1	1.9	2.0	6.1

Run inference

✓ Image 32.jpeg



EDS IMAGES (CLICK ELEMENT FIRST)

Fluorine upload ✓ Image 32_analysis_1_map_F...

Oxygen upload ✓ Image 32_analysis_1_map,...

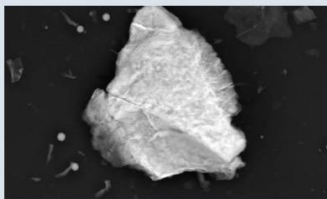
Nitrogen upload ✓ Image 32_analysis_1_map_...

Aluminum upload ✓ Image 32_analysis_1_map_A...

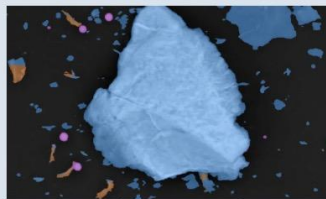
Chlorine upload ✓ Image 32_analysis_1_map_C...

Titanium upload ✓ Image 32_analysis_1_map_T...

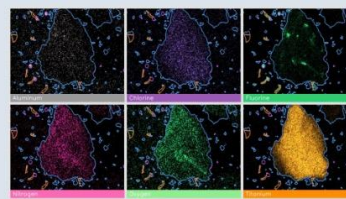
SEM (CROPPED)



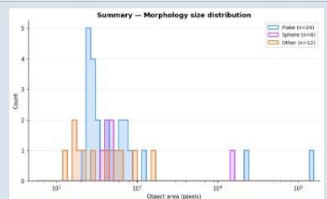
MASK OVERLAY ON SEM



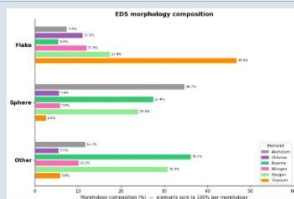
EDS OVERLAYS



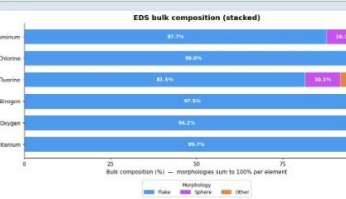
MORPHOLOGY SIZE HISTOGRAM



EDS MORPHOLOGY COMPOSITION



EDS BULK COMPOSITION (STACKED)



RUN SUMMARY

Summary

Class #	Class	Objects	Area %	Dominant element	Stoichiometry	Similar to	Cosine sim. (≥ 0.85)
1	● Flake	24	89.1%	● Titanium (46.8%)	Ti3.6AlN3.104FC11.1	—	0.472
2	● Sphere	6	8.1%	● Aluminum (34.7%)	TiAl24.1N7.8028.1F27.1C12.9	● Other	0.868
3	● Other	12	2.8%	● Fluorine (36.1%)	TiAl3.6N6015.9F15.7C11.3	● Sphere	0.868

Device: CUDA - Inference size: 1024x1024 - Display crop: 30px/side - Stoichiometry excludes Carbon (substrate contamination)

Figure 10 SEMEX.Lab interactive interface for morphology-resolved SEM–EDS analysis. Representative screenshot of the deployable dashboard, illustrating the image analysis view applied to a single SEM–EDS acquisition.

3 Discussion

We have presented a lightweight, data-efficient framework for morphology-resolved SEM–EDS analysis. Training a 15 M-parameter U-Net++ on five labeled MXene micrographs together with synthetic data generated by our canvas-based strategy, the model reaches an mIoU of 0.743 ± 0.003 with an inference time of ≈ 1.3 s per image on a standard CPU. Its three-class masks, intersected pixel-wise with co-acquired EDS maps, recover morphology-specific compositional statistics inaccessible to field-averaged EDS.

The sufficiency of five labeled images, when expanded through the canvas-based synthetic pipeline, is not specific to this dataset. It reflects a structural property of SEM micrographs that show discrete morphologies on a relatively uniform background. Object morphologies are position-invariant: a flake-shaped particle has the same geometric signature whether it lies in the corner or center of the field of view. A small instance pool, once extracted, can therefore be reused across thousands of synthetic placements. Sample backgrounds were found to be statistically homogeneous within a single image acquisition session. A 10×10 -pixel mosaic tiled from real background regions therefore reproduces the correct distribution. Together, these properties let the pipeline compose new images, yielding a synthetic dataset whose pixel statistics match those of the source instrument and sample.

