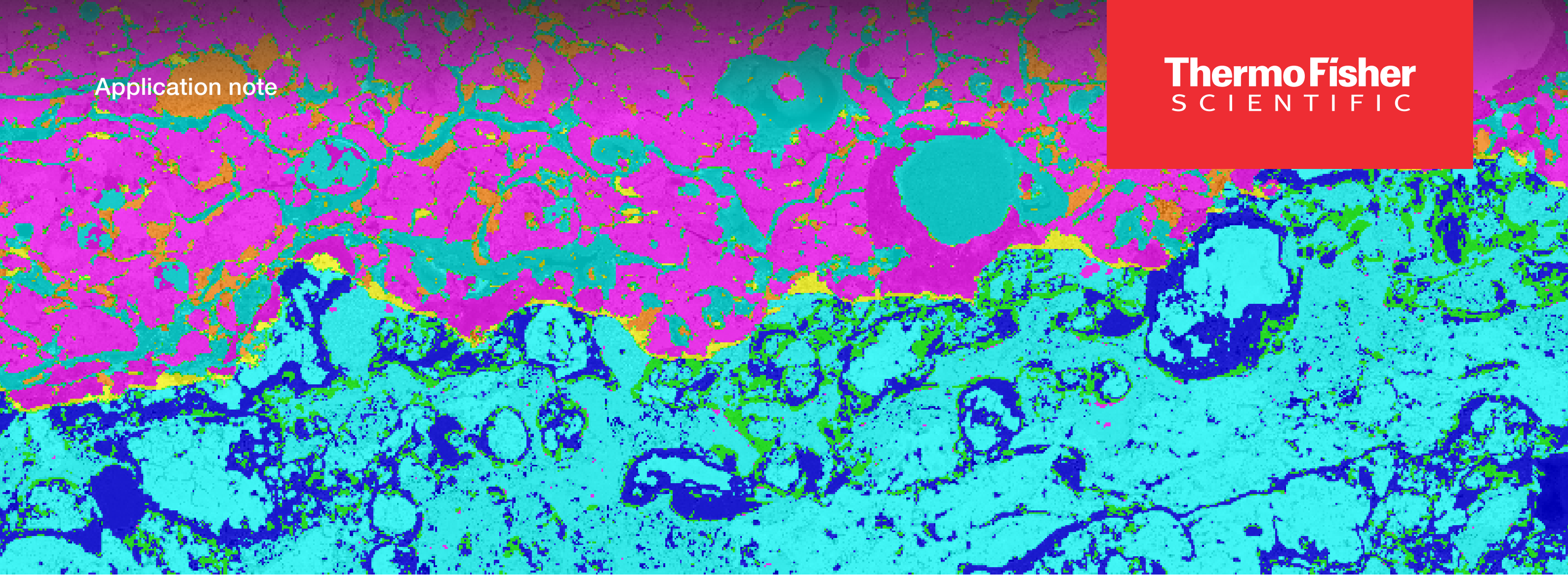


# Correlative microscopy and AI-assisted image analysis of high-temperature-application coatings


## Authors

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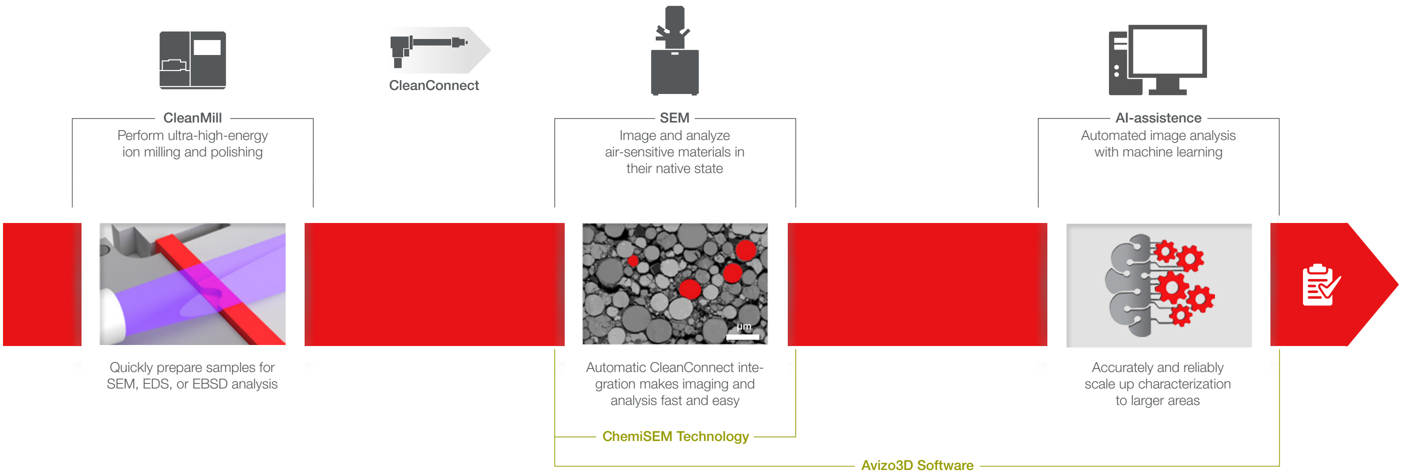
## Introduction

