

COMPASS (Multivariate Analysis) and Xphase (Phase Analysis) Analysis of a Complicated Alloy

Minoru Suzuki, Thermo Fisher Scientific, Yokohama, Japan
Pat Camus, Ph.D., Thermo Fisher Scientific, Madison, WI, USA

Particles in Turbine Material

In most engineering alloys, the formation of particles modifies the physical properties of the final product. Sometimes the particles are planned by the alloy design engineer to enhance properties and sometimes they form inadvertently during fabrication or use and degrade properties. In either case, analysis of the particles is crucial in optimizing the properties during use. This analysis can include chemical identification using energy dispersive spectroscopy (EDS).

A region containing particles in a turbine material was observed, Figure 1, and a spectral imaging data set was collected using the following conditions:

Beam Voltage: 5 kV

Magnification: 8 kx

Collection time: 40 minutes

The low beam energy was chosen to reduce the interaction volume and improve the spatial resolution of the EDS analysis.

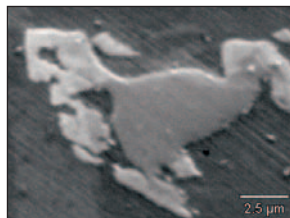


Figure 1

The cumulative spectrum for all of the pixels in the data set is shown in Figure 2. Note the heavy overlap of many of the peaks. These overlaps are expected to complicate the interpretation of the analysis.

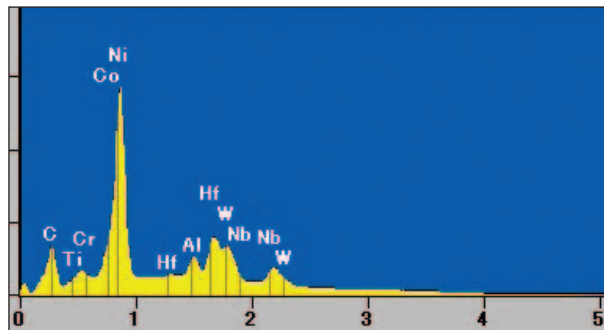


Figure 2

Traditional EDS Analyses

The cumulative spectrum shows that the Cr-L peak overlaps with the Ti-L peak, and the Hf-M peak overlaps with the W-M peak. These peak overlaps lead to elemental peak count maps which have a similar appearance, Figure 3. Interpretation of the maps under these circumstances will lead to an incorrect report.

