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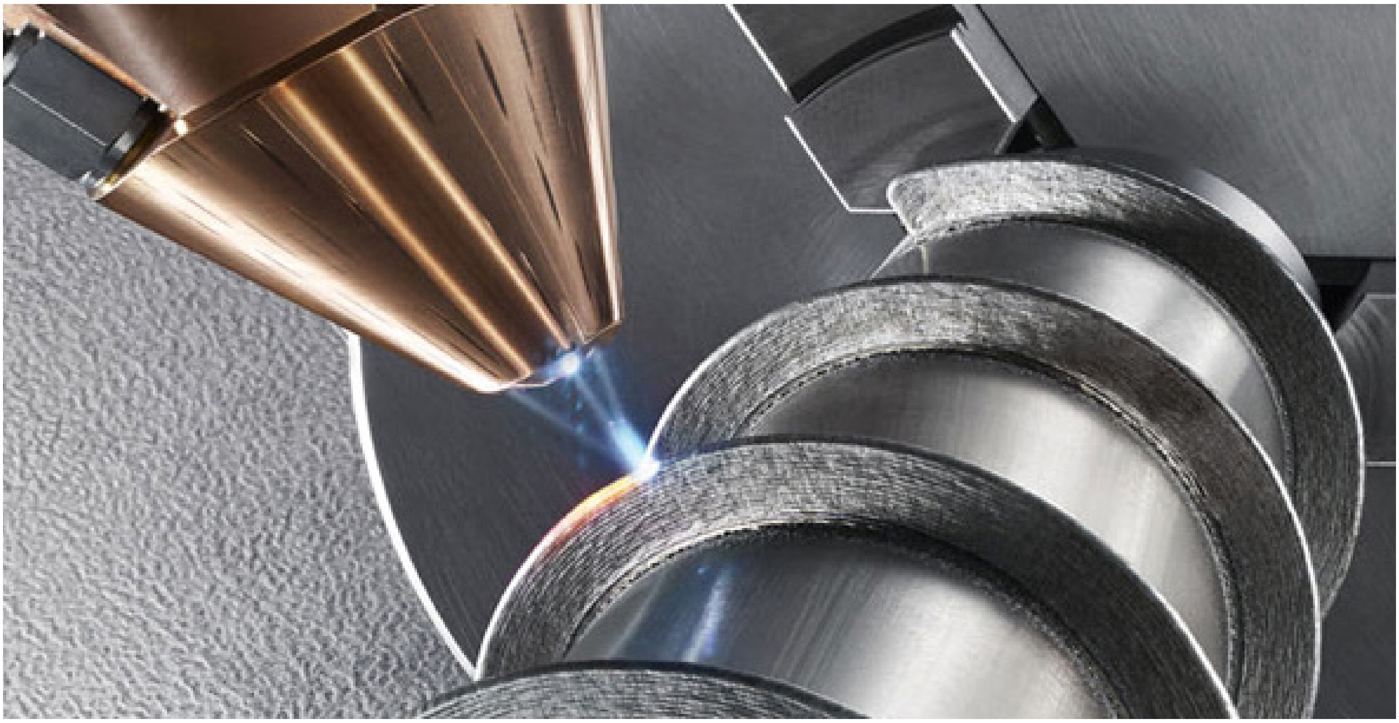
APPLICATION NOTE



Correlative microscopy characterization of material failure within an additively manufactured Inconel 718 turbine component

Maps Software provides the tools to efficiently conduct multi-scale, multi-modal material characterization

The relevant scales for characterizing the physical attributes that ultimately control material properties span many orders of magnitude. Researchers are constantly challenged by the acquisition and tracking of data necessary for the accurate assessment of microstructural control impact on performance. This is especially true in additive manufacturing methods, where new frontiers in build methodology are being explored.

ThermoFisher SCIENTIFIC

Enabling technology

Thermo Scientific™ Maps™ Software is the microscope automation and correlative microscopy software package for the Thermo Scientific line of electron microscopy equipment (SEM, DualBeam and TEM). Maps Software is a project-based software platform that automates imaging acquisition and provides an intuitive platform to bring all imagery collected on a sample together, in one place, for correlative investigations of multi-scale microstructural data.

Introduction

Additive manufacturing (AM) methods are revolutionizing how materials are designed and built. The strength of components with complex shapes is often drastically increased by additive manufacturing, as the typical areas of weakness, such as welds or mechanically joined surfaces, are removed. However, using additive manufacturing to control microstructure development in metals is still an area of active research. This is particularly true for methods like blown powder direct laser deposition (BPLD), where build condition control is critical for generating a material with the correct performance criteria (see image on the right).