Thermal barrier coatings (TBCs) provide insulation and protection for parts used in a variety of high-temperature applications, finding particular use in gas turbines as well as other aerospace, transportation, and military components. The strategic importance of TBCs lies in their ability to enhance energy efficiency, prolong component life, and reduce maintenance costs. Characterization and understanding of TBCs enables faster and more accurate assessment of their properties and performance under service conditions. Recent advancements in correlative microscopy and AI-assisted image analysis allow for the integration of multiple imaging modalities, providing a comprehensive understanding of the material's microstructure and composition. Consequently, this holistic approach facilitates a more thorough investigation of TBCs, leading to more efficient design and optimization processes that are critical for high-performance applications.

This application note demonstrates a synergistic approach that integrates correlative microscopy with AI-assisted image analysis to improve the time-to-results for TBC characterization. Specifically, a cross section of a TBC from the afterburner of a SR-71 Blackbird is examined after it had been in service for more than 15,000 hours, in order to see how such a material degrades over many thermal cycles and over many decades of use. Automated sample polishing, scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), and advanced AI algorithms are consolidated into a single process that takes the sample from surface polishing through to data acquisition and analysis.

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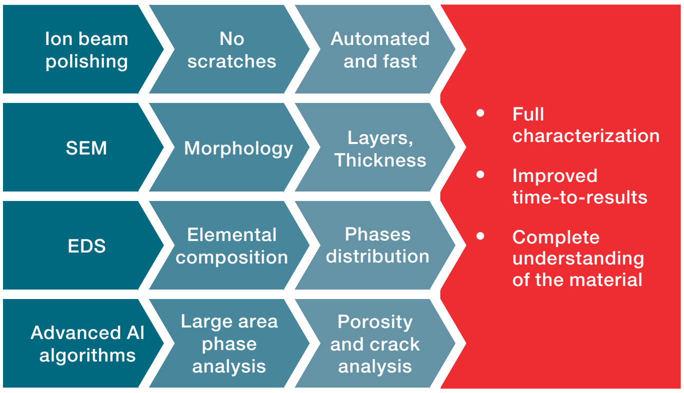
This method utilizes the Thermo Scientific™ Apreo™ ChemiSEM, a scanning electron microscope with unique Thermo Scientific™ ChemiSEM™ Technology, in combination with Thermo Scientific™ Avizo™ 3D Pro Software and the Thermo Scientific™ CleanMill™ Broad Ion Beam System. The integration of these technologies not only accelerates analysis but also enhances the accuracy and reliability of final results.




## Efficient broad ion beam system improves time-to-results

TBCs are multi-layered systems typically consisting of a ceramic top coat, a bond coat, and thermally grown oxides. The effectiveness of TBCs depends on several factors including their morphology, chemistry, as well as their physical and mechanical properties. These characteristics collectively determine the performance and longevity of TBCs in service environments.

Traditional methods of TBC sample preparation are time-consuming and often require multiple steps and systems for cutting, embedding, grinding, and polishing, together with the manual skills needed to perform these steps. Obtaining high-quality surfaces can therefore be a lengthy and complex process, especially when the TBC consists of several layers made of different materials with variable densities.

