Rigaku/MSC, Inc.
XRD Application Note

XRD Characterization of Refractory Powders by Search/Match Analysis

Introduction and Purpose

X-ray Diffraction (XRD) is an effective method for identifying the phases present in unknown polycrystalline powders. The analysis is performed by comparing the diffraction pattern collected from an unknown sample with the diffraction patterns of known compounds. The automated process is called Search/Match (S/M) analysis.

XRD is an important technique in the manufacture of ceramic materials. It provides phase analysis of materials throughout the manufacturing process, from raw materials to products.

For this characterization report, the diffraction patterns of ten refractory powder specimens were compared with the diffraction scans of pure compounds maintained in the ICDD Powder Diffraction File (PDF). Data reduction and S/M analysis was performed using the JADE+ software package.

Sample Preparation and Analysis

Ten refractory powders, labeled 1-10 were submitted for XRD analysis. Each powder was subsequently identified as:

Powders 1-3 Product Materials
Powders 4 and 5 Bauxite (South American and Chinese)
Powders 6 and 7 Refractory Powders
Powders 8-10 Starting materials (Mullite)

The powders were front packed into standard round aluminum specimen holders and leveled with a glass slide. No special effort was made to control the preferred orientation in the grains. Standard θ/2θ data were collected using the MINIFLEX+ diffractometer.

Operating Parameters

The MINIFLEX+ vertical-oriented goniometer, uses a Copper sealed tube operated at 30kV, 15 mA with a 6º take-off angle. The goniometer radius is 150 mm. The slit system consists of a variable (theta compensating) divergence slit, a 4.2° scatter slit, a 0.30 mm receiving slit and a Nickel K_β filter. The detector is a Scintillation Counter.

A region of 5-100° 2θ was scanned at 4 degrees/minute. The total scan time for each sample was 24 minutes.

Results and Comments

All scans were smoothed, theta corrected, and the background was removed. The results of Search/Match analysis are shown below each plot as a set of stick lines from the PDF.

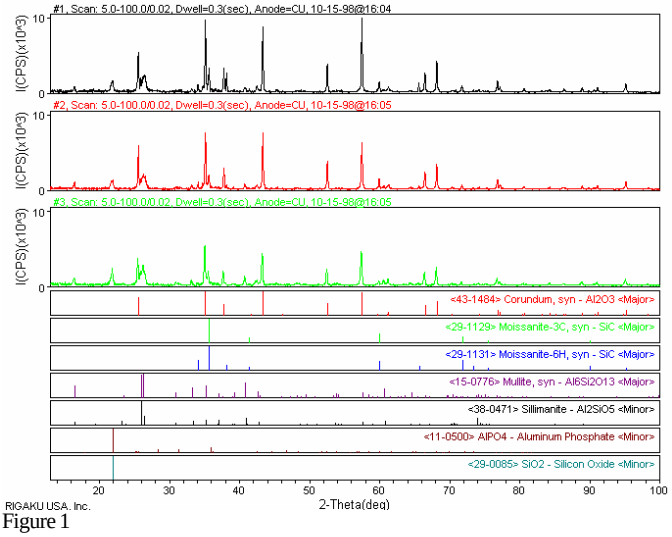

Figure 1

The diffraction profiles of powders 1, 2 and 3 suggest that each specimen is composed of nearly identical components. Good matches are Corundum, Mullite, Moissanite, Sillmanite, Aluminum Phosphate and Silicon Oxide. Mullite and Sillmanite are both aluminosilicates. They have very similar diffraction patterns. Aluminum Phosphate and Silicon Oxide are isostructural. Additional elemental information would facilitate positive identification.

There are some intensity differences between samples. Qualitatively some conclusions may be drawn, since the intensity of randomly oriented peaks is proportional to the amount of the substance. The major phase in each sample is Corundum. Powder 1 has the most Corundum and powder 3 the least. Powder 1 contains more Moissanite than the other two samples. It would be possible to get more accurate quantitative information if a series of standards was available to relate the area intensity to a concentration.

