USE OF DGC SEPARATION, OPTICAL MICROSCOPY AND EDAX TECHNIQUES IN THE PHASE ANALYSIS OF BRAKE COMPONENTS

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Introduction

The performance of friction materials is a complex function of numerous parameters. One of the most significant, the formulation (phase composition) is very important to the brake performance. However, the relationship between brake formulation and performance is not well understood and this is a significant limitation to tailored design. In fact, it pushes industry to a "trial and error" formulation strategy. The ability to precisely analyze the phase composition of brake materials is needed both to investigate this relationship and to maintain quality control in production. Because a typical brake composite can contain more than ten phases that can be both crystalline and amorphous, electron probe analysis (EDAX), x-ray diffraction analysis and optical microscopy (reflected and fluorescent light) techniques are needed to identify all of the phases. The objective of this study was to investigate an analysis protocol using these techniques for the identification of the various phases in brake composites. Because electron probe and optical techniques fail to give definitive identification on some components, x-ray diffraction analysis is used because it can reliably identify all crystalline components. However, in composites such as brake materials the large number of components can give a multitude of overlapping diffraction patterns that can not be reliably interpreted. This problem has been overcome by using density gradient centrifugation to separate the micronized sample into density fractions that are easier to identify.

Experimental

A sample of a brake pad from industrial production was used for demonstration of our analytical approach. The sample was pulverized to -200 mesh by srandard techniques and this material was divided into divided into magnetic and non-magnetic parts using magnetic separation techniques. The non-magnetic fraction was micronized in a fluid energy mill and then processed int 56 density fractions in the range of 1.3239 g/ml to 2.2623 g/ml using a density gradient centrifugation (DGC) technique.. Metal particles from the residuum (sink fraction) obtained after application of DGC technique were separated under microscope to identify their composition more precisely non-magnetic metals (copper, brass etc.).

XRD analyses were performed on an INEL powder diffractometer with the position sensitive detector PSD 120. Operating conditions: transmission mode, sample in the capillary, Cu radiation and Ge-monochromator. Surface of the brake composite was investigated by light microscopy (LM, Nikon, polarized, white and fluorescent light) to identify types of carbon and organic materials (like a plastic, rubber etc.). To obtain information about composition of individual particles of brake composite scanning electron microscope equipped with Link Energy Dispersive X-ray Analysis (Philips SM-30 analytical system) was used.

Results and Discussion

The whole sample (before fractionatin) of the brake composite is very complex material. SEM analysis of the surface of brake composite reveals particles of Mg-vermiculite (Mg,Si,Al,O), graphite, copper, iron , and Ca-silicate. The XRD powder pattern of the whole sample (before fractionatin) is also very complex with the dominant 002 diffraction of graphite and overlapping diffractions of zircon ($ZrSiO_4$), stibnite (Sb_2S_3), molybdenite (MoS_2), copper and iron. Intensities of their overlapping diffractions are significantly smallest than the intensity of 002 diffraction of graphite, and therefore, the identification of individual phases and their resolution is very difficult.

To improve the resolution of diffraction pattterns of the individual phases both magnetic and DGC fractionation process were applied to the whole sample. In the XRD pattern of magnetic fraction only iron was found. The non-magnetic part wast fractionated using density

gradient centrifugation (DGC) technique to prepare 56 individual fractions with the densities between 1.3239 g/ml and 2.2623 g/ml. The density profile of the individual fractions reveals three different peaks representing three different groups of materials of the brake composite - one containing plastic, rubber and another organic materials with the lowest density. The second one corresponds to poor-crystalline carbon material (coke) and the last one a well-crystallized graphite (see figures 1 and 2).

Conclusions

The results of this study show that all three techniques (with XRD being the most definitive) can identify phases made of graphite, copper, iron, vermiculite, zircon, and stibnite, electron probe analysis was best for identifying zinc and metal alloys, and the polarized reflected light optical microscopy was best for identifying petroleum and metallurgical coke, carbon fibers, chars, coal, and rubber, while fluorescence microscopy was best for organic fibers, plastics, and phenoloic resins

Table 1. Results of the phase analysis in the brake composite material using XRD, LM and ELMI (EDXA) techniques. Identified phase are denoted by solid circle.

Phase	**Phase identification procedure**		
	XRD	**LM**	**ELMI**
Graphite	·	·	·
Coke	·	·	·
Plastics	-	·	-
Rubber	-	·	-
Organic fibers	-	·	-
Phenolic resin	-	·	-
Copper	·	·	·
Iron	·	·	·
Zinc	-	-	·
Mg-vermiculite	·	·	·
Zircone ($ZrSiO_4$)	·	·	·
Molybdenite (MoS_2)	·	-	·
Stibnite (Sb_2S_3)	·	·	·
Mg,Al-chromite *	-	-	·
Ca-silicate *	-	-	·

* Only approximate phase identification from the chemical composition of particles determined by EDXA.