For this reason, the sample of interest has been prepared using the CleanMill Broad Ion Beam System, an ultra-high-energy ion source that enables rapid surface and cross-section polishing. The CleanMill System reduced the sample preparation time from up to 12 hours down to 90 minutes.

**Figure 1. CleanMill Broad Ion Beam System.**

The results of the CleanMill sample preparation are shown in Figure 2. The backscatter electron (BSE) image of the TBC shows a flat surface, with pores and crack that are clearly not the result of sample preparation but rather the material's extreme working conditions. (When in service, this TBC is subjected to hot gasses close to 1,800°C along with cooling air of about 600°C.)

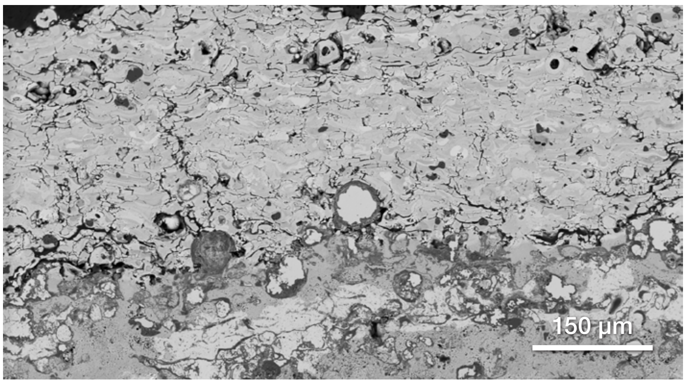
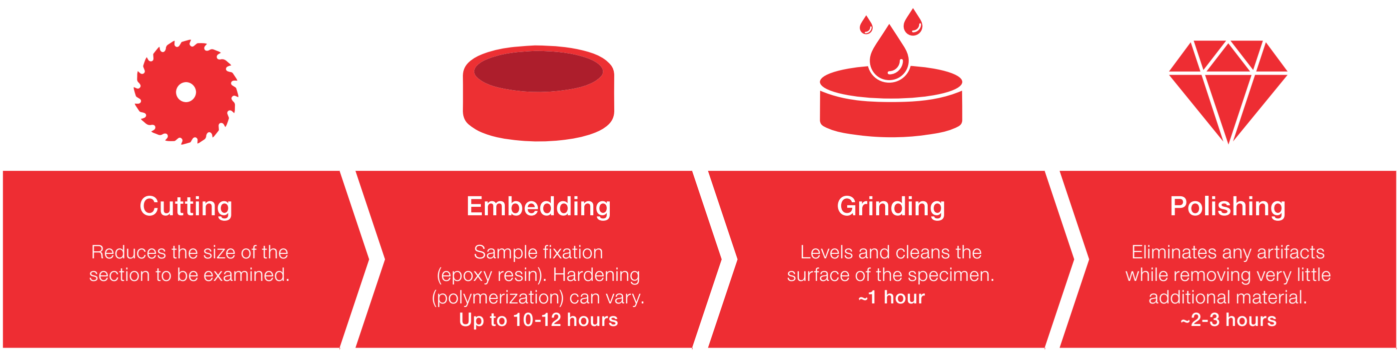



**Figure 2. BSE image of the TBC surface, showing several layers of different materials, together with pores and cracks.**



## Top coat characterization

ChemiSEM Technology played a pivotal role in the overall characterization of the layers. As it is fully integrated into the imaging workflow, it provides complete chemical characterization while significantly reducing analysis time. This integration allows for the rapid identification and quantification of all the materials present.