This design contrasts with three alternative strategies for SEM data augmentation. First, generative adversarial models, such as the simulated-particle GAN^{19, 36, 55, 59}, produce realistic SEM-like micrographs but require training a separate generator on a dedicated input dataset. Second, copy-paste augmentation³⁵, by contrast, adds new objects to existing training images. The augmented image retains the background and overall scene composition of the original, so that its object density and class ratio are fixed by the source.

Third, physics-based rendering of explicit 3D particle models⁴⁰⁻⁴² yields fully controllable scenes but requires external software such as Blender and demands detailed geometric and imaging models of the sample. Our canvas-based strategy keeps the simplicity and label-preserving nature of copy-paste while generating fully synthetic, fully labeled images, where object density, spatial arrangement, and class balance are tunable by design. Combined with a lightweight, data-efficient U-Net++ for SEM-EDS morphology segmentation, this yields a practical solution: controllable, scalable SEM augmentation without the overhead of full GAN pipelines or external physics-based rendering software, and with more explicit control than standard copy-paste.

A closely related effort is UTILE-Gen⁶⁰, which shares our copy-paste-style approach to synthetic data generation. Our methods differ in two respects. First, UTILE-Gen treats shape classification and instance segmentation as separate image-analysis tasks, whereas we use a single semantic-segmentation model and recover per-particle statistics through connected-component analysis, prioritizing CPU-deployable simplicity. Second, and most importantly, UTILE-Gen operates purely on morphological image content and does not incorporate compositional information. By coupling each predicted morphology mask with co-acquired EDS maps, our framework resolves elemental composition per morphology class. This capability is absent from prior synthetic-data microscopy pipelines and central to the use cases we target.

Microscopy datasets often contain many large/common particles but relatively few small or rare morphologies. This creates class imbalance, so networks trained only on real images tend to miss or under-segment these rare structures. Synthetic data pipelines directly target this gap. The direct consequence of a well-designed synthetic-data pipeline is a stronger, more balanced training signal for small and rare particle types, leading to clearly improved segmentation of these classes compared with training on real, imbalanced images alone. Without such augmentation (or other imbalance countermeasures), small “Sphere” and “Other” morphologies carry too little signal, so networks tend to under-segment or ignore them.

The choice of U-Net++ with a ResNet-18 encoder reflects a deliberate prioritization of deployability over peak accuracy. The 15 M-parameter network runs in ≈ 1.3 s per image on CPU, so the framework deploys directly on standard SEM workstations without dedicated GPU hardware.

The synthetic-data pipeline is equally lightweight, requiring only five labeled images and a simple copy-paste procedure with no auxiliary generative model to train. The cost of this simplicity is most visible in the rare Other class, which plateaus near $\text{IoU} = 0.59$. With only 15 Other particles at $|\mathcal{D}| = 5$, the copy-paste method can reuse these few instances across many synthetic placements but cannot create new Other morphologies, so the achievable accuracy for this class is capped by the diversity of the labeled pool. Enlarging the labeled set is the most direct way to raise this ceiling, and adopting a higher-capacity model such as the Segment Anything Model^{61, 62} is a second option, but both conflict with our deployability target. Indeed, the Segment Anything Model and its microscopy variant^{20, 43}, trained on the same five labeled images, underperformed our pipeline despite $\sim 6\times$ more parameters (Table S12; see Supplementary Note 4 for the setup and full comparison), confirming that our gains stem from the synthetic-data pipeline rather than model size. Because the synthetic-data component is architecture-agnostic, substituting a larger backbone remains a straightforward extension, and this becomes increasingly valuable as models grow. We verify this directly in Supplementary Note 5 (Figure S7), where the same pipeline improves segmentation accuracy across six different architectures, showing that its benefit is not tied to any single model. Training a larger model on only a handful of real MXene SEM–EDS images risks memorizing those few micrographs, so a rich and controllable synthetic set from the canvas-based strategy becomes important: it prevents this memorization, it covers the morphology and class-imbalance variations that a large model could otherwise overfit, and it lets the added capacity be spent on genuine generalization rather than noise fitting. However, this does not remove the need for real labels: the diversity of the synthetic set is bound by the labelled pool, so as the model scales up, the number of real labelled images must increase as well, to keep supplying genuinely new morphology rather than reshuffled copies of the same few instances.