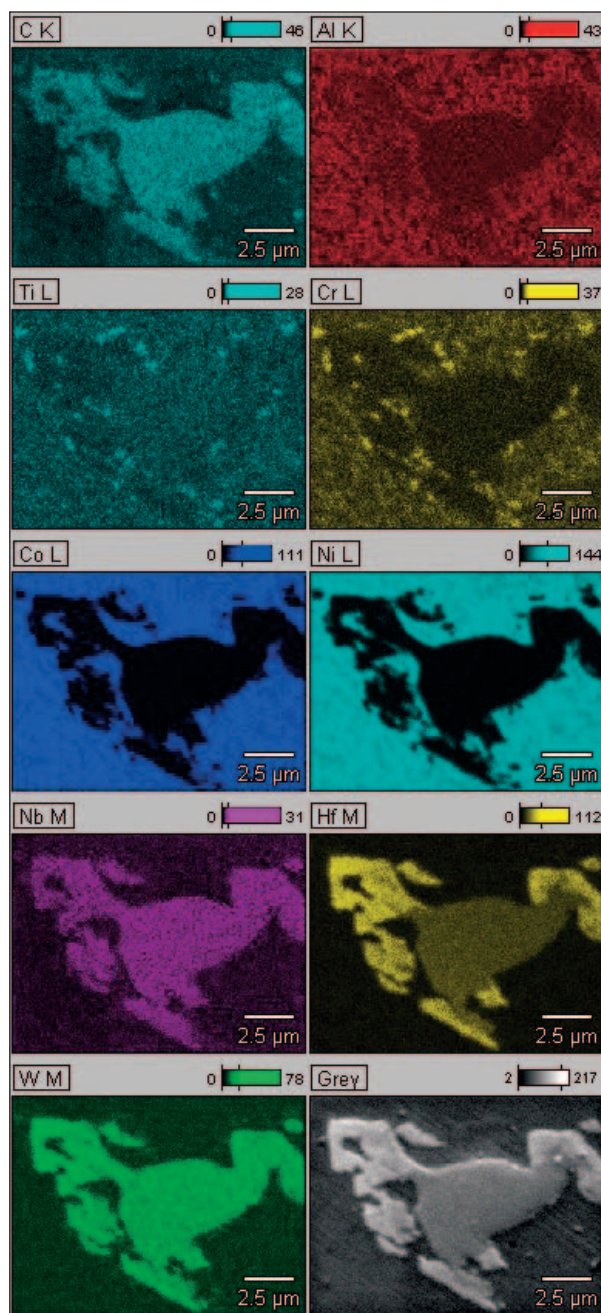


Figure 3

Key Words

- COMPASS
- EDS
- Multivariate Analysis
- NSS
- Peak Maps
- Xphase

However, a large difference is found between these sets of elemental maps when full quantitative corrections are applied, Figure 4. The process of creating quantitative net count maps removes the spectral background and performs peak separation. Because of these peak corrections, the maps are a more accurate representation of the elemental distributions in the sample than the peak count map and will produce a correct report. When the results of the quantitative maps are observed, the analyst infers the existence of multiple phases within the analysis area because of a variety of elemental intensity overlaps at many pixels. However, it is difficult to describe the number and elemental combinations of phases because there are so many elements and phases present.

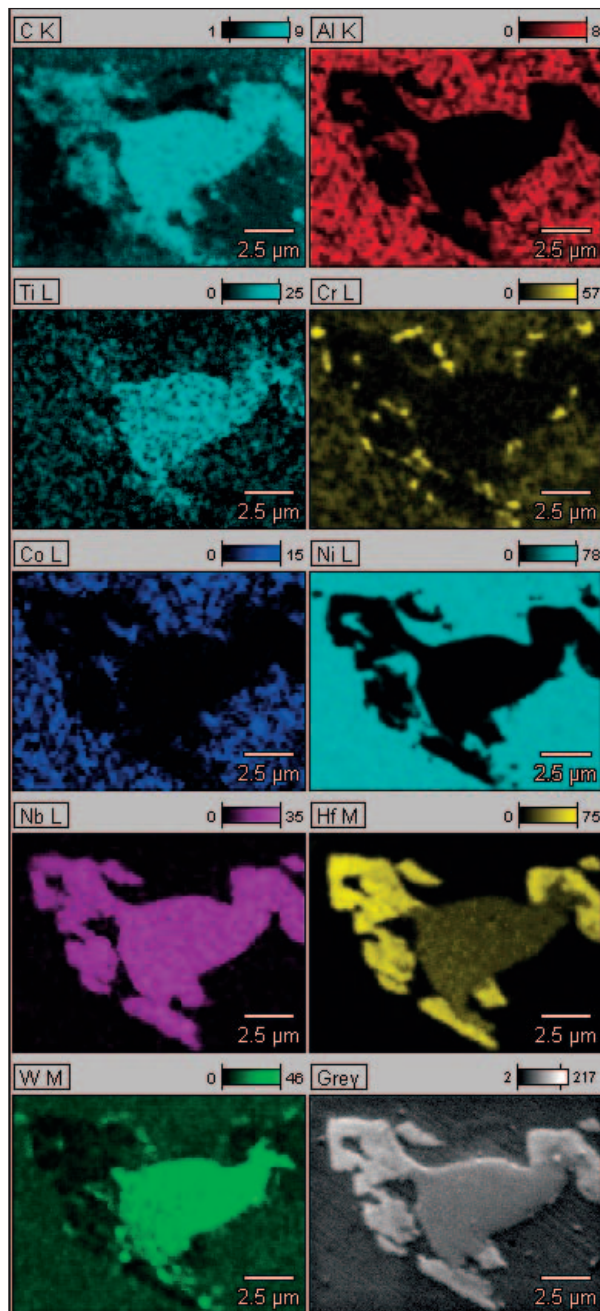


Figure 4

COMPASS (Multivariate Statistical Analysis)

COMPASS is a software option for NSS that uses multivariate statistical analysis to find spectral-similar regions in a spectral imaging acquisition. It analyzes the spectrum at each pixel location and groups pixels with similar spectra into principal components. The result is a list of chemically-unique component spectra and component maps.

COMPASS Result

Five primary component spectra and maps were produced automatically for this data set, Figure 5. Because it was observed that the spectrum of the first principal component and the spectrum of the fifth principal component were similar, these components are displayed together for comparison purposes. If only the results of the quantitative map are considered, the phases which form the sample are difficult to identify. However, COMPASS has extracted them automatically, with no user bias on the input data. In addition, notice how COMPASS has isolated components 2, 3 and 4 even when these components have a huge overlap of the elemental peaks for Hf and W. This shows that COMPASS is not only spatially deconvoluting but also spectrally deconvoluting.