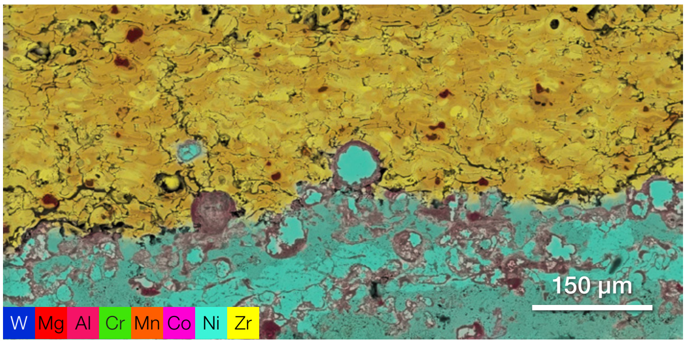



**Figure 3. Large-area ChemiSEM characterization of the area shown in Figure 2.**

The large-area EDS characterization shown in Figure 3 was performed as the sample was imaged in order to obtain an initial overview of the elemental distribution. The ChemiSEM analysis clearly differentiates between the bond coat and top coat. The layer distribution shows that the interfaces are not flat, and that this TBC suffered some elemental diffusion and phase formation after 15,000 hours of intensive service. This information could not be inferred from the SEM image alone.

ChemiSEM Technology was subsequently used to characterize the top coat, which is typically manufactured with yttria and magnesia-stabilized zirconia. The after-service material showed a heavily modified structure along with elemental segregation. Examining regions with various distributions of MgO and $ZrO_2$ provides a better understanding of the material's behavior under different conditions, which can be used to improve the durability and performance of the TBC.

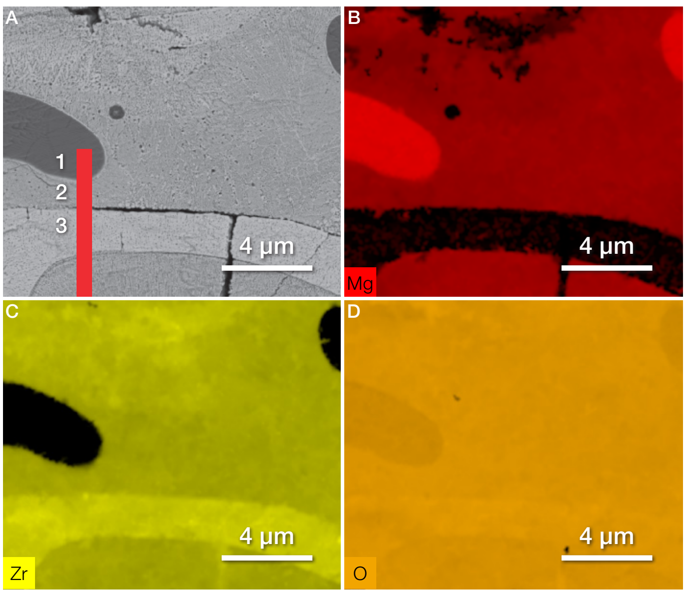



**Figure 4. BSE (A) and ChemiSEM (B-D) images of the TBC top coat, showing the distribution of magnesium (B), zirconium (C), and oxygen (D).**

Figure 4 illustrates the elemental distribution of magnesium, zirconium, and oxygen within the analyzed region, revealing the presence of various materials with differing elemental ratios. While ChemiSEM Technology effectively maps these distributions, it does not provide a comprehensive understanding on its own. To enhance this analysis, a line scan was extracted from the acquired data cube, providing a more detailed elemental distribution and allowing for quantification of the elements within the different materials (Figure 5).

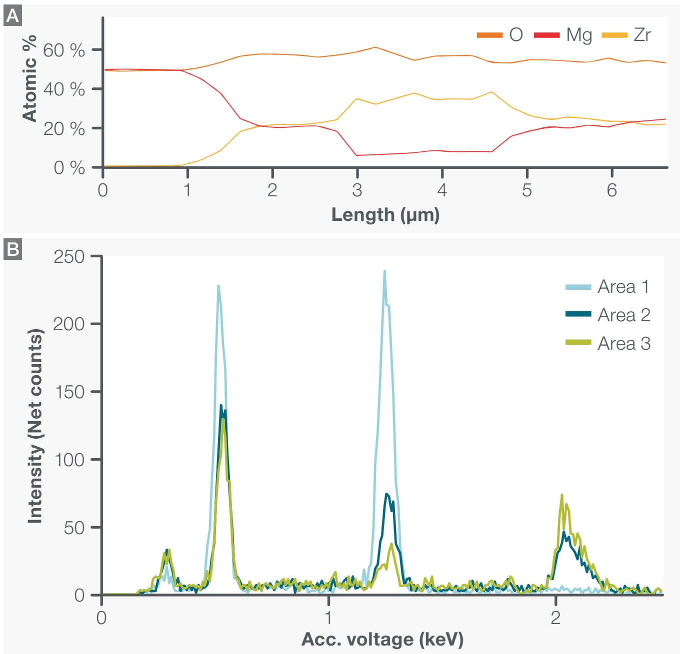



**Figure 5. A) Line scan extracted across three materials with different MgO/$ZrO_2$ ratios. B) Spectral comparison of the different materials.**