The framework is positioned as a deployable interface that can be layered seamlessly on top of standard instrumentation. Nevertheless, there are limitations inherent to this approach. At this time, the framework's validation remains narrow as it has been demonstrated on a single two-dimensional energy-storage material acquired on a single instrument. A substantial body of research in the fields of microscopy and medical imaging demonstrates that such models frequently undergo a precipitous degradation when transferred across different microscopies and imaging or mapping protocols. This decay is attributed to variations in noise and texture statistics. Consequently, cross-instrument and cross-material generalization remains to be explored, particularly for heavily clustered morphologies or textured backgrounds that violate our position-independence assumptions. To demonstrate true generality and move beyond this proof of concept, it is necessary for future work to establish robustness through pretraining on diverse microscopy data or tailored domain-generalization methods.

The pixel-wise intersection of segmentation masks with co-acquired EDS maps reveals compositional heterogeneity invisible to field-averaged analysis. The Flake class is Ti-dominant (37.8%) with moderate O (20.2%), consistent with $\text{Ti}_3\text{C}_2\text{T}_x$ MXene. Spheres are depleted in Ti (7.2%) and enriched in Al (26.5%), O (27.5%), and F (16.0%), pointing to Al-O based residuals, while the Other class shows elevated F (18.9%) that is suggestive of fluoride-based residues. Bulk partitioning confirms that 98.6% of the total Ti signal originates from Flakes, whereas Al and F are disproportionately concentrated in the minority populations that a distinction field-averaged EDS cannot make. Grouping materials by phase, the Sphere and Other classes share broadly similar stoichiometries, that are both Ti-depleted and Al/O/F-enriched, leading to the question of whether they represent distinct phases or morphological variants of the same incompletely etched precursor observed by pairwise elemental similarity analysis as demonstrated in our SEMEX.Lab interface (Figure 10). This meaningful information could guide users toward targeted follow-up measurements such as X-ray diffraction (XRD). The EDS elemental maps used in this work represent raw X-ray intensity counts per pixel as acquired in imaging mode and have not been subjected to Atomic Number Effect (Z), Absorption Effect (A), and Fluorescence Effect (F) or ZAF correction; the reported

compositional values should therefore be interpreted as semi-quantitative relative ratios rather than absolute atomic percentages.

The SEMEX.Lab UI (Figure 10) visualizes these MXene and impurity populations, countable and compositionally analyzed by class in one click. The SEMEX.Lab provides three levels of insight inaccessible to conventional SEM–EDS workflows: (i) morphological yield in the population-weighted fraction of the target phase by particle count and area; (ii) class-specific elemental quantification with intrinsic composition of each morphology, enabling phase identification and comparison to known reference compounds; and (iii) phase compositional similarity analysis by grouping of morphologically distinct populations that share the same elemental identity, enabling the user to distinguish true phase diversity from morphological evolution. For high-throughput deployment, a complementary bulk-mode interface (Supplementary Figure S8) processes entire image folders in a single operation, returning the same class-specific statistics aggregated across thousands of images, as demonstrated on a 2,589-image corpus comprising over 360,000 segmented particles in total (Supplementary Figure S9).

Conventional SEM–EDS analysis is slow, expert-driven, and often limited to bulk composition or a handful of manually picked particles, so morphology–composition links are rarely explored systematically^{63, 64}. Manual micrograph analysis typically requires measuring hundreds of particles by hand^{47, 65}, and existing automated SEM-EDS tools still rely on predefined classes and rule-based thresholds rather than flexible learning from data^{63, 64, 66}. An AI-driven framework that (i) segments particles automatically, (ii) uses those masks to guide EDS sampling/interpretation, and (iii) runs at CPU-friendly throughput across thousands of images, directly tackles these bottlenecks.