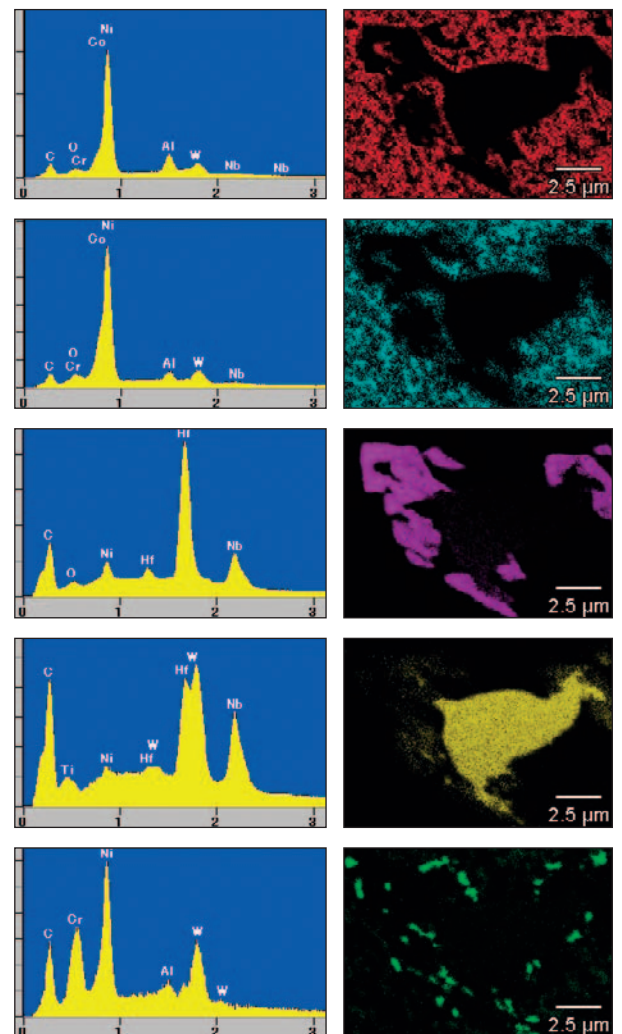


Figure 5

Because the component spectra are a result of the mathematics of the routine, they do not accurately represent the composition of the components. In order to accurately determine the composition of each phase that COMPASS has extracted, there must be an independent method to generate an extracted spectrum for each component.

XPhase (Compound Location Analysis)

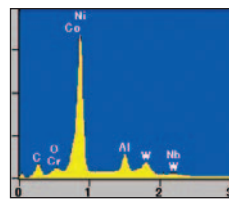
The software option, XPhase, is created around the strength of the COMPASS map and the quantitative map, and produces binary extraction images. These map images are used to extract compound spectra directly from the spectral imaging data set. By processing the analytical results of the COMPASS and XPhase together, location and quantitative analysis of each phase is performed.

XPhase Results

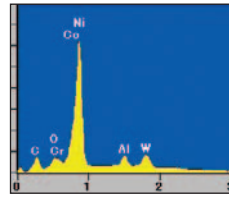
Using XPhase data, each phase can be located and analyzed quantitatively. The extracted spectrum and the quantitative results, which are extracted from each indicated phase, are shown in Figure 6. When Phase 1 (derived from the first principal component) and Phase 5 (derived from the fifth principal component) compositions are compared, the Al content of Phase 1 and the Cr and the Co content of Phase 5 are found to be higher than the other phase. The automatic COMPASS analysis has identified the difference composition in the two phases even though there is little if any contrast in the elemental maps. The quantitative analyses confirm the initial COMPASS results of the separation of phases 2-4 with Hf:W ratios that vary from 54:10, to 23:30, to 3:20.

Summary

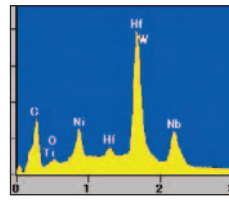
This turbine sample was formed with many elements, including Al, Ti, Cr, Co, Ni, Nb, Hf and W. In addition, the respective elements segregated in a complicated way and multiple particle phases were observed. To obtain accurate EDS analyses at the necessary high magnification (8,000 x), the acceleration voltage must be reduced to a low value (5 kV) to reduce the interaction volume and increase the spatial resolution. This causes many of the elemental peaks to be overlapped. Analysis for each of the respective phases was nearly impossible using traditional peak count map. It is possible, however, to extract five unique phases simply by using the automated COMPASS analysis. In addition, XPhase provides a quantitative analysis for each of the phases. The combination of COMPASS and XPhase is very effective in phase analysis and identification of particles at high magnification and low beam energy with EDS.