The use of line scans enables precise mapping of elemental distributions across various layers of the coating, offering valuable insights into the diffusion processes and phase transformations that occur under high-temperature conditions. As shown in Figure 6, the quantification reveals a uniform distribution of MgO and $ZrO_2$ in area 2 (Figure 5). Just a few micrometers away, areas 1 and 3 exhibit distinct segregation of MgO and $ZrO_2$, with significant variations in elemental distribution.

| | Area 1 | Area 2 | Area 3 |
|---|---|---|---|
| **Element** | **Atomic %** | **Atomic %** | **Atomic %** |
| O | 50.4 | 52-54 | 57-60 |
| Mg | 48.9 | 20-22 | 5-8 |
| Zr | 0.7 | 22-24 | 33-36 |

**Figure 6. Quantification extracted from the line scan for each of the areas of interest identified in Figure 5.**

## Bond coat characterization

EDS characterization of the bond coat provides crucial information on its elemental distribution. While quantitative maps offer some insights, several questions remain unanswered: How many distinct materials are present? What are their compositions? How do elemental interactions influence phase formation? The complexity of the bond coat layer necessitates advanced techniques beyond simple line scan or point analyses.

These challenges were addressed with ChemiPhase, a component of ChemiSEM Technology that enables automated phase analysis. EDS spectra are collected for each pixel of the SEM image, generating a data cube. ChemiPhase then processes this data using multivariate statistical analysis to identify all the phases present in the analyzed area. This provided a more comprehensive understanding of the bond coat's composition and interactions.

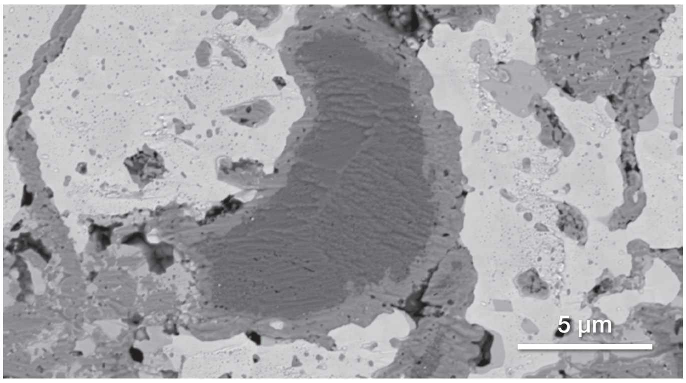



Figure 7. BSE image of the bond coat layer.

Figure 7 shows a region of the bond coat layer; the BSE material contrast features various greyscale levels, suggesting the presence of different materials. ChemiPhase validates these observations (Figure 8), providing a complete characterization of all the materials present, some of which were quite difficult to distinguish using only the material contrast.

