

# Brake pad analysis for wear characteristics, reverse engineering or failure analysis

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## Key Words

Pathfinder X-ray Microanalysis System, COMPASS PCA, automotive brake pad, energy dispersive spectroscopy, oxides, principal component analysis, phase mapping, X-ray microanalysis

## Introduction

Design and manufacture of automobiles take advantage of the lightweight, strength and energy absorbing qualities of composite materials. The braking systems of automobiles are no exception.

Brake pads and their lining are complex materials composed of four parts: binder, reinforced fibers, friction performance modifiers, and padding. Studying these materials at the microscopic level requires the ability to separate each of the many elemental phases in the material. Such a study is useful in analyzing wear characteristics, reverse engineering or failure analysis.

While elemental mapping in X-ray microanalysis will show the elemental distribution in the material, that only tells part of the story. Complex materials such as brake pads and linings consist of several discrete material phases which are generally individual mineral oxides. In order to perform a thorough analysis of the material, the analyst needs tools to characterize each unique phase.

Thermo Scientific™ X-ray Microanalysis Systems include a proprietary principal component analysis algorithm called COMPASS PCA. COMPASS PCA examines the collection of spectra obtained in a SEM/EDS acquisition using Spectral Imaging, which consists of a spectrum at every pixel in the acquired SEM image. This dataset serves as the statistical platform for the PCA analysis. COMPASS PCA groups each statistical unique phase without regard to element composition. This process is described in detail in a webinar recording titled “Unleashing the Power of COMPASS PCA.”

Currently there are four recognized types of friction brake compositions: asbestos, semi-metallic, non-asbestos organic (NAO), and ceramic.<sup>1</sup>

### Asbestos

Asbestos brake pads are the first generation of brake pads and contain about 40–60% asbestos materials. The biggest of its advantages is the low cost. However, asbestos materials can cause lung cancer and have largely been discontinued.

### Semi-metallic friction formulations

A semi-metallic brake pad consists of a lining that uses steel wool as a reinforcing fiber; most contain at least 60% steel by weight. The steel fibers act as the framework to lock the friction ingredients together.

### Non-asbestos organic friction formulations

Non-asbestos organic (NAO) brake pads consist of organic fibers that are used to reinforce the friction materials and provide strength to the brake pad. NAO friction material contains less than 20% steel by weight. These pads were designed to replace asbestos pads.

### Ceramic formulations

Ceramic brake pads have been in use since the mid-1980s and contain no steel fibers. Instead, they use ceramic and copper fibers to manage heat dissipation.

A major challenge in the manufacturing of automotive brake pads/shoes is controlling the ingredients and distribution of these components. Reproducibility of the formulation and the distribution of the raw materials is the key to obtaining good quality control of the final pad or shoe.

X-ray microanalysis in the scanning electron microscopy (SEM) uses energy dispersive spectroscopy (EDS) to characterize the composition of brake pad materials. The addition of a statistical approach to the analyses has been used to determine the distribution and phases of the components of brake pads/shoes.

### Materials and methods

The examined brake pad was worn and, thus, had undergone numerous heating and cooling cycles, exposure to various weather conditions, and, in addition, exposed to salt used to melt road snow.

An approximately 1 inch by 1 inch section of the pad was removed from the backing plate and embedded in epoxy. The embedded sample was polished through a series of grit sizes with a final polish of 1 micron grit. The uncoated sample was examined on a JEOL JSM-7001F scanning electron microscope (SEM). The EDS system used to

acquire and analyze X-ray data was a Thermo Scientific™ NORAN™ System 7\* (version 3.3 software) with a 10 square millimeter silicon drift detector. Spectral Imaging data was acquired at 20 Kv accelerating potential, 30× magnification and at a pixel resolution of 1024.

### Results

SEM examination of the sample shows various phases of the brake pad material with some fairly large phases (Figure 1).

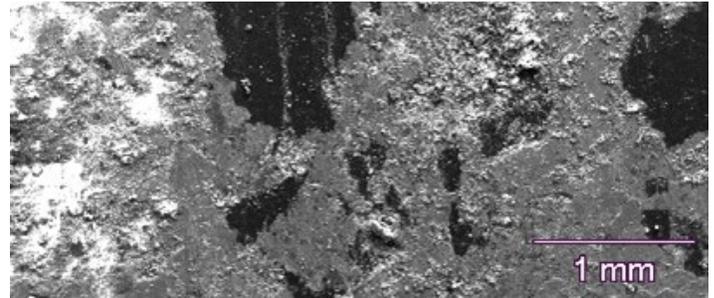


Figure 1: SEM image of brake pad sample

X-ray analysis of the sample shows 16 elements (Figure 2). Some elements, notably Fe, O, and C, are the most abundant. The presence of the Zn K line peak indicates that Zn is present; however, the Na K lines overlaps with Zn L lines which makes element identification of Na difficult. Figure 3 shows the elemental X-ray maps.

