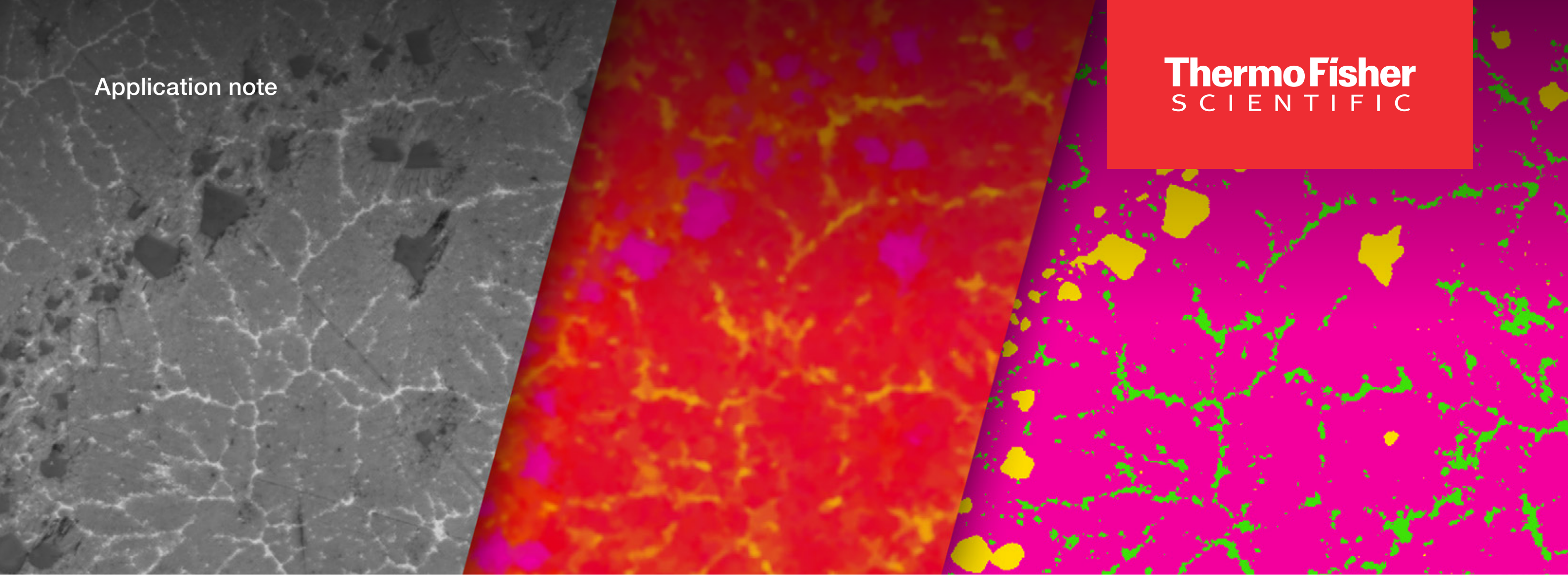
Application note



# Distinguishing the unique components of composite materials with ChemiPhase Analysis Software

## Introduction

Scanning electron microscopy (SEM) has long been used to identify materials with energy-dispersive spectroscopy (EDS), which allows you to choose a region of interest and instantly determine its material composition. Additionally, backscattered electron imaging (BSE) visually aids the search for unique materials as the signal varies with atomic weight. A typical workflow of BSE imaging combined with point mode EDS analysis doesn't always provide a compete characterization of the material of interest. BSE contrast can be poor if materials have similar atomic weight or the chemistry of the material is too complex, leading to potentially erroneous conclusions or an incomplete characterization. This would force you to run multiple analyses in order to obtain a more complete answer, or interpret the results based on previous assumptions.

Thermo Scientific ChemiPhase Software is a novel phase identification and quantification engine within Thermo Scientific™ ChemiSEM™ Technology, designed to address these challenges, identifying unique phases through a combination of advanced statistical analysis and the ChemiSEM SEM-EDS platform. ChemiPhase Software helps you answer critical questions, including: what the composition is, how it is distributed, and how much of each phase is present.