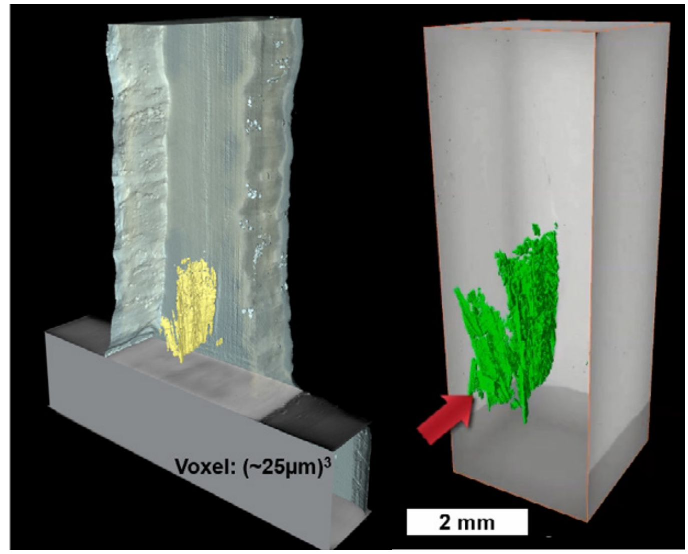


Figure 1. Avizo Software visualization of the test turbine showing locations of fractures (green) inside the component.

Understanding BPLD process parameters and material properties and, more specifically, controls on material failure/performance fundamentally require a multi-scale approach. The fractures that define material failure are typically many orders of magnitude larger in scale than the microstructural features and associated defects that control their initiation and growth. Maps Software provides an efficient and powerful way to interrogate image-based data across all relevant length scales in a single environment.

Here, we illustrate how a correlative workflow enabled by Maps Software makes characterization of multi-scale, multi-modal datasets easy and efficient. In this example, we explore a region of mechanical failure within an Inconel 718 test turbine component created using a high-power setting of blown powder direct laser additive manufacturing. The test coupon was selected from a series of AM build tests designed to optimize the BPLD process parameters, thus obtaining ultimate microstructural and mechanical properties.

Correlative workflow

A coupon of material was sampled from the bulk and scanned in a Thermo Scientific™ HeliScan™ microCT to interrogate the internal structure of the sample and plan for locations for further analysis. HeliScan microCT data was opened in Thermo Scientific™ Avizo™ Software to locate and quantify the extent, size and shape aspects of any voids or fractures identified in the sample (**Figure 1**). Once a region of interest was identified, the sample was cut and the surface prepared for high-resolution analysis in a Thermo Scientific™ Helios™ PFIB (Plasma FIB) DualBeam. Prior to electron microscopy work, the sample was imaged using an automated optical microscope to provide an overview of the sample.

Upon loading of the sample into the imaging platform, an optical navigation image was taken of the sample using the Thermo Scientific™ Nav-Cam™ Camera. Once loaded into Maps Software, this Nav-Cam Camera image was a useful base for exploring a sample's surface, as well as acting as an anchor for import and alignment of future imaging work.

To begin the workflow, the area to be imaged was selected, along with the field size and resolution. Maps Software initiated the acquisition and ran, unattended, until imaging completed. In this case, we wanted to focus the imaging in the region around the obvious fractures observed in the microCT and seen on the surface of the optical image (**Figure 2**).

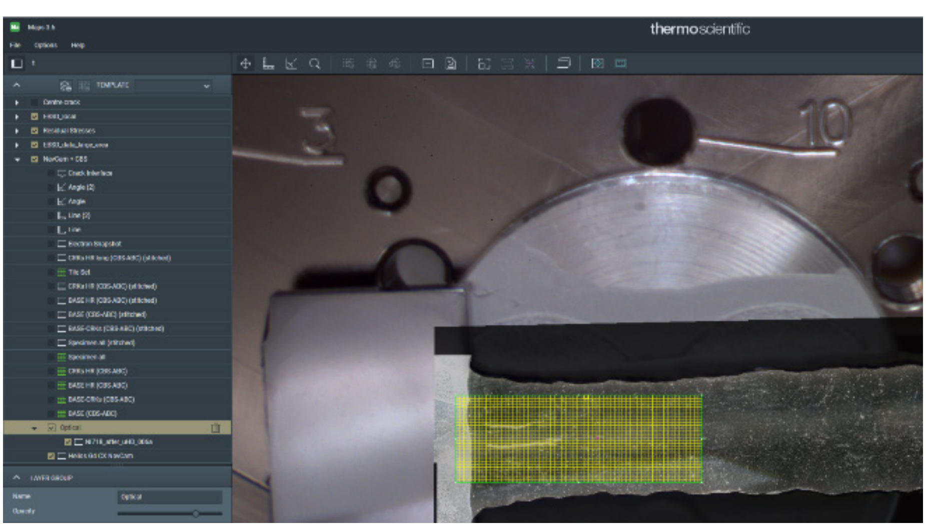


Figure 2. Maps Software UI illustrating the Nav-Cam Camera image background with over-layed optical image and tileset (yellow grid) that is the target for high-resolution BSE image acquisition.

