Rigaku/MSC, Inc.
XRD Application Note

Fast Phase Analysis of Mineral Powders with the MINIFLEX+ Diffractometer

Introduction and Purpose

X-ray diffraction (XRD) is an effective method for determination of the phase composition of unknown crystalline and amorphous materials. The analysis is performed by comparing diffraction peak positions and intensity values with the reference patterns of known compounds maintained in the ICDD Powder Diffraction File (PDF). Routine characterization is completed quickly with a minimum of operator involvement. Subsequent analyses, including quantitative analysis of identified phases, are easily accomplished.

The goal of this work is to quickly identify the major phases in a series of unknown mineral powders using the MINIFLEX+. Data reduction and Search/Match analysis was performed using the JADE+ software package.

Sample Preparation and Analysis

Bulk samples were ground into fine powders using a mortar and pestle. Specimens were prepared from the samples by smearing on glass slides coated with a thin layer of Vaseline. 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, was operated using the following conditions:

450 W Cu Sealed Tube System, 30 kV @ 15 mA
6.0° Take Off Angle
150 mm Diffractometer Radius

Slit System
Variable (theta compensating) Divergence Slit
4.2° Scatter, 0.30 mm Receiving
Ni K_β filter

Scan Conditions
0.02 ° step; 9 degrees/minute scan rate

Results and Comments

The MINIFLEX+ was able to identify the major phases of the minerals with little sample preparation and a short analysis time. The total time of each of the first 4 scans presented was 10 minutes. Increased analysis time will significantly increase the signal to noise ratio and will thus improve the detection limits of minor phases. Data set 5 was acquired in a longer period of times, since the peaks were not as intense as in the other samples.

The data presented below has been smoothed and background corrected. The stick patterns from the PDF file of the best search/match results are printed below each scan.

"Good" Search/Match results match both the position and intensity of peaks. In a good match, there will be no unidentified peaks. Since many compounds have similar crystal structures, S/M results will list several "good" matches. Additional information about the sample may be needed to definitively identify a phase.

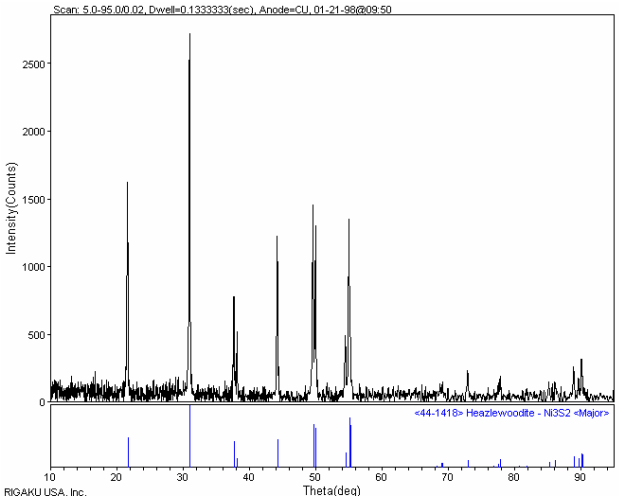

Fig. 1

Figure 1 shows one of the minerals analyzed. The top hit is for this sample is Heazlewoodite, a Nickel Sulfide mineral.

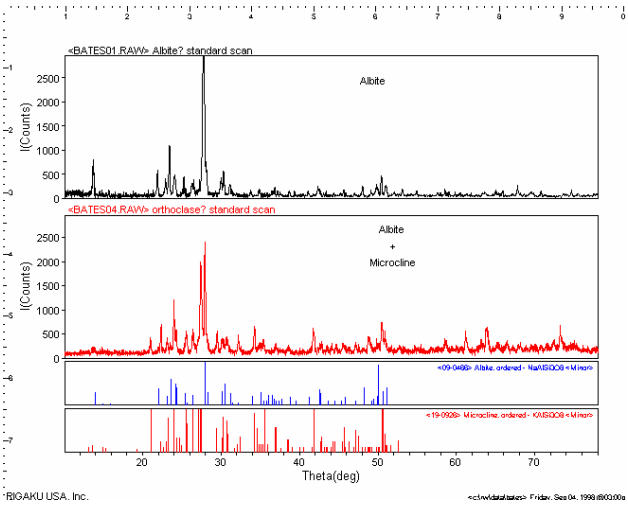

Fig. 2

Figure 2 shows the XRD patterns from two separate samples. The first is identified as albite. The second scan has the same albite peaks, as well as a number of additional peaks. Another phase is present. This phase is identified as microcline.