## What is ChemiPhase Software?

ChemiSEM Technology uses a big data approach to detect all statistically significant spectra within the acquired datacube. ChemiPhase Software then provides a simple probability for each pixel, indicating if it belongs to each of these spectra. This makes interpretation of complex samples much more straightforward and intuitive, as each pixel can only belong to a single phase.

Notably, ChemiPhase Software is a comprehensive, unbiased statistical engine. This avoids problems associated with traditional methods, which can yield erroneous results if unexpected elements are missed (i.e., due to overlapping peaks or insufficient intensities). Additionally, traditional phase determination is highly dependent on assumptions about the sample, whereas ChemiPhase analysis ensures that all instrument users will obtain the same result, even for challenging samples.

## Benefits of ChemiPhase Software

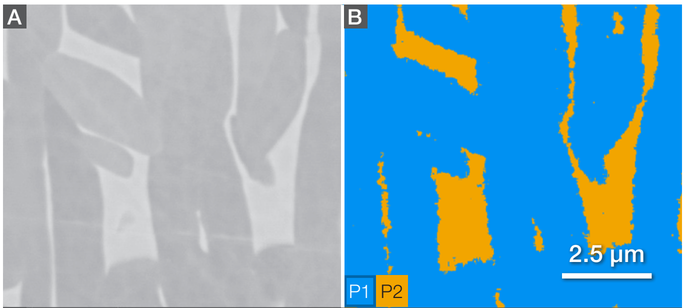
- Eliminates user bias
- Runs fully automated, with no prior identification of elements
- Locates minor and trace elements without extensive user experience
- Provides complete and comprehensive analysis
- Unambiguously identifies major and minor components down to a single pixel
- Locates unique components where peak overlaps obscure significant elements
- Offers fast data acquisition
- Can begin phase determination with very little X-ray data; as few as 10 counts per pixel
- Completes most acquisitions in less than a minute, even for complex phase maps

The following examples showcase ChemiPhase Software at work.

## 1. Additively manufactured Ti-6Al-4V alloy

Additive manufacturing with metals is akin to building a structure by welding, one powder bead at a time. The laser rapidly, but very briefly, melts the metal, allowing it to form into a completely new structure which can have complex variations in composition.

Being able to study and map this composition is key for quality assessment of the final material. Even if some of this information can be determined with SEM, a certain level of expertise is still required to interpret the results, and multiple acquisitions can be necessary to produce statistically significant observations. As shown in Figure 1, ChemiPhase Software easily maps the composition of a Ti-6Al-4V alloy, revealing the unique phases present and their composition/area fraction. Sample courtesy of GKN Aerospace Global Technology Centre, Bristol.

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| Element | at.% P1 | at.% P2 |
|---|---|---|
| Ti | 88.2 | 79.1 |
| Al | 8.4 | 4.5 |
| V | 3.4 | 14.9 |
| Fe | 0 | 1.5 |

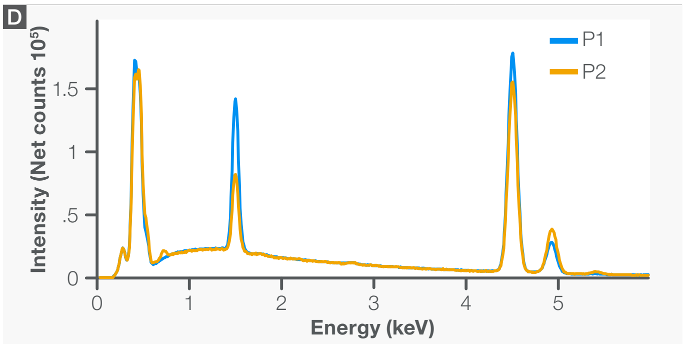
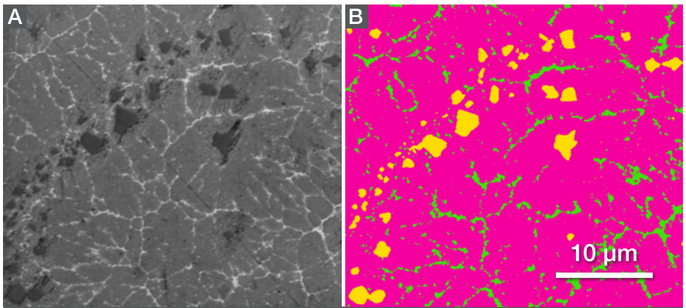