Using Maps Software to explore the various high-resolution BSE images, it quickly became evident that the material lacked a preferred weld microstructure (**Figure 3**). Laves phases were dispersed within a columnar-shaped γ phase. Residual melt phases in the form of dendrites were clearly visible in high-resolution SEM imagery and were concentrated around the macro fractures in the sample, indicating the impact of these phases on localized embrittlement of the sample. The large mosaic images easily captured via automation in Maps Software made these impactful contextual observations straightforward to obtain and easy to share with collaborators.

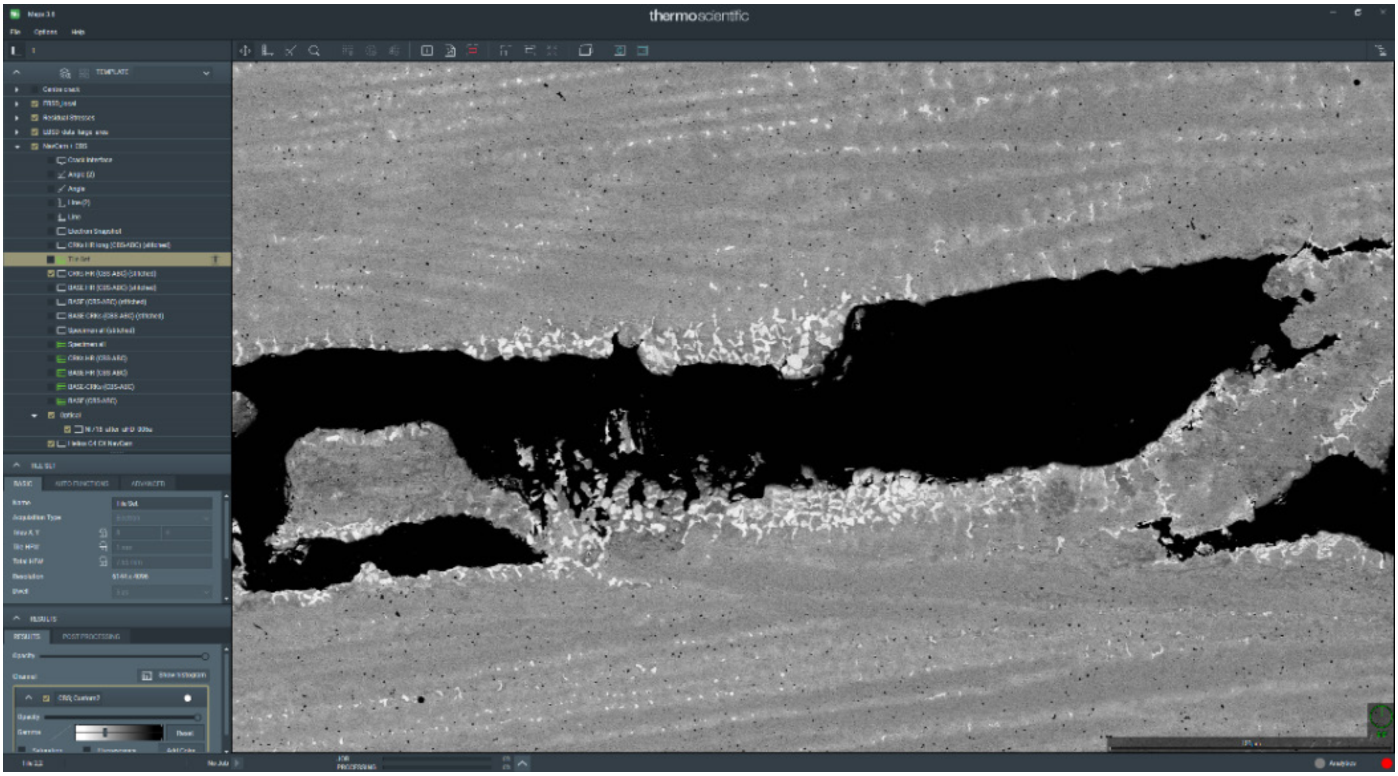


Figure 3. High-resolution BSE image of fracture along grain boundaries within the Inconel coupon. High concentration of dendritic melt phases indicates improper build conditions leading to an unstable microstructure during cooling of the build. BSE imagery was used to select locations for EBSD analysis.