To further analyze the spectral imaging data, a red, green and blue (RGB) overlay of elements was used to discern some of the compounds that may be present. Because of the presence of Ba and S in the sample, the first compound to be explored was BaSO<sub>4</sub>. The yellow particles (red and green create yellow in color) indicate the presence of Ba and S in the same locations; thus, Ba and S are probably in the form of BaSO<sub>4</sub> (Figure 4). The red particles in Figure 4 indicate S present in some other compound. Many of the elements are in very low concentrations and sparse distribution. Figure 5 shows the distributions of Na, and Zn (yellow particles) which leads to the conclusion that Na may not be present.

The extracted spectrum of the larger yellow (Na and Zn) particle (Figure 6) shows the Zn L peaks are present but Na K peak is not present, suggesting that Na is not present in the locations of Zn. With so many light elements in this sample accompanied with the wide distribution of O, many of these elements might be in the form of oxides which can get quite complex in composition.

With this number of elements, using the RGB method is quite cumbersome and time consuming. To take advantage of a mathematical approach to further analyze the sample, COMPASS principal component analysis (PCA) was used.

Figure 2: X-ray spectrum from pad sample

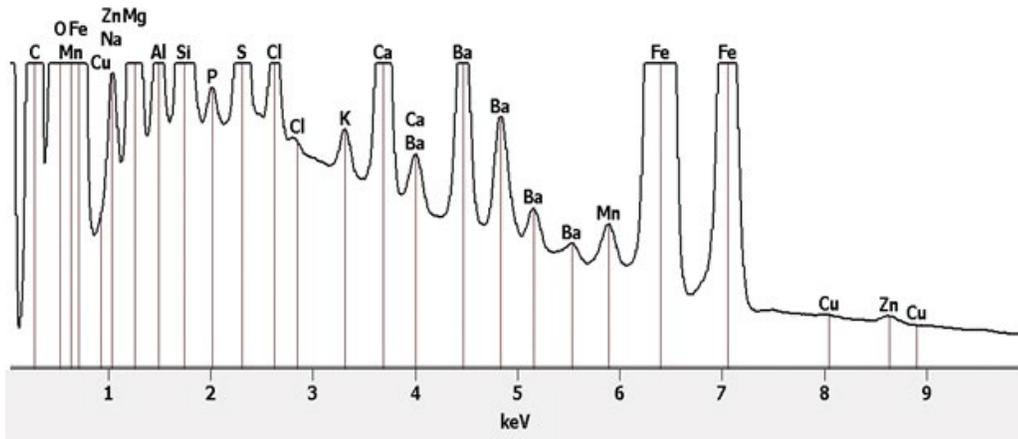


Figure 3: X-ray maps showing the element distribution from the brake pad sample

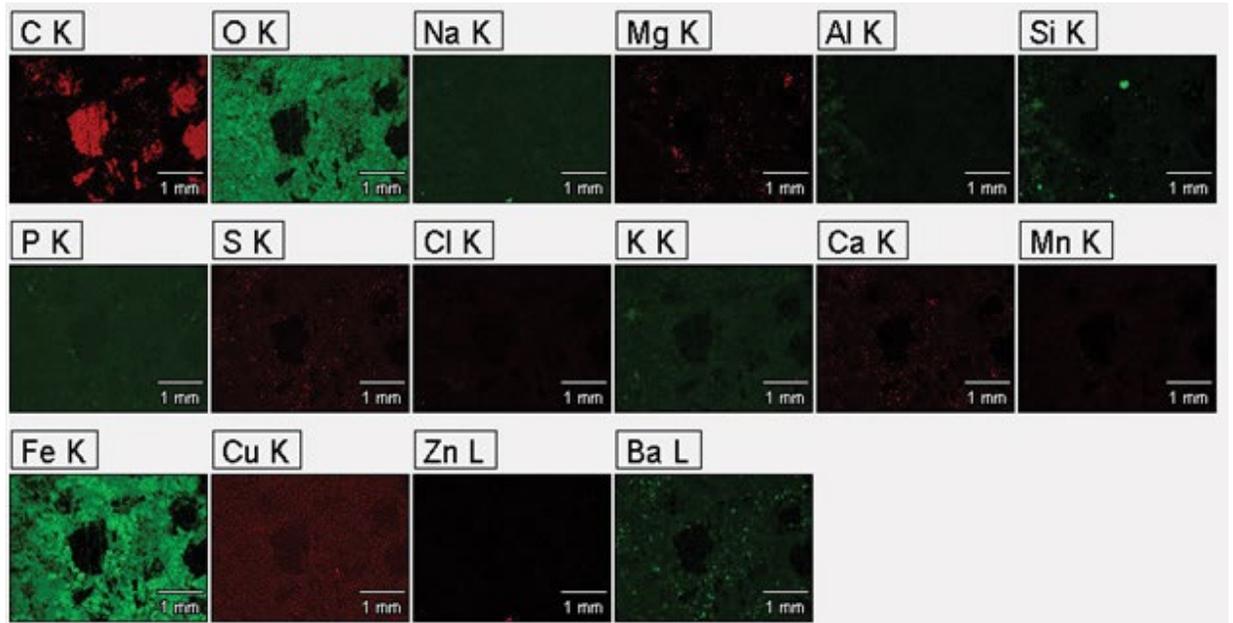


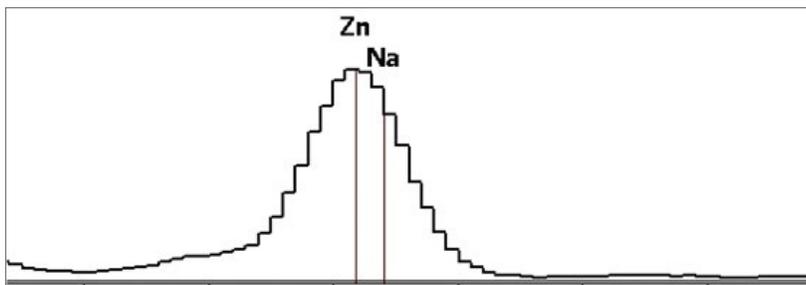
Figure 4: Distribution of Ba and S



Figure 5: Overlay of Zn and Na X-ray maps onto the gray reference image



Figure 6: Extracted spectrum of Na/Zn particle region



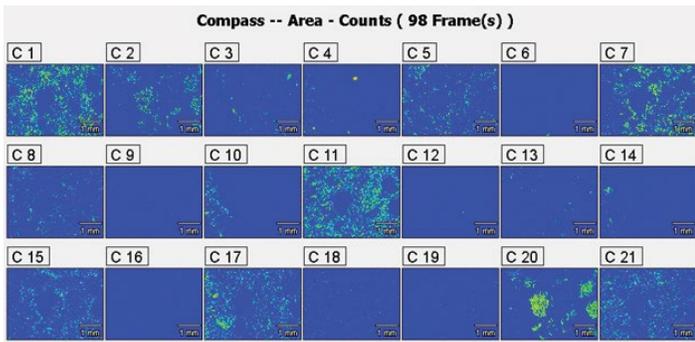


Figure 7: Principal components of the pad sample using the area method of analysis

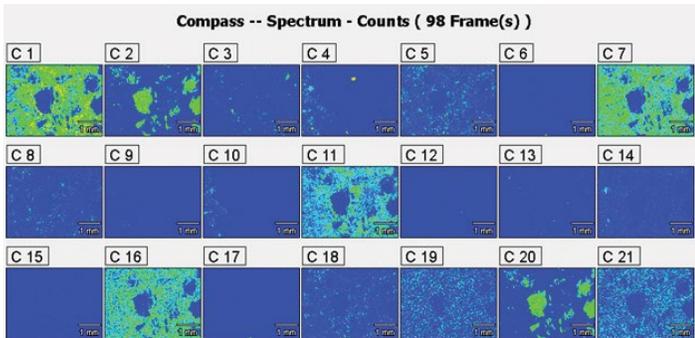


Figure 8: Principal components of the pad sample using the spectrum method of analysis



Figure 9: Comparison of Component 20 and Component 2 showing similarities in the C components