**Figure 1. a) BSE image of a Ti-6Al-4V titanium alloy. b) Phase maps with area fractions P1 (alpha-Ti) = 85.6% and P2 (beta-Ti) = 14.4%. c) Elemental composition of the titanium alloy phases. d) Overlaid spectra reveal more titanium and aluminum are present in the blue areas, whereas more iron and vanadium are found in the yellow areas.**

## 2. Additively manufactured AlSiCu alloy

More complex than the previous Ti-6Al-4V alloy example, additively manufactured AlSiCu contains three unique phases as well as challenging sub-micron features. Analyzing this structure with EDS would necessitate several different characterization, mapping, and point analyses and might still only produce incomplete answers.

ChemiPhase Software, meanwhile, can provide all the pieces of the puzzle in one click: it maps all the different phases, calculates their composition, and provides their area fractions in the analyzed field of view. Additionally, each phase has a corresponding component map, which reveals the intensity of the unique spectrum for each pixel in the image. For example, Figure 2e shows the silicon-rich component while Figure 2f shows the corresponding phase map.

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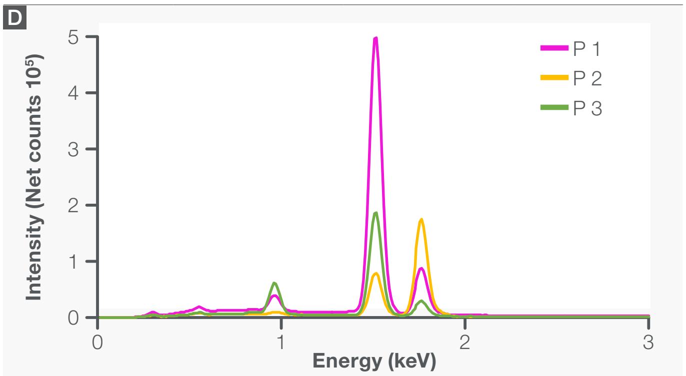
| Element | at.% P1 | at.% P2 | at.% P3 |
|---|---|---|---|
| Al | 78 | 23.9 | 72.4 |
| Si | 19.3 | 75.4 | 14.5 |
| Cu | 2.7 | 0.7 | 13.1 |



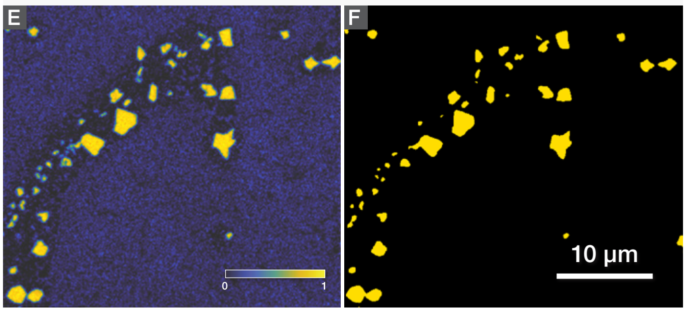




**Figure 2. a) Secondary electron image of a AlSiCu aluminum alloy. b) Phase maps with area fractions P1 = 84.5%, P2 = 9.2%, and P3 = 6.3%, where P1 is base metal, P2 are silicon "seed" particles used for heterogeneous nucleation, and P3 is the $Al_2Cu$ σ-phase used for precipitation hardening. c) Elemental composition of the aluminum alloy phases. d) Overlaid spectra showing base aluminum in pink, silicon in yellow, and copper in green. e) Component map of the silicon-rich phase, as indicated by the color gradient bar. f) Phase map of the silicon-rich phase.**

## Refractory oxide grains

Two different types of synthetic refractory oxide grains were studied to identify impurities; fused zirconia mullite and brown fused alumina. Fused zirconia mullite should contain exclusively zirconia and mullite. ChemiPhase Software revealed the shape and amount of mullite (P1) and zirconia (P2) present, plus an undesirable contaminant (P3) with a low melting point that is rich in soda and silica (Figure 3). ChemiPhase analysis of the brown fused alumina revealed the expected alumina (P1), plus unwanted titania (P3), iron oxide (P4), calcium-aluminum-silicate (P5), and other contaminants (Figure 4).

