# Correlative Microscopy in Composite Material Processing

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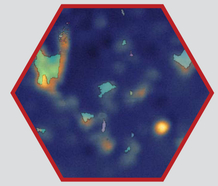

## Introduction: The Complexity of High-Performance Composite Materials

High-performance tool materials, such as WC-Co composites, play a pivotal role in demanding industrial applications, ranging from machining to aerospace. Their exceptional mechanical properties, such as hardness and toughness, result from carefully controlled processing steps. Coatings further enhance the performance of WC-Co composites requiring detailed knowledge and control over the microstructure including the delicate balance between WC grains and the Co binder phase. Conventional characterization methods and workflows often fail to capture the intricate relationships between microstructure and properties, necessitating advanced solutions like correlative microscopy.

## Processing Routes and Material Challenges

The production of WC-Co composites typically begins with mixing WC and Co powders, complemented by binders like paraffin for primary shaping. Liquid phase sintering densifies the material where Co forms a liquid metallic binder phase solidifying into a continuous network in between the WC grains **[1]**. Due to required elevated processing temperatures, side effects such as grain coarsening and elemental intermixing via diffusion are promoted, having wanted and unwanted implications on the final material's performance. Although feasible and widely applied in modern production, several critical challenges persist:

- **Binder Phase Optimization**: While Co provides ductility and toughness, excessive amounts can dilute the mechanical advantages of WC. The distribution and composition of the Co phase are crucial for achieving the desired balance of properties.
- **Phase Distribution and Magnetic Properties**: Localized differences in magnetic properties caused by variations in Co phase composition, such as W and C diffusion, can affect performance unpredictably.
- **Chemical Surface Etching**: Co can be chemically etched from near-surface regions to prepare the material for subsequent surface coatings, improving wear resistance and performance in specific applications. For that, controlled Co phases are essential for consistent results.

In that respect, Magnetic Force Microscopy (MFM) plays a pivotal role in addressing these challenges. By mapping magnetic properties at the nanoscale **[2]**, MFM reveals even subtle variations in the Co binder phase that are otherwise invisible to conventional techniques. Such insights help link processing conditions to magnetic and structural features, providing a deeper understanding of the material's final behavior.

## The Role of Correlated Microscopy

Correlated microscopy provides an excellent pathway to connect processing conditions with material properties. The FusionScope® **[3]** exemplifies this approach by integrating SEM navigation and MFM capabilities down to the nanoscale. The workflow involves:

*FusionScope:*

1. **SEM Navigation**: Precisely locating regions of interest in polished WC-Co composites.
2. **AFM and MFM**: Evaluating nanoscale Co distribution and identifying magnetic variations related to W and C diffusion.

*Subsequent Analyses:*

3. **EBSD Analysis**: Mapping Co phases to distinguish different Co-W compositions and their effects on magnetic signals as acquired by MFM.
4. **TEM Validation**: Using TEM, including advanced techniques like STEM-EELS and EFTEM, to verify elemental intermixing and identify defects down to the atomic scale.

For example, detecting variations in the MFM signal between similar-sized Co regions may indicate differences in W and C interstitials related to sub-ideal processing routes. Subsequent EBSD and TEM analysis can confirm these observations, providing actionable insights for process refinement.