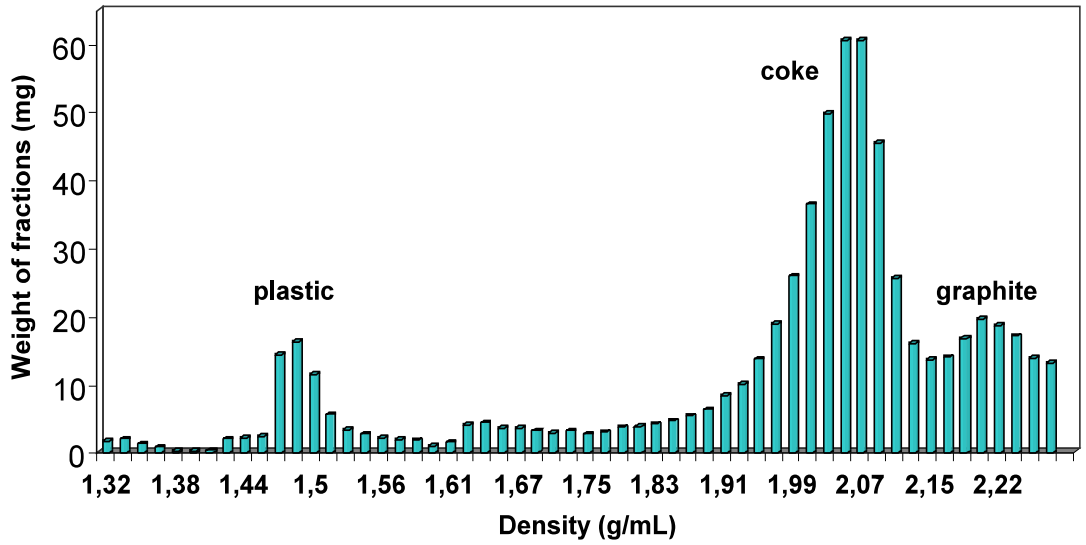

Figure 1. Densiy profile of the lighter portion of the brake sample.

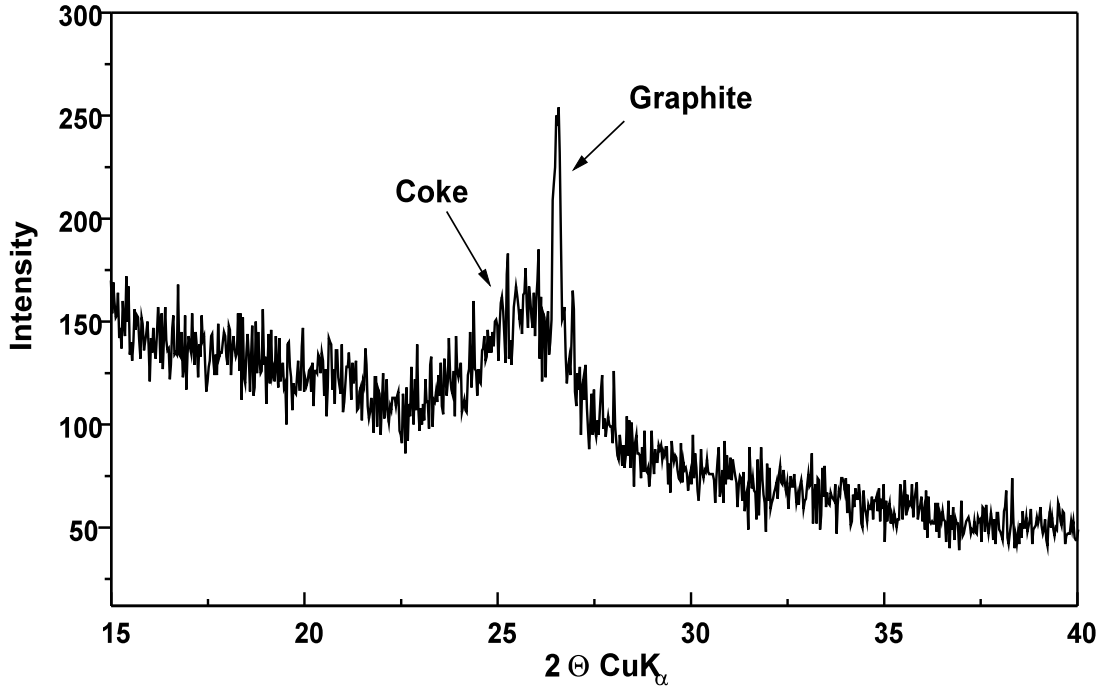

Figure 2 . Portion of XRD pattern showing the weight fraction with the density of 2.1157 g/ml.