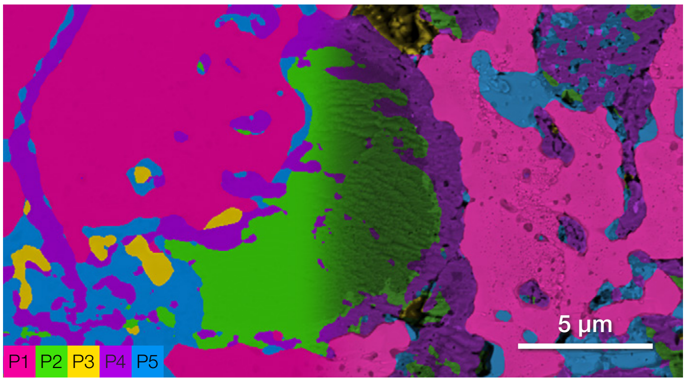
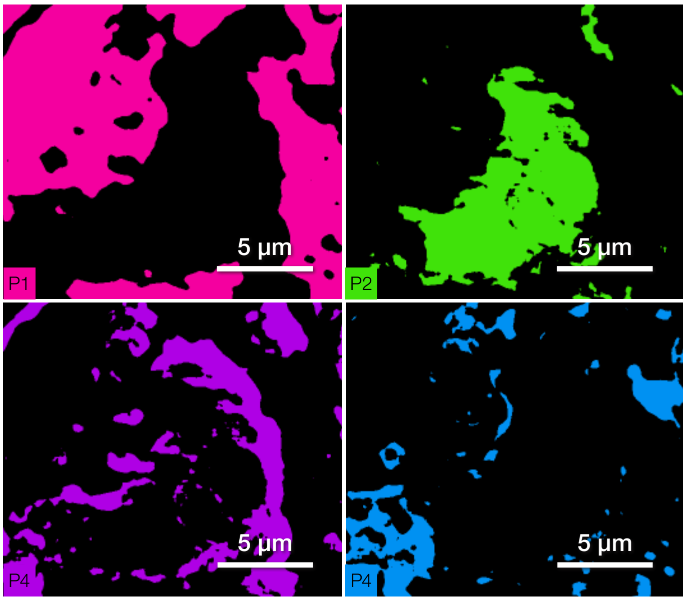



Figure 8. ChemiPhase characterization of the area shown in Figure 7. On the right, the SEM image contrast is overlaid onto the phase distribution map.




| | γ - matrix | Aluminum oxide | β - NiAl | γ' - $Ni_3Al$ |
|---|---|---|---|---|
| **Element** | **Atomic %** | **Atomic %** | **Atomic %** | **Atomic %** |
| Ni | 85.3 | 2.6 | 23.3 | 31.1 |
| O | 10.9 | 57.3 | 52.6 | 52.3 |
| Al | 3.8 | 40.1 | 24.1 | 16.6 |

Figure 9. Phase distribution maps and quantification of all the different materials automatically identified by ChemiPhase analysis.

Figure 9 illustrates the distribution of different phases within the bond coat. The quantification of each phase was automatically calculated, enabling easy recognition of known materials common in TBCs. Among these, the γ-phase was found to be the most abundant throughout the bond coat. The β-NiAl and γ'-$Ni_3Al$ phases are significantly oxidized, which can be attributed to the intense operational conditions and increased number of thermal cycles that the material endured. This oxidation process is further facilitated by interconnected cracks and pores in the top coat (visible in Figure 2), which allow oxygen to penetrate the coating. Aluminum oxide, typically expected to form at the interface between the bond coat and top coat, was instead found abundantly dispersed throughout the bond coat.

ChemiPhase revealed critical insights into the diffusion processes and phase transformations that occur in the TBC during high-temperature exposure. Without ChemiPhase, comprehensive characterization would have required extensive work, including numerous point or region analyses coupled with data averaging, to draw conclusions about the chemical composition of the different materials.

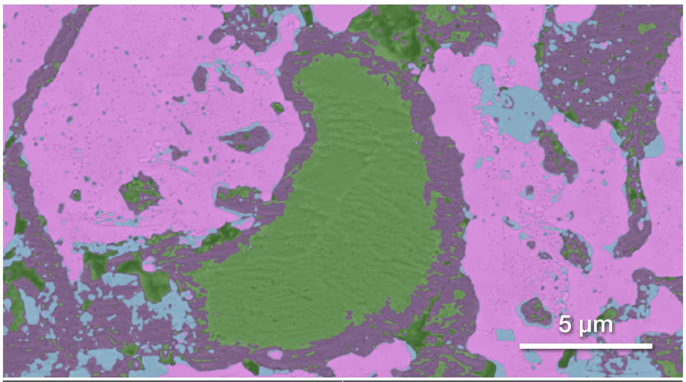
## Machine learning based image characterization

The use of machine learning enables automated image analysis, significantly reducing the need for manual intervention and allowing for the extraction of additional information. In this application note, phase information from the ChemiSEM acquisition was used to train convolutional neural networks (CNNs), with the aim of recognizing feature patterns for segmentation and identifying similar phases in other areas of the sample. The advantage of using these deep CNNs (also known as deep learning networks) is that much of the rich information from the images can be captured without manually adjusting the feature extraction. Multiple models were trained in a hierarchical manner from large overview patches down to specific smaller patches in the bond and top coat. These deep learning models were then applied to other patches, and the results were compared with the ChemiSEM phase data. Typical machine learning standards were followed; a majority of patches with known phases were used for training, a small fraction for validation, and the final set for prediction. In all cases, recognition accuracy for phase segmentation was above 95% for standard segmentation metrics such as the Dice metric.