To further our understanding of the microstructure, Maps Software was used to plan for areas of interest for EBSD data collection. The goal of obtaining EBSD was to understand the crystallographic relationships in the sample, their relation to the fracture and crack tips, and ultimately, how the final microstructure impacts stress relaxation upon cooling.

After acquisition, Maps Software made it trouble-free to import EBSD maps and supporting data, such as inverse pole figures, conveniently present as a separate layer. Image layers from any source were easy to load, visualize and enable while reviewing data. In the sample, EBSD revealed large columnar grains with little preferred orientation of major axes. EBSD also illustrated regions where the build speed changed during the additive manufacturing, as shown in **Figure 4**.

For example, we can see in **Figure 5** how EBSD data is related to the true shape and extent of a fracture in the Inconel coupon.

After initial imaging was completed, a series of micro-hole patterns were PFIB milled into the surface to quantify the residual stresses in the critical locations in the sample. Again, using Maps Software, the imagery data used to evaluate the experiment was tied directly to the other images and modalities already present in the project. Co-visualization and interpretation were made easier by implementing a single point of data collection and visualization. Efficiency in finding areas already imaged as well as in interpreting and sharing of observation were increased heavily by utilizing Maps Software as a base of sample characterization.

Using a high-resolution BSE image as a backdrop permited quick inspection of what microstructural elements were worth spending time on, in addition to making it easy to preserve the context of where each dataset was relative to one another. In addition, the microstructural details in a high-resolution image could have been used to enhance the interpretation of modalities that may not have had the same spatial resolution. In Maps Software, we can quickly view the backdrop of data in the form of BSE to observe the spatial relevance of data that may have been missed or otherwise obscured based on the methodology used.

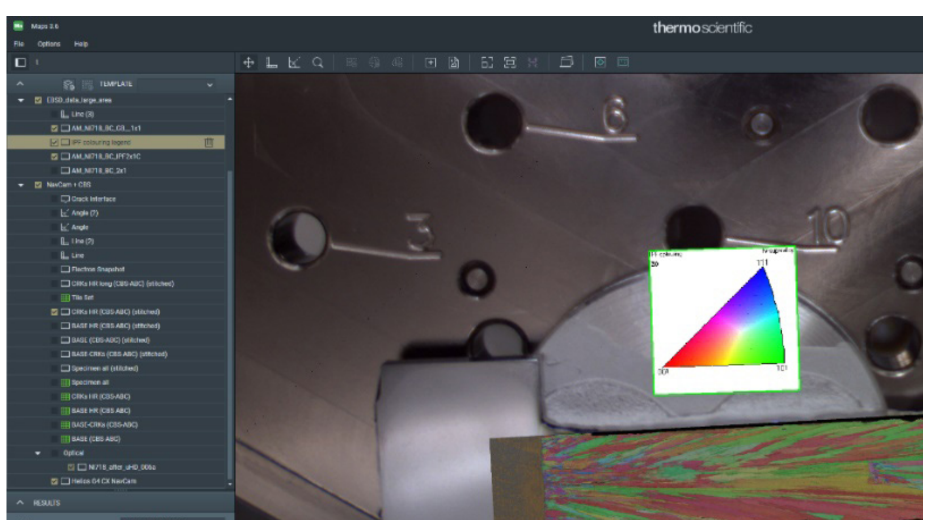


Figure 4. Tiled EBSD map of full sample overlaid on Nav-Cam Camera background image. Inverse Pole Figure image is also placed directly in Maps Software. Each layer can be turned on or off as the use requires for visualization. EBSD map shows crystallographic orientation of large columnar grains.

Comprehensive multi-scale and multi-modal analyses performed with HeliScan micro CT and PFIB DualBeam, handled by Maps and Avizo Software, allowed excellent understanding of blown powder direct laser deposited process parameters, microstructural and compositional characteristics and residual stress states of the BPLD deposited test turbine component. It was determined that Inconel 718 additively manufactured components, with optimized process parameters, possess fine-grained, flawless microstructure and geometrical parameters, and they can withstand thermomechanical loads typical for in-service conditions.