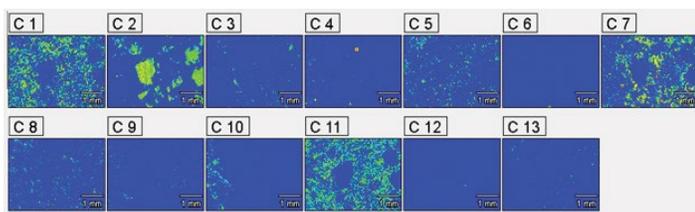


Figure 10: Extracting of the X-ray maps limited to 13 unique components

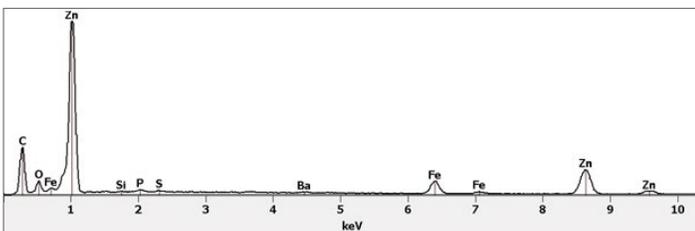


Figure 11: Component 6, Zn rich component

## COMPASS PCA results

There are two methods available for COMPASS PCA: 1) Area method and 2) Spectrum method. The area method places emphasis on the area of each component; whereas the spectrum method utilizes the spectrum to derive the components.

The results from COMPASS PCA show 21 components using the area method (Figure 7) or spectrum method (Figure 8) of analysis. Inspection of the component images and the corresponding spectrum, some of the components can be combined, for example C2 and C20 are the carbon component (Figure 9).

After inspecting the spectra from each of the components, some of the components are very similar in composition, differing only in the concentration of the elements. Comparison of the various components indicates that there are only 13 distinct components. Setting the total number of components to 13 in COMPASS PCA and re-extracting the components yields 13 fairly unique components (Figure 10). From the component maps, C6 is the Zn component (Figure 11) and C12 is an FeCuS component (Figure 12). Both are preserved as individual components in the analysis. Now the task is to examine each of the component spectra and determine if more can be combined.

Comparing C1, C7 and C11 show similar elements (Figure 13). However, component 1 contains a higher Al content than C7 and C11. On the other hand, C1 and C11 contain Zn whereas C7 does not. The comparison of each of the components is time consuming. However there is a simpler, faster way, which is to take advantage of the power of statistics and let the COMPASS engine mathematically determine the phases. Figure 14 shows the phases extracted from the reduced 13 COMPASS components.

Inspection of the spectra of each of the phases and the distribution of each phase paints a picture of the composition of the brake pad (Figure 15).

Overlaying the ten phase distribution images onto the reference electron image shows the unique distributions of each of the phases (Figure 16).