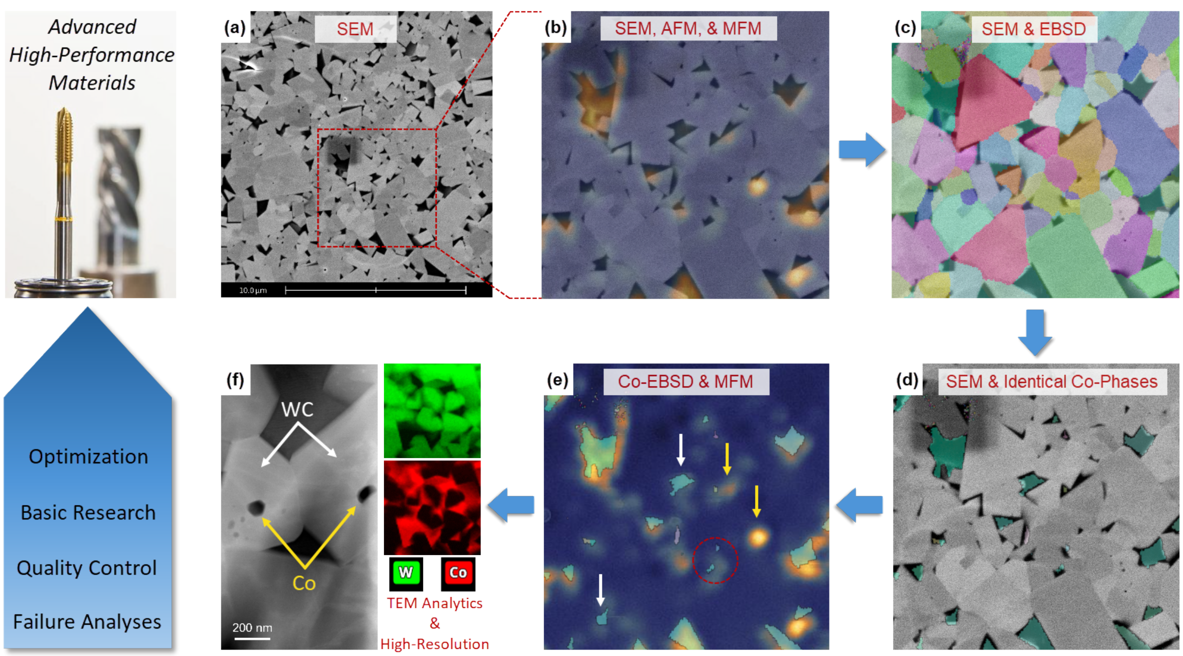


***Figure 1:*** *R&D in composite materials processing utilizing the FusionScope as central element for correlated microscopy.* ***(a)*** *SEM overviews identify the region of interest, followed by AFM and MFM imaging* ***(b)*** *to locate magnetic regions associated with Co phases.* ***(c)*** *EBSD in the same region provides comprehensive crystal orientation data, isolating the desired Co phases* ***(d)****. Direct correlation with MFM data* ***(e)*** *highlights partially magnetic Co phases (yellow arrows) and nonmagnetic phases (white arrows). Regions of interest, such as those marked by red circles, precisely guide TEM-based elemental mapping and related high-resolution imaging* ***(f)****, revealing deviations as encapsulated minimally magnetic Co phases. Essential for fundamental understanding in this case, the FusionScope serves as a central tool for optimization, quality control, and failure analysis.*

## Why FusionScope

The FusionScope integrates the capabilities necessary for complex workflows like this, streamlining the process of navigating, analyzing, and correlating nanoscale features in correlation with complementing techniques. With high-resolution SEM imaging for precise navigation and advanced MFM for detailed magnetic property mapping, the FusionScope ensures that key information is consistently accessible and aligned. Its design minimizes the risk of misalignment or data loss during transitions between techniques, which is critical for accurate analysis. Key advantages include:

- **Seamless Workflow Integration**: The combination of SEM and AFM in one platform simplifies even the most intricate workflows. Additional AFM modes such as MFM, EFM, c-AFM or mechanical mapping further enhance its functionality for diverse applications.
- **Enhanced Precision**: SEM-guided targeting and nanoscale MFM measurements provide unmatched accuracy. Further expansions like EDS simplify chemical identification in the same system for accelerated workflows.
- **Data Coherence**: Structural, magnetic, and compositional data are then collected within the same system, enabling a more cohesive understanding of material behavior.

For multi-phase materials like WC-Co composites, the FusionScope's integrated capabilities are not just beneficial but essential for ensuring reliable and comprehensive characterization.

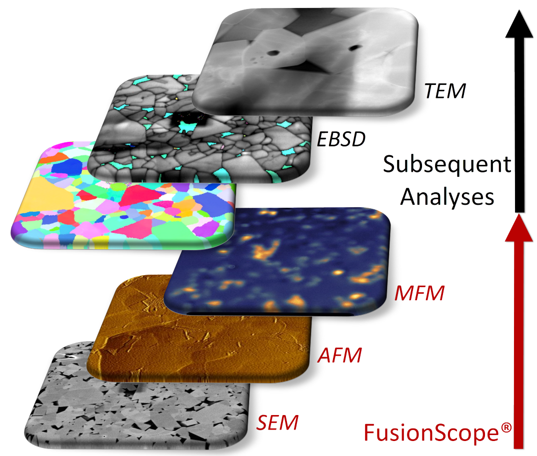


***Figure 2:*** *The FusionScope as a central tool for correlated microscopy. It facilitates region-of-interest identification and advanced surface characterization through its unique, fully integrated multimodal capabilities. Its nanoscale resolution in the initial steps ensures precise alignment for subsequent complementary material analyses.*

## Broader Integration Across Applications

While this use case highlights a specific challenge in WC-Co composites, the FusionScope's true strength lies in its remarkable flexibility, making it a highly invaluable tool for modern R&D environments. Its versatility enables seamless adaptation across a wide spectrum of applications:

- **Process Optimization**: Linking processing parameters to final properties in advanced material systems, offering actionable insights for refinement.
- **Quality Control**: Rapidly assessing and ensuring consistency in production, making it indispensable for high-throughput environments.
- **Failure Analysis**: Identifying root causes of material degradation or performance issues with unparalleled precision.

- **Basic Research**: Delving into phase interactions, defect dynamics, and other fundamental phenomena with unmatched depth.

The ability to seamlessly integrate multiple techniques in a single workflow ensures that the FusionScope is not just a tool, but a pivotal asset in shaping the future of material science and engineering.

## Conclusion

The FusionScope empowers researchers and engineers to address the complexities of modern materials with unparalleled precision and efficiency. By uniting SEM, AFM, and MFM within a single, integrated workflow, it enables comprehensive insights into microstructural and magnetic properties. This capability not only bridges the gap between understanding and application but also supports both fundamental research and practical innovation. By enabling a seamless connection between understanding and application, the FusionScope redefines possibilities for modern material science, driving both innovation and practical breakthroughs.

## About the Author

Harald Plank is a professor at Graz University of Technology and Director of the "Christian Doppler Laboratory for Direct-Write Fabrication of 3D Nanoprobes". His focus lies in research & development around "Functional Nanofabrication" with the aspiration of transferring basic research into industrially relevant applications. In addition, Professor Plank coordinates activities related to advanced microscopy for industrial services to support clients with complex material problems, time-sensitive challenges, or forensic analysis of patent infringements.

## References

**[1]** I.O. Kamolova, R.Kh. Saydakhmedov, *Metal Science and Heat Treatment* **2024**, 65, 582.

**[2]** R. Winkler, et al.; *Nanomaterials* **2023**, 13 (7), 1217.

**[3]** www.fusionscope.com