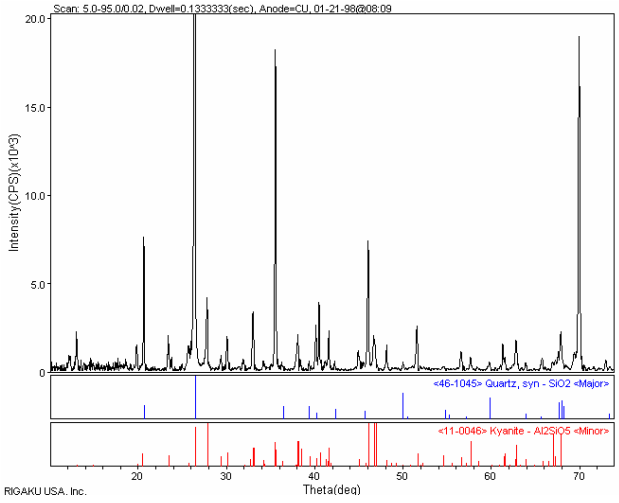

Fig. 3

Figures 3 and 4 are both patterns that do not match just one pattern from the PDF. Figure 3 is a mixture of quartz and kyanite, an aluminosilicate mineral. Figure 4 is best matched by a combination of pyrophyllite, andalusite and kaolinite.

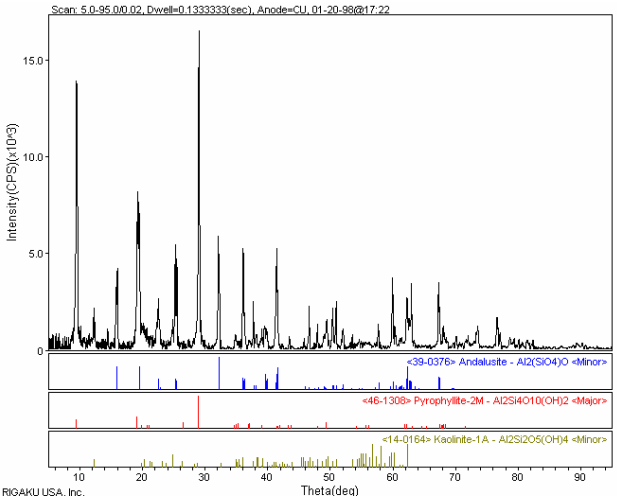

Fig. 4

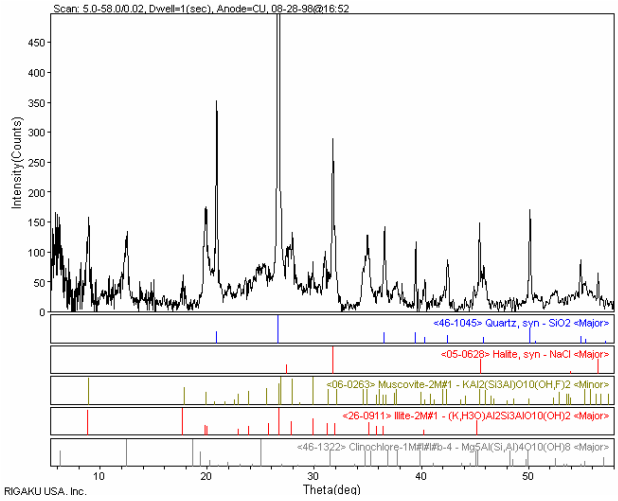

Fig. 5

Figure 5 is a multiphase sample. This sample was run more slowly than the other samples, in order to get higher intensity which makes it easier to effectively identify many of the peaks. Major phases include quartz, halite, clinochlore and either illite or muscovite.

Conclusions

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

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