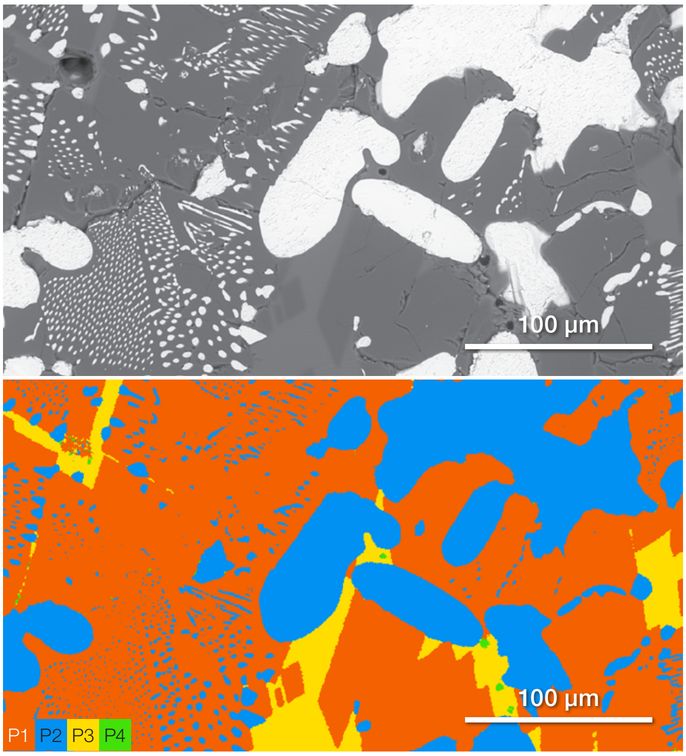



**Figure 3. a) BSE image of fused zirconia mullite grain. b) Phase maps with area fractions P1 (mullite) = 30.5%, P2 (zirconia) = 62.2%, P3 = 7.1%, and P4 = 0.2%, where P3/P4 are unwanted contaminants.**

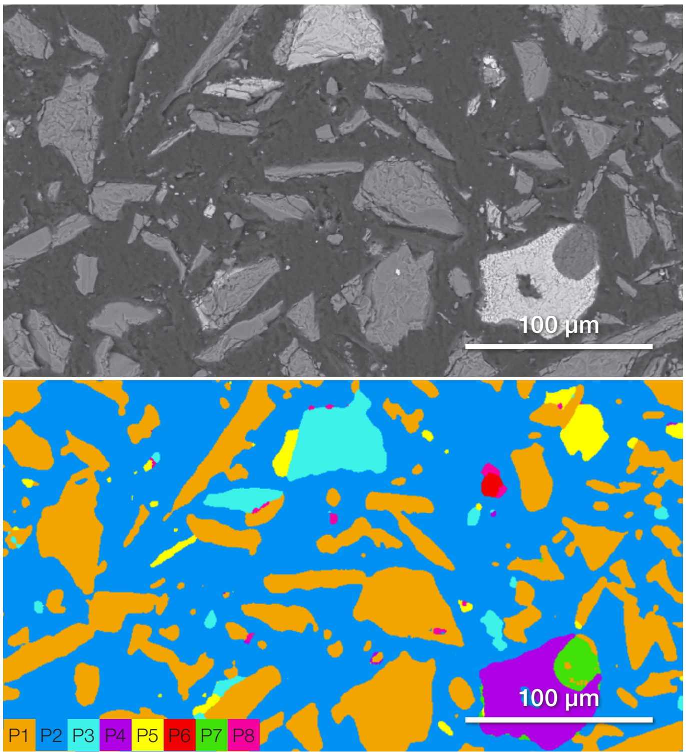



**Figure 4. a) BSE image of epoxy-mounted brown fused alumina. b) Phase maps with area fractions P1 (alumina) = 82.0%, P3 (titania) = 7.3%, P4 (iron oxide) = 6.8%, and P5 (calcium-aluminum-silicate) = 3.8%. The epoxy (P2) was excluded.**

## Conclusions

ChemiPhase Software offers a novel approach for compositional characterization, eliminating user bias and quickly providing answers to your material questions. With ChemiSEM Technology and ChemiPhase Software, complex materials are clearly separated into components/phases, with compositions identified and area fraction tabulated.

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