Figure 12:  
Component  
12, FeCuS rich  
component

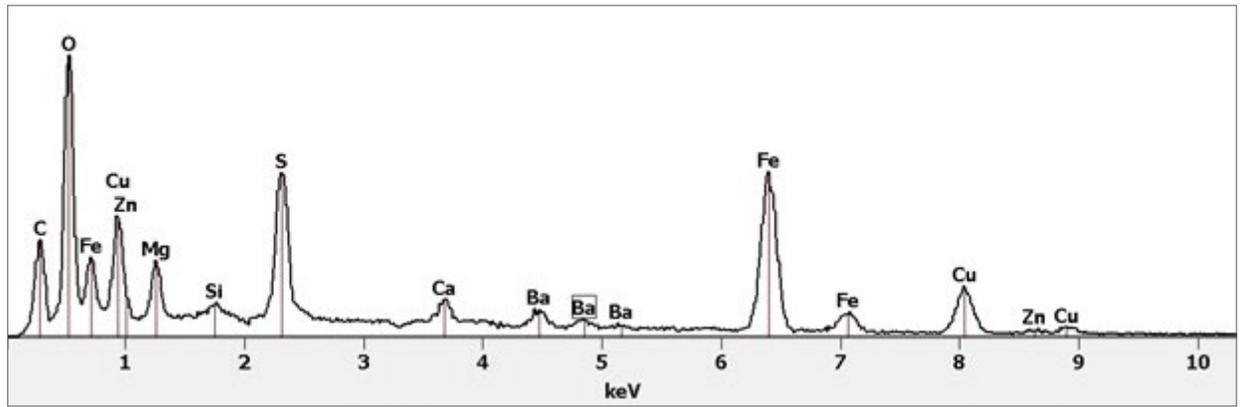


Figure 13:  
Comparison of  
spectra from C1, C7  
and C11

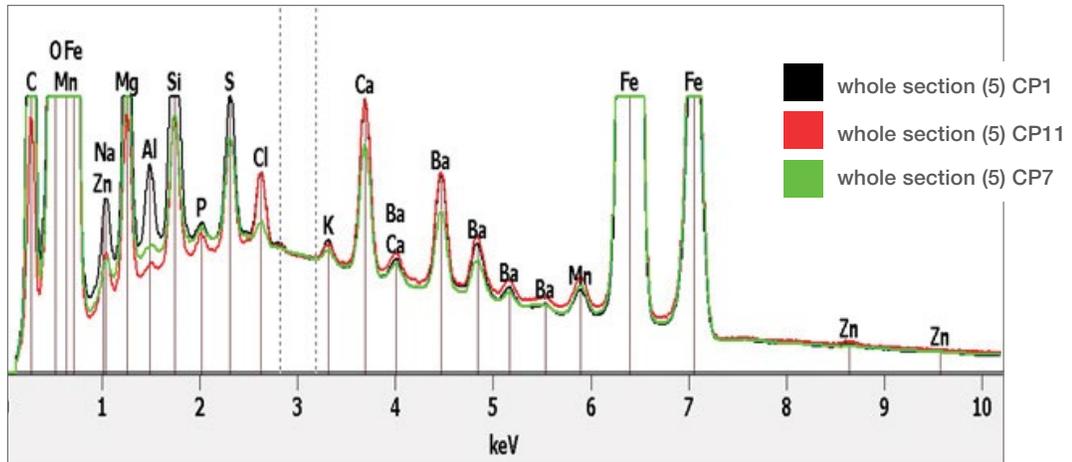


Figure 14:  
Phases calculated  
from 13 components

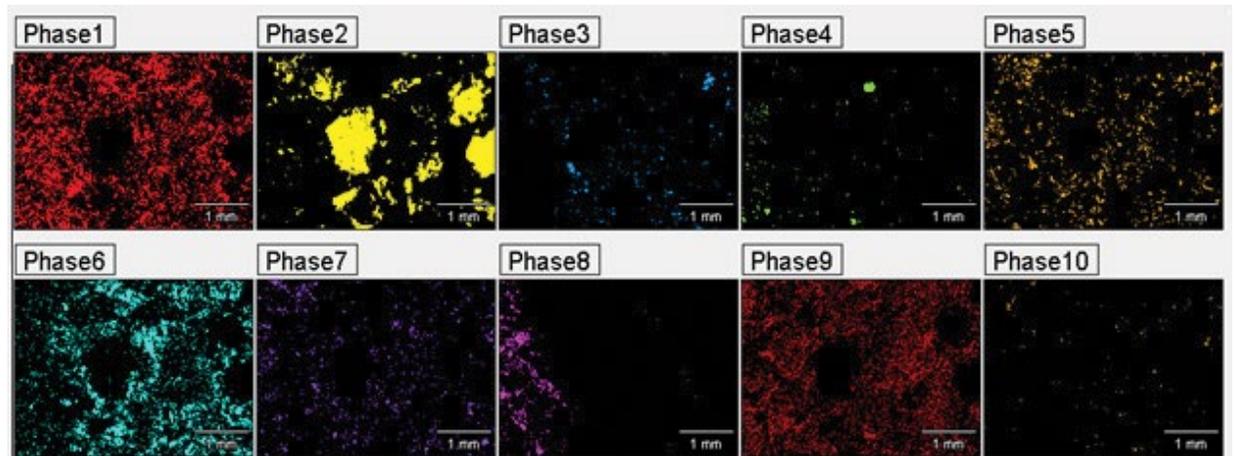
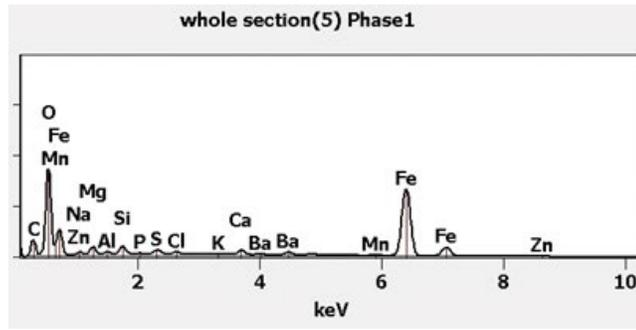
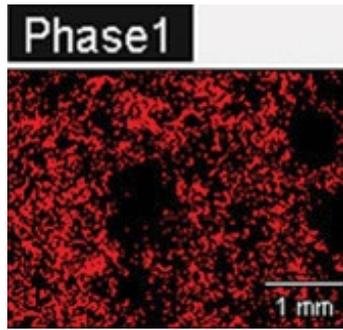
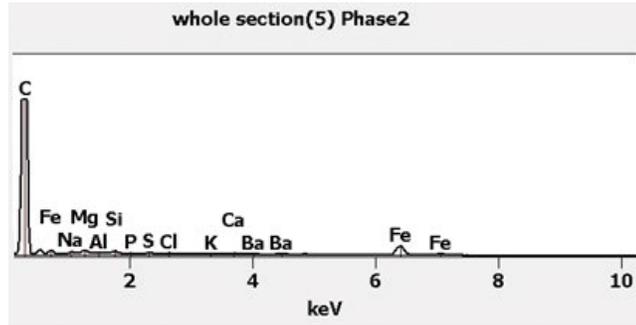
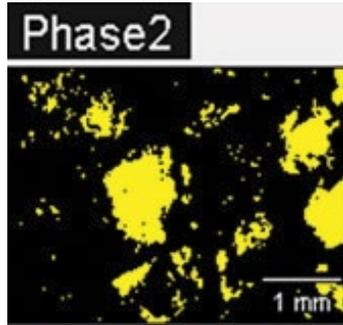