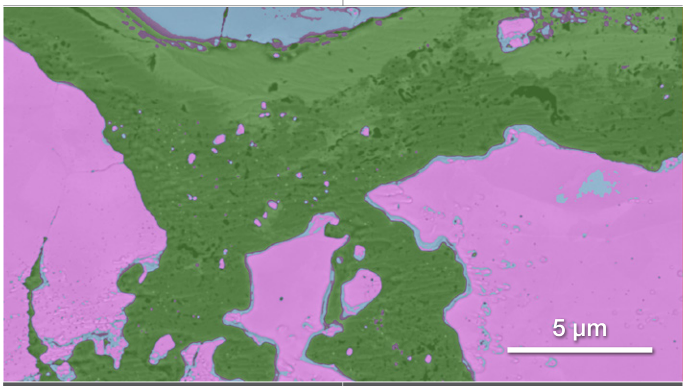
| Name | Color |
|---|---|
| γ - matrix | |
| Aluminum oxide | |
| β - NiAl | |
| γ' - $Ni_3Al$ | |

**Figure 10. Identified material phases used to train the deep learning model in Avizo 3D Pro Software.**

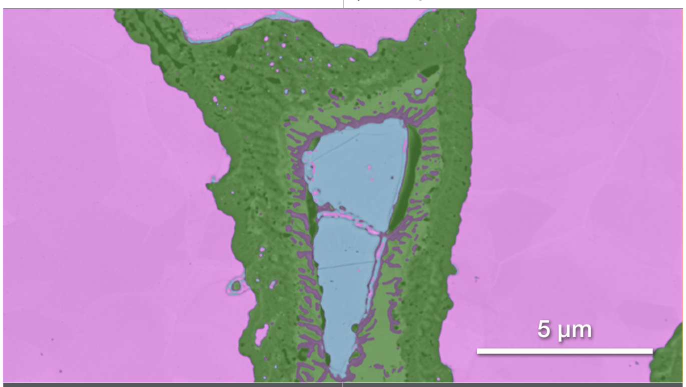
Deep learning model training was performed in Avizo 3D Pro Software using the compositional phases identified by SEM and EDS, as shown in Figure 11. Training the model with Avizo 3D Pro Software took only a few minutes, and the model could be trained on any number of phases. Once trained, it could be applied to other images to automatically extract phase information. To validate the trained model, it was first applied to other locations with known EDS maps to compare predictions with the actual acquired elemental data. Predictions within ±4 percentage points of the actual area fraction (% of the image) were considered sufficiently accurate for subsequent deep learning model training.




| Area fraction | Phases |
|---|---|
| 42.76% | γ - matrix |
| 21.07% | $Al_2O_3$ |
| 25.97% | β - NiAl |
| 10.18% | γ' - $Ni_3Al$ |




| Area fraction | Phases |
|---|---|
| 37.53% | γ - matrix |
| 50.83% | $Al_2O_3$ |
| 3.61% | β - NiAl |
| 8.02% | γ' - $Ni_3Al$ |




| Area fraction | Phases |
|---|---|
| 67.01% | γ - matrix |
| 24.04% | $Al_2O_3$ |
| 3.15% | β - NiAl |
| 5.78% | γ' - $Ni_3Al$ |

**Figure 11. Phase percentages estimated through machine learning. The analyses assumed four phases were present in each of the three chosen locations.**