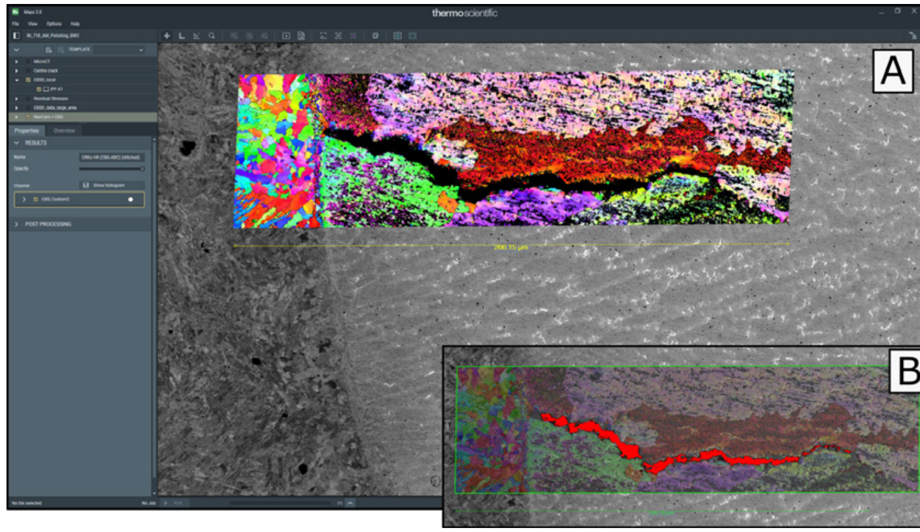
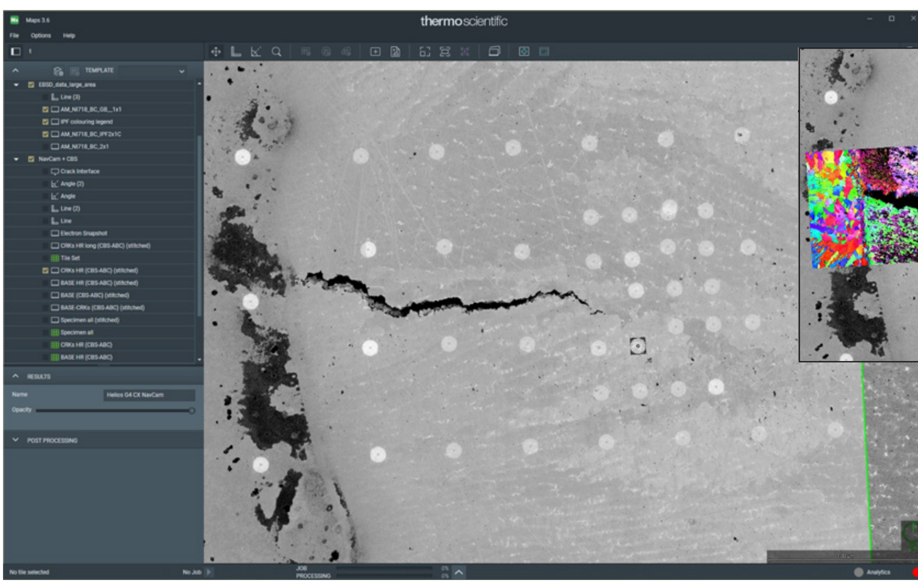


Figure 5. A) BSE image background with an EBSD map around a fractured region of the Inconel coupon. B) Same image as A, illustrating transparency of EBSD map on the BSE background image. Maps Software also provides the ability to threshold an image to highlight specific features. In this case, illustrating the difference in fracture aperture captured in EBSD versus BSE imagery.



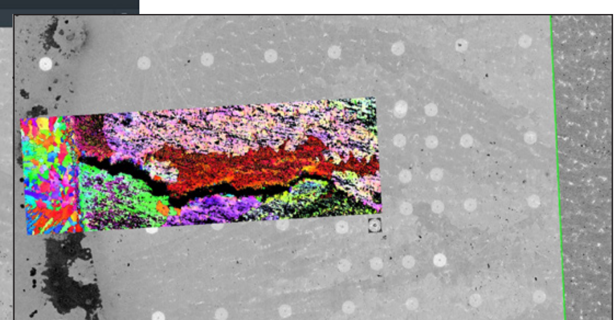


Figure 6. Maps Software UI with an image of the same fracture as shown in Figure 5, but collected after changes to sample prep and milling of features to measure residual stresses. Maps Software makes realignment and correlation of data straightforward between imaging sessions. INSET: EBSD data from Figure 5 overlain on locations of residual stress measurement.

Acknowledgments
We would like to acknowledge Philip J. Withers, Bartlomiej Winiarski and Grace M. Burke from The University of Manchester, U.K for supplying the research material.

Further reading

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