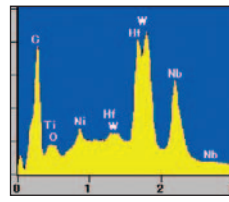
Element Line	Net Counts	K-Ratio	ZAF	Weight %	Atom %
C K	120957	0.023	1.414	3.02	13.36
O K	25545	0.009	0.968	0.82	2.71
Al K	181058	0.054	0.983	4.93	9.69
Cr L	39689	0.048	1.092	4.84	4.94
Co L	47360	0.029	1.023	2.71	2.44
Ni L	1894739	0.717	1.032	68.05	61.50
Nb L	37580	0.027	1.214	3.05	1.74
W M	168359	0.092	1.490	12.59	3.63



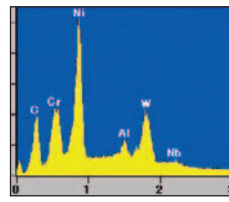
Element Line	Net Counts	K-Ratio	ZAF	Weight %	Atom %
C K	52133	0.022	1.346	2.68	12.18
O K	6500	0.005	0.937	0.43	1.48
Al K	47344	0.032	0.977	2.76	5.59
Cr L	53409	0.145	1.066	13.69	14.40
Co L	64991	0.088	1.071	8.38	7.77
Ni L	705787	0.598	1.098	58.14	54.14
Nb L	5951	0.010	1.204	1.03	0.61
W M	80764	0.099	1.476	12.89	3.83



Element Line	Net Counts	K-Ratio	ZAF	Weight %	Atom %
C K	114407	0.044	1.259	4.98	34.65
O K	12567	0.009	1.053	0.85	4.46
Ti L	3449	0.013	1.272	1.48	2.59
Ni L	96083	1.072	0.855	5.59	7.96
Nb L	169983	0.245	1.013	22.55	20.27
Hf M	453361	0.529	1.133	54.45	25.49
W M	81406	0.088	1.263	10.09	4.58



Element Line	Net Counts	K-Ratio	ZAF	Weight %	Atom %
C K	149992	0.057	1.276	6.31	37.50
O K	6429	0.005	1.188	0.47	2.10
Ti L	26362	0.099	1.321	11.22	16.72
Ni L	28814	0.022	0.906	1.69	2.06
Nb L	207219	0.300	1.033	26.70	20.52
Hf M	196903	0.231	1.174	23.33	9.33
W M	263141	0.286	1.231	30.29	11.76



Element Line	Net Counts	K-Ratio	ZAF	Weight %	Atom %
C K	14835	0.040	1.233	4.18	18.82
Al K	3732	0.016	0.926	1.23	2.48
Cr L	27974	0.471	1.077	43.46	45.20
Ni L	53125	0.279	1.176	28.08	25.87
Nb L	2930	0.030	1.173	2.97	1.73
W M	21938	0.166	1.412	20.08	5.91

Figure 6: Quant method is relative quant analysis without standards

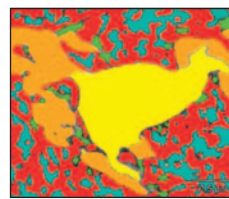


Figure 7

	Color	Area %
Phase 1	Red	37.38
Phase 2	Orange	16.44
Phase 3	Yellow	18.08
Phase 4	Green	2.76
Phase 5	Cyan	15.86

SEM/EDS data provided by JFE Techno Research Corporation

©2008 Thermo Fisher Scientific Inc. All rights reserved. All trademarks are the property of Thermo Fisher Scientific Inc. and its subsidiaries. Specifications, terms and pricing are subject to change. Not all products are available in all countries. Please consult your local sales representative for details.

In addition to these offices, Thermo Fisher Scientific maintains a network of representative organizations throughout the world.

- Africa**
+43 1 333 5034 127
- Australia**
+61 2 8844 9500
- Austria**
+43 1 333 50340
- Belgium**
+32 2 482 30 30
- Canada**
+1 800 530 8447
- China**
+86 10 8419 3588
- Denmark**
+45 70 23 62 60
- Europe-Other**
+43 1 333 5034 127
- France**
+33 1 60 92 48 00
- Germany**
+49 6103 408 1014
- India**
+91 22 6742 9434
- Italy**
+39 02 950 591
- Japan**
+81 45 453 9100
- Latin America**
+1 608 276 5659
- Middle East**
+43 1 333 5034 127
- Netherlands**
+31 76 579 55 55
- South Africa**
+27 11 570 1840
- Spain**
+34 914 845 965
- Sweden/Norway/Finland**
+46 8 556 468 00
- Switzerland**
+41 61 48784 00
- UK**
+44 1442 233555
- USA**
+1 800 532 4752

www.thermo.com



Thermo Electron Scientific Instruments LLC, Madison, WI USA is ISO Certified.

AN51220_E 07/08M