In Mxene research, Machine Learning and AutoML studies have already accelerated the screening of MXene compositions and electrochemical performance using simulated or tabular data⁶⁷⁻⁷⁰. Feeding these models with high-volume, morphology-resolved SEM-EDS descriptors that automatically extracted across thousands of particles would bridge microstructure to performance, supporting rapid optimization and

inverse design. In other applications such as high-value materials and nanodevices, automated SEM-based mineralogy quantifies size, geometry and liberation of phases for process control in ores, slags, and ceramics⁶³. A CPU-deployable morphology–composition tool generalizes this idea to catalysts, nanodevices, and environmental particles, where particle size/shape plus local chemistry dictate functionality^{49, 71, 72}.

By combining data-efficient deep segmentation, synthetic training data, and mask-guided SEM-EDS, our framework extends prior AI-SEM work from “morphology only”^{49, 65, 71, 73} or “rule-based SEM-EDS”^{63, 64, 66} to automated, morphology-resolved compositional mapping at scale as a key step toward routine, closed-loop characterization and quality control in MXenes, batteries, and broader advanced materials manufacturing.

4 Methods

4.1 Sample preparation and SEM-EDS acquisition

MXene titanium carbide material was synthesized from MAX phase (Ti_3AlC_2) according to the previous method⁷⁴. A Phenom XL G3 Desktop SEM equipped with Energy-dispersive X-ray (EDS) detector (Thermo Fisher Scientific Inc., CeB₆ electron source) was used to collect SEM images and EDS maps, scanning the large sample area at a cm^2 scale. The sample was prepared by drop casting MXene suspension (in water) on a glassy carbon plate ($1 \times 1 \text{ cm}^2$ area). The SEM images were acquired using beam acceleration Voltage of 15 kV and beam intensity set at IMAGE MAP (intermediate intensity), using the Backscatter electron detector (BSD). The Energy-dispersive maps were collected using the X-ray (EDS) detector instrument model MVE080522-10768-L, and electron beam acceleration Voltage of 20 kV and beam intensity set at MAP (maximum intensity).

4.2 Dataset and annotation

Datasets. A single high-throughput SEM acquisition run was performed on the MXene sample under the conditions described in *Section 4.1*, yielding 2,679 SEM images. From this single acquisition pool, three disjoint subsets were defined and used for distinct purposes throughout the study (Table S13):

Dataset 1 — Annotated SEM corpus (model development). Eighty SEM images were selected from the acquisition pool and annotated by two human experts using the Supervisely platform², requiring approximately 30 minutes per image. Each particle was annotated as one of three morphology classes: Flake, Sphere, or Other. Flakes are thin, sheet-like particles characteristic of MXene; spheres are round or near-spherical particles; and the Other class includes irregular fragments and structures that do not match either of the first two classes. Dataset 1 contained 12,486 annotated objects in total (11,290 Flakes, 984 Spheres, and 212 Others). Images were acquired at a native resolution of 1920×1275 pixels and resized to 1024×1024 pixels prior to training and inference. The 80 images were split into 10 training, 35 validation, and 35 test images. The training split was deliberately selected by selecting the 10 most representative images, mirroring the practical deployment scenario in which a user identifies a small set of images that best capture the morphological diversity of the sample for use with our synthetic data pipeline. Dataset 1 is the only dataset used for synthetic data generation (*Section 2.1*), model training (*Section 2.2*), and the ablation study of generation parameters (*Section 2.3*).

Dataset 2 — SEM-EDS sets (morphology-resolved compositional analysis). Nine SEM images were drawn from the acquisition pool; for each image, co-acquired EDS elemental maps were collected for six elements (Ti, Al, O, F, Cl, N). EDS mapping was acquired only for this subset because of its substantially longer acquisition time per image compared with SEM imaging alone. These images carry no human-annotated segmentation masks. The masks here are obtained at inference time from the model trained on

² <https://supervisely.com> (accessed May 2026).