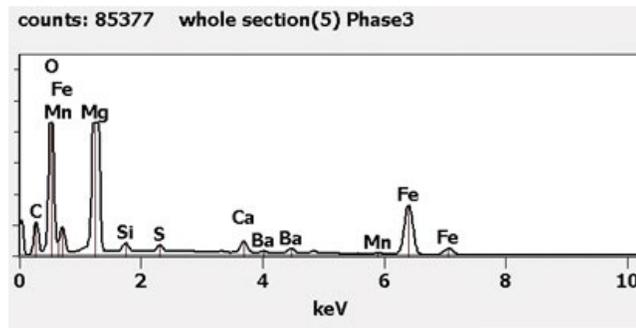
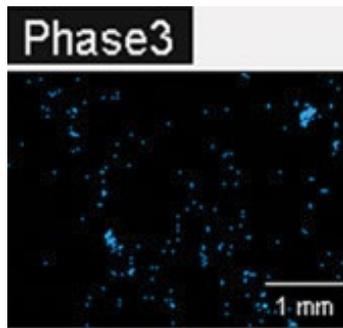
Figure 15: Phases analysis showing the composition of the brake pad



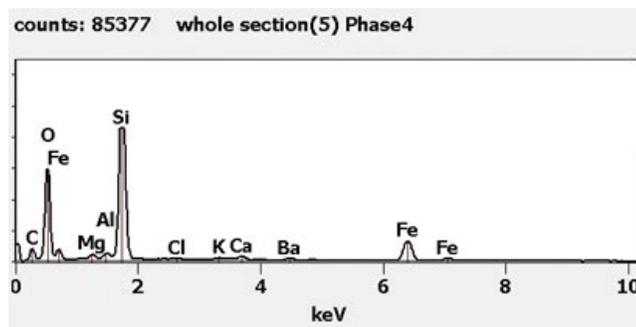
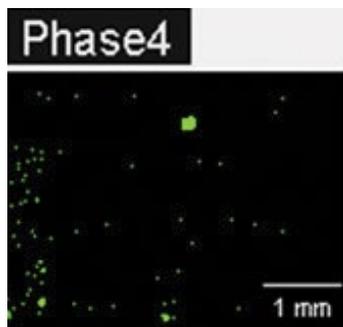
Phase 1: Iron Oxide rich phase