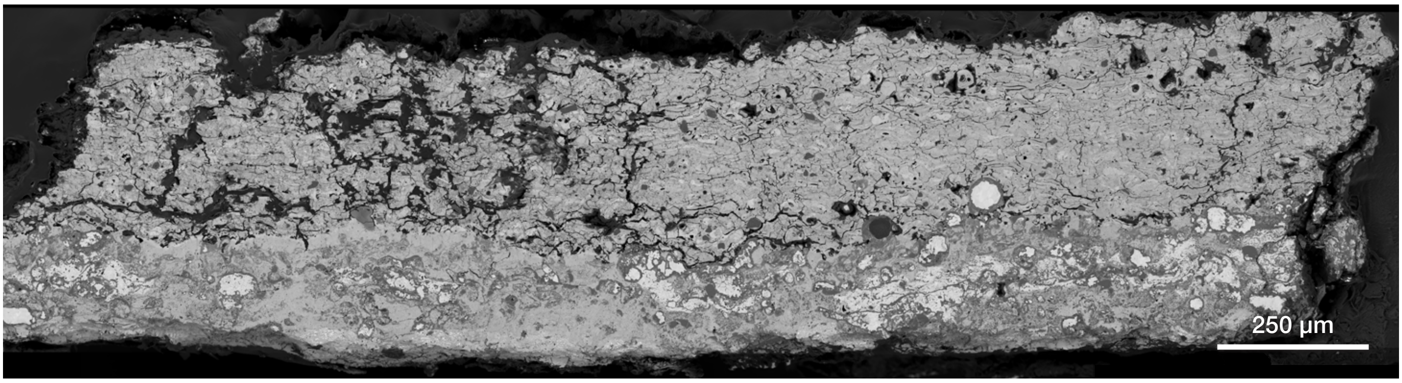



Figure 12. Large-area image acquired with Maps Software.

A traditional machine learning approach (employing a Bayesian classifier) was used to generate predictions and Bayesian probability values for a large cross section of the entire TBC (Figure 12). (This full view was acquired using Thermo Scientific Maps Software, which provided automated acquisition with tiling and frame stitching.) The resulting values were used to train a CNN in Avizo 3D Pro Software; Figure 13 shows the result of the trained model analysis. Remarkably, after training on just a portion of this cross-section, the model could be extended to the entire SEM image.

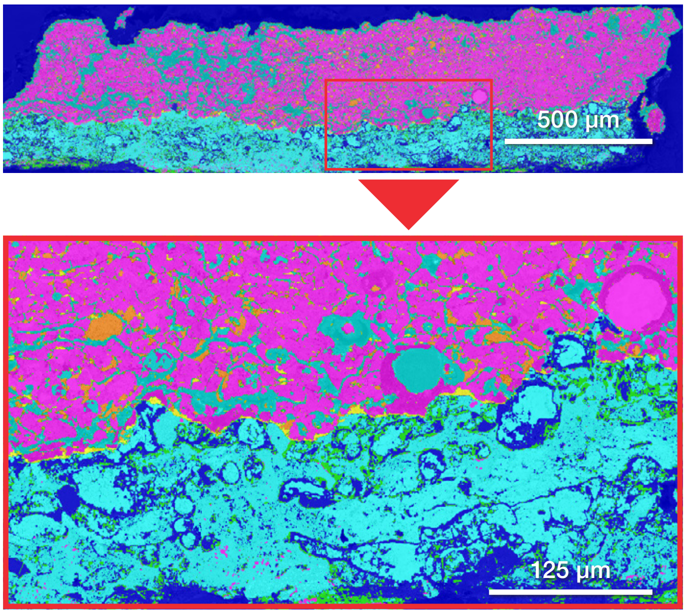



Figure 13. CNN phase analysis of a large TBC cross section.

Overall, the addition of machine and deep learning techniques clearly enhanced the efficiency and accuracy of the TBC analysis. The time-to-results was drastically improved, with significantly reduced processing time. Deep learning models enabled extensive data to be extracted over a large surface area, which would have been impractical with traditional EDS data processing methods.

## Conclusions

The integration of correlative microscopy and AI-assisted image analysis marks a significant breakthrough in the characterization of coatings for high-temperature applications. This approach not only accelerates time-to-results but also enhances the accuracy and depth of analysis. Applying these techniques to TBCs provides a thorough understanding of their properties and behavior, ultimately allowing performance and durability to be improved in critical applications.

Future research should aim to refine these techniques and explore their application to other complex materials. Continued innovation in this field will undoubtedly lead to more efficient and durable high-temperature coatings.

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