Dataset 1. Dataset 2 is used exclusively as a demonstration of morphology-resolved EDS analysis (*Section 2.4*) and is not used for any model training.

Dataset 3 — Bulk SEM corpus (deployment-scale demonstration). Dataset 3 consists of the remaining 2,589 SEM images from the acquisition pool, used to demonstrate the framework's throughput under realistic high-throughput conditions. These images carry neither human-annotated masks nor co-acquired EDS data. Like Dataset 2, segmentation masks are obtained at inference time from the model trained on Dataset 1. Dataset 3 is used solely to report aggregate, bulk-scale segmentation statistics, and is not used for any model training.

4.3 Synthetic data generation

The pipeline is illustrated in Figure 3b. From each annotated image in D , connected-component analysis is applied to the segmentation masks to extract individual particles, which are stored together with their binary masks in a per-class morphology pool. Particles with an area below 100 pixels are discarded to exclude annotation noise. At the default $|D| = 5$, this procedure yields a pool of 473 Flakes, 89 Spheres, and 15 Other particles.

The background of each synthetic image is constructed by mosaic tiling. For each synthetic image, one source image is randomly selected from D , and 10×10 pixel patches sampled from its background regions are tiled until a 1024×1024 canvas is filled. Particles are then sampled from the morphology pool and placed at uniformly random positions on the canvas. Sampling proportions are controlled by a configurable class ratio Flake:Sphere:Other, so that the per-image class distribution is independent of the pool size. Each particle is randomly rotated in 2D before placement; no scaling or photometric augmentation is applied. When particles overlap, they are layered with Flake at the bottom, Sphere in the middle, and Other on top, with the same ordering applied consistently to the image pixels and the segmentation mask. Placement continues until k particles have been pasted onto the canvas.

Default settings used throughout the paper, unless otherwise stated, are $|D| = 5$ real annotated images, $|D'| = 100$ synthetic images, $k = 120$ particles per synthetic image, and a Flake:Sphere:Other class ratio of 1:1:1. The comparison in Section 2.2 (Table 1) and the ablations in Q1–Q2 were performed with $k = 60$, the setting used prior to the object-density ablation (Q3), which established $k = 120$ as the final default.

4.4 Segmentation model architecture and training

We used U-Net++⁵⁶ with a ResNet-18⁷⁵ encoder pre-trained on ImageNet⁷⁶, implemented in PyTorch. This architecture was chosen for its favorable accuracy-to-parameter trade-off, with approximately 15M parameters enabling CPU-deployable inference on standard SEM workstations without dedicated GPU hardware. The network was trained to segment four classes: Background, Flake, Sphere, and Other. Input images were resized to 1024×1024 pixels and normalized using ImageNet mean and standard deviation; the same deterministic preprocessing was applied to training, validation, and test splits. Random 2D rotation is incorporated at the morphology level during synthetic data generation (*Section 2.1*). Training used the Adam optimizer with an initial learning rate of 1×10^{-4} , batch size of 4. The loss function was standard cross-entropy without class weighting. Class balance was instead controlled by the 1:1:1 sampling ratio in the synthetic pipeline. A ReduceLROnPlateau scheduler halved the learning rate when validation loss failed to improve for two epochs. Training was terminated by early stopping with a patience of five epochs, up to a maximum of 300 epochs. All models were trained on a single NVIDIA RTX 3090 GPU; a typical run completed in approximately 20 minutes. Three random seeds (1, 52, 88) were used across all experiments for reproducibility. The complete training configuration is summarized in Table S14.