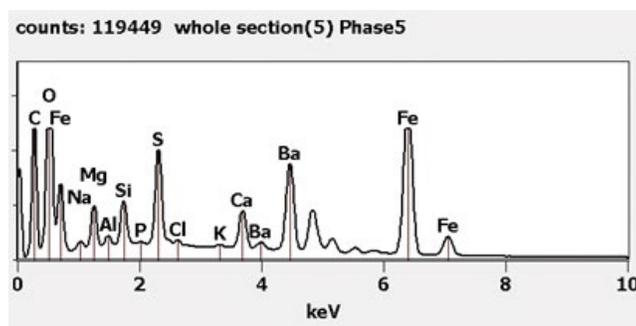
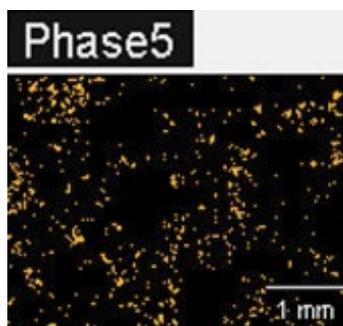
Phase 2: Carbon rich phase



Phase 3: Magnesium Oxide rich phase

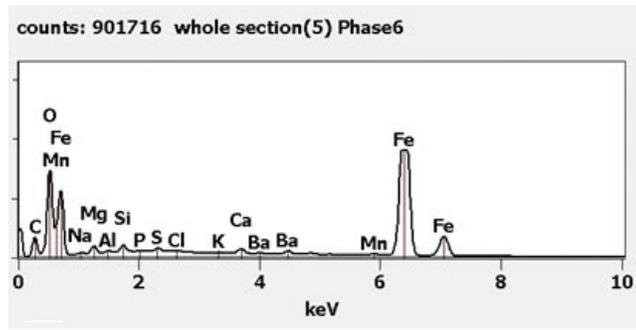
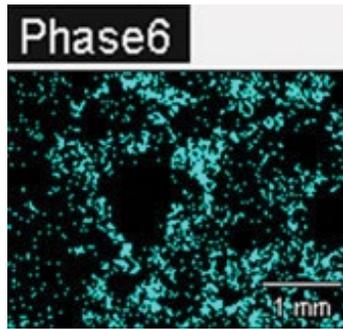


Phase 4: Silicon dioxide rich phase

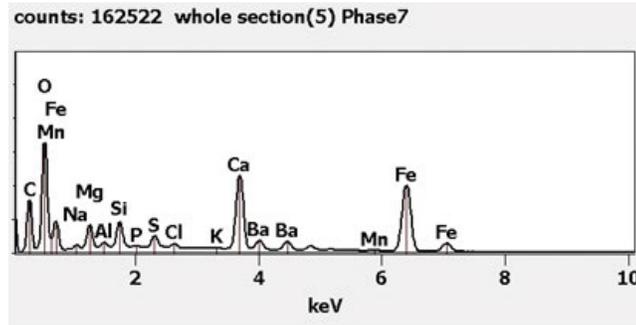
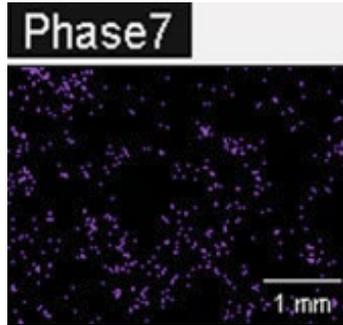


Phase 5: Barium sulfate rich phase

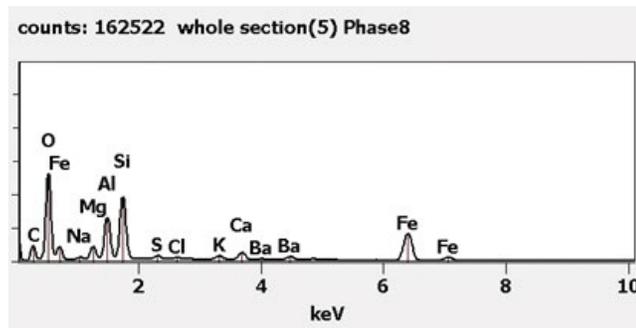
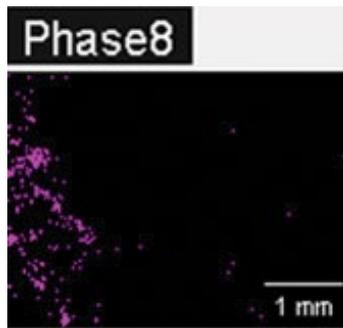
Figure 15: Continued