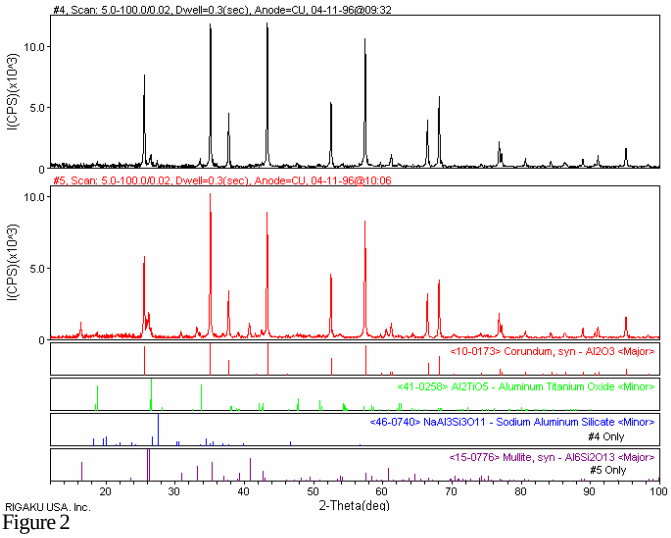

Figure 2

Data from powders 4 and 5 indicate both specimens have Corundum, and Aluminum Titanium Oxide present. The diffraction pattern of Powder 4 has a peak at 27.5° 2θ which corresponds to the major peak of Sodium Aluminum Silicate. Powder 5 contains Mullite as an additional phase.

Conclusions

The Rigaku MINIFLEX+ X-ray Diffractometer, combined with the Jade Analytical software package, is an effective tool for phase identification of unknown powder samples. Clear interpretation of the presence of absence of various phases can

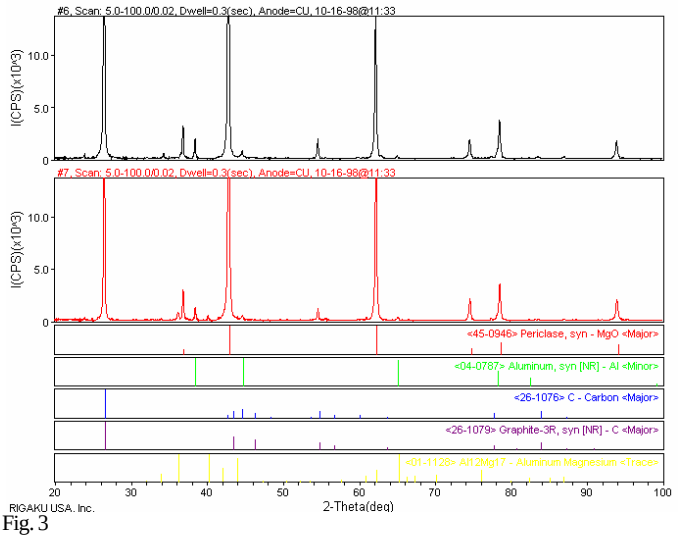

Fig. 3

Powders 6 and 7 contain 3 common phases; Periclase, Aluminum and Carbon. XRF analysis of these samp les confirms that Aluminum is present, and is not an artifact from the sample holder. Additionally, in powder 7, an Aluminum Magnesium phase is present.

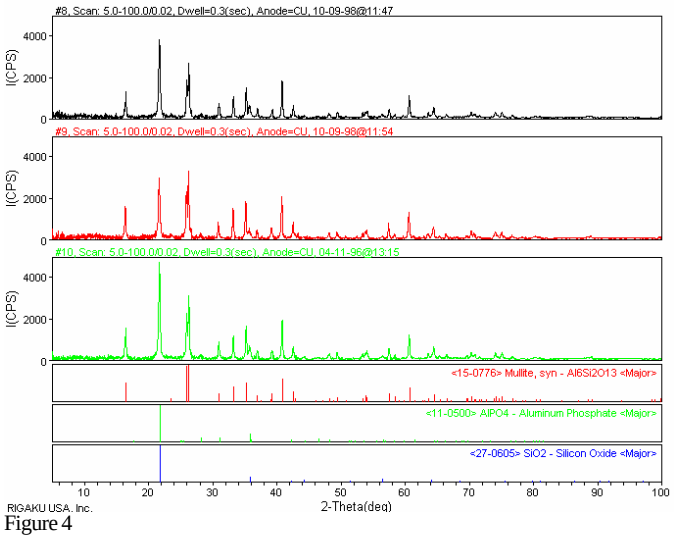

Figure 4

Powders 8, 9 and 10 all contain Mullite, Aluminum Phosphate and/or Silicon Oxide.
be performed quickly and with a high degree of confidence. Phase identification is easily performed, even on quick data collection scans with high statistical noise. Specimen preparation for most standard materials is minimal since bulk materials as well as powder samples can be analyzed.

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