4.5 Evaluation metrics

Segmentation performance was quantified using the mean Intersection-over-Union (mIoU) and mean Dice coefficient, computed as the unweighted average of per-class IoU and Dice across all four classes (Background, Flake, Sphere, Other). For each class c , the per-class IoU and Dice are defined as

$$\mathbf{IoU}_c = \frac{\mathbf{TP}_c}{(\mathbf{TP}_c + \mathbf{FP}_c + \mathbf{FN}_c)} \quad 1$$

$$\mathbf{Dice}_c = \frac{\mathbf{TP}_c}{(2\mathbf{TP}_c + \mathbf{FP}_c + \mathbf{FN}_c)} \quad 2$$

where $\mathbf{TP}_c$, $\mathbf{FP}_c$, and $\mathbf{FN}_c$ denote the number of true-positive, false-positive, and false-negative pixels for class c , respectively. The mean metrics are obtained by averaging across all four classes:

$$\mathbf{mIoU} = \frac{1}{C} \sum_{c=1}^C \mathbf{IoU}_c \quad 3$$

$$\mathbf{Dice} = \frac{1}{C} \sum_{c=1}^C \mathbf{Dice}_c \quad 4$$

with $C = 4$. Background was included in the mean to reflect overall pixel-level segmentation quality. All metrics were computed on the held-out test set of 35 images (4,865 annotated objects) and are reported as mean $\pm$ standard deviation across three independent training runs with three random seeds.

4.6 Morphology-resolved EDS analysis

To obtain elemental statistics resolved by particle morphology, the segmentation masks predicted by the trained model M_{seg} were combined with co-acquired EDS elemental maps through pixel-wise intersection; see Figure 7. EDS maps for six elements (Ti, Al, O, F, Cl, and N) were acquired simultaneously with 9 selected SEM images. Each EDS map E_e is a 2D image whose pixel value reports the X-ray intensity for element e . For each SEM image, the image itself and its co-acquired EDS maps were resampled to a common 1024×1024 pixel to ensure pixel-level alignment.

The resampled SEM image was passed through M_{seg} to obtain a semantic mask assigning every pixel to one of three morphology classes. For each morphology class $c \in \{Flake, Sphere, Other\}$ and each element e , the EDS signal attributable to that morphology was obtained by pixel-wise intersection of the binary class mask Ω_c with the elemental intensity map E_e :

$$\mathbf{I}_{c,e} = \Omega_c \cap E_e \quad 5$$

where $\cap$ denotes pixel-wise multiplication, Ω_c is the binary mask of class c , and $I_{c,e}$ is the resulting intensity image of element e restricted to pixels assigned to class c . The total X-ray intensity of element e attributable to class c is then obtained by summing over all pixels:

$$S_{c,e} = \sum I_{c,e} \quad 6$$

From the per-class intensities, two complementary views were derived:

Morphology class composition (Figure 9a). For each morphology class c , the relative contribution of element e is

$$f_{c,e}^{class} = \frac{S_{c,e}}{\sum_{e'} S_{c,e'}} \times 100 \% \quad 7$$

where the sum in the denominator runs over all six elements, so that the six element fractions sum to 100 % within each class. This view characterizes the elemental composition of each particle type.

Bulk composition (Figure 9b). For each element e , the fractional contribution of morphology class c to the total signal is

$$f_{c,e}^{bulk} = \frac{S_{c,e}}{\sum_{c'} S_{c',e}} \times 100 \% \quad 8$$

where the sum in the denominator runs over all three morphologies, so that the three morphology fractions sum to 100 % per element. This view splits the bulk EDS signal of each element into contributions from each morphology class.

Particle-level statistics — number of objects per class and the image area fraction occupied by each class — were obtained from connected-component analysis of the predicted masks. The values reported in Fig. 8 and Table 2 are relative EDS intensity fractions computed directly from raw X-ray intensity maps without ZAF correction and should therefore be interpreted as semi-quantitative compositional ratios rather than absolute atomic percentages.

The same procedure can be applied to arbitrarily large SEM–EDS image sets without modification; the per-image inference time of ~1.3 seconds on CPU (~34 ms on GPU) makes the analysis practical for high-throughput SEM datasets.

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Bannapol Limanond; Method, data preparation, coding, visualization, writing original draft, review & editing.

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Kajjana Boonpalit; Data preparation and annotation.

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9 Competing Interests

The authors assert that they have no conflicts of interest or personal relationships that could have influenced the work reported in this paper.

10 Data and Code Availability

The data and code supporting this study are archived on Zenodo (DOI:10.5281/zenodo.21638779). *For review purposes, the record can be accessed here. This link will be replaced by open access to the record upon acceptance.*

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