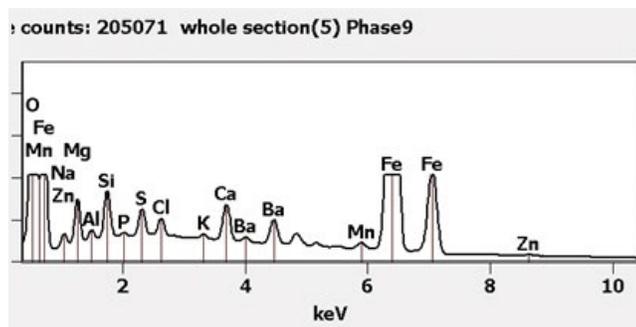
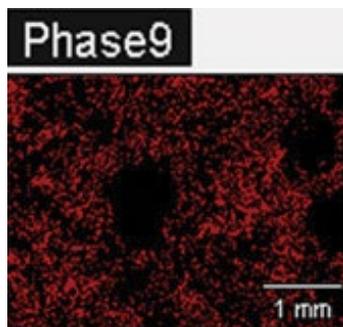
Phase 6: Iron rich phase



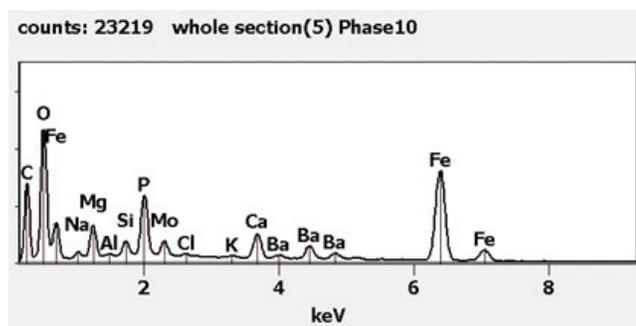
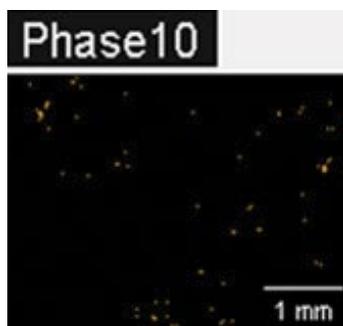
Phase 7: Calcium silicate rich phase



Phase 8: Si Al Mg Na O rich phase



Phase 9: Zn and Cu phase but overall elements are the same as for the entire field of view



Phase 10: Phosphate rich phase

## Conclusions

The automotive disc brake examined is a semi-metallic type pad indicated by the high iron content from EDS analysis. In addition to Fe there are at least 15 other elements that make up the brake pad formulation. This particular sample is very complex in composition and is difficult to analyze. Using the RGB method of overlaying the element maps would be very time consuming. However, taking advantage of a statistical approach to the analysis makes the analysis easier and less time consuming. The approach of using Spectral Imaging to obtain element maps followed by COMPASS PCA to derive principal components and then using these components to determine phases is less time consuming than using just X-ray maps alone. Further, there is very little human bias introduced into the analysis using this approach.

This application note demonstrates a fast and comprehensive approach to analyzing complex materials that include a large number of elements that form several compounds through the use of Spectral Imaging and Principal Component Analysis.

## Reference

1. [www.waynesgarage.com/docs/brake\\_material.htm](http://www.waynesgarage.com/docs/brake_material.htm)

*\* The Noran System 7 has been replaced by the Thermo Scientific™ Pathfinder™ System.*

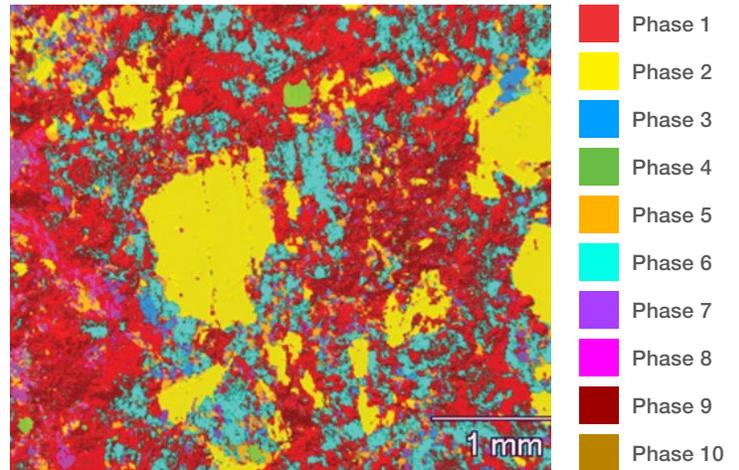


Figure 16: Overlay of the 10 phases onto the gray level reference image showing phase distributions

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