SIMULTANEOUS XRD/XRF WITH LOW-POWER X-RAY TUBES

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ABSTRACT

A test bench instrument constructed at MOXTEK, Inc. is capable of simultaneously capturing X-ray diffraction (XRD) and X-ray fluorescence (XRF) information using a charge-coupled device (CCD) as the X-ray detector. NASA is funding the instrument's construction because of its low-power consumption and compact size; it could be used for in-situ planetary exploration missions for mineral analysis. A powdered sample of material is placed in front of the CCD. A collimated X-ray beam bombards the sample, and the CCD captures the scattered X-ray events. Sorting algorithms are used to separate the XRF and the XRD information captured by the CCD. A small low-power X-ray source is needed to make the device portable. We have examined the instrument with a rotating-anode tube, a commercially available Svetlana transmission tube, and two miniature low-power prototype tubes constructed at MOXTEK. The data capturing rates were compared for the different sources. We have verified the feasibility of capturing both XRF and XRD with the MOXTEK source, using under five watts for both the tube and its high-voltage power supply.

INTRODUCTION

A first prototype XRD/XRF instrument was developed at NASA Ames Research Center and is currently in use at Los Alamos National Laboratory [1,2]. The intended applications for this instrument are for planetary exploration and as a portable instrument for terrestrial use. The planetary missions have used methods that give elemental composition, such as the Mars Pathfinder probe that used XRF. XRF gives the elemental composition and suggests certain minerals, but the traditional method of mineral identification is by XRD [3]. With a small, portable XRD/XRF instrument, both diffraction and fluorescence information can be simultaneously gathered, providing accurate mineral identification.

The purpose of the test bench setup constructed at MOXTEK is to incorporate and test the low-power X-ray tube in an XRD/XRF instrument design. Initially we tested the CCD detector for X-ray detection and developed algorithms for sorting the data. For the initial setup we used a Rigaku RU-200 rotating anode tube for the X-ray source. For the next stage of testing we compared data taken with a high-power rotating anode tube and three low-power sources—a commercially available transmission anode tube and two MOXTEK side-window tubes. We successfully captured both XRD and XRF data with each of the X-ray sources. The information gained from the test bench will lead to a small portable prototype instrument. We will first describe instrument and the sample preparation. We will discuss how the CCD captures the data. We will then give a description of the MOXTEK tube. Finally, we will present the test data obtained using the low-power MOXTEK tubes and the commercial sources.

EXPERIMENTAL SETUP AND SAMPLE PREPARATION

The components of the XRD/XRF instrument are the Charge-Coupled Device (CCD) camera, pinhole optics, sample holder and X-ray source (Fig. 1). Figure 2 is a picture of the instrument with the MOXTEK tube. A Princeton Instruments CCD camera was used to acquire data. The camera has a front-side illuminated (FSI) CCD with an active area of 8.4 mm x 12.7 mm (EEV CCD02-06 deep depletion). The X-ray tube supplies the X-rays that are either absorbed or scattered by the powder sample. The tubes that have been used are a Rigaku Rotaflex RU-200 rotating anode tube, a Svetlana Electron Device Manufacturing Corporation (St. Petersburg, Russia) tube type bC1 copper anode, and two MOXTEK ***Bullet***™ side-window tubes. The pinhole optics are used to collimate the X-ray beam from the X-ray tube for diffraction. Pinhole sizes used range from 50 μm to 300 μm and the distance between the pinholes used ranges from 10 mm to 60 mm. The sample holder supports the sample in the beam and has minimal X-ray scattering [4,5].

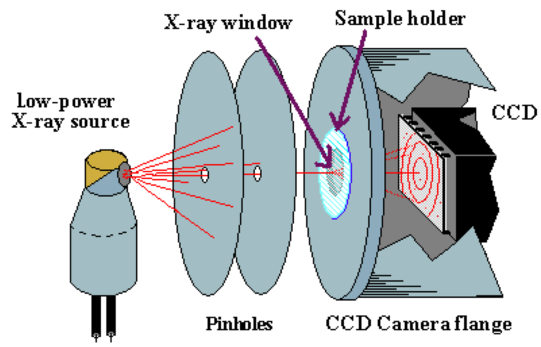

Figure 1. Concept drawing for the XRD/XRF instrument.

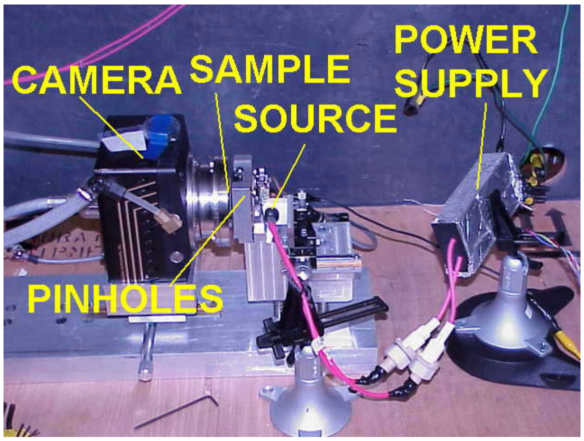

Figure 2. Picture of the XRD/XRF bench top instrument.

The samples discussed in this paper were prepared by grinding the KCl sample with a mortar and pestle into a powder. MOXTEK's AP1 films were used to support the samples because of their X-ray transparency. A 30 μm to 40 μm thick layer of the powder sample adheres to the film by using a small amount of vacuum grease (silicon or graphite). Finally, the sample is placed in front of the CCD, which captures the diffracted X-rays transmitted through the sample.

X-RAY DETECTION USING A CCD

The CCD uses each pixel as an individual energy-dispersive detector. This allows the spatial position (XRD information) and the energy of an X-ray (XRF information) to be recorded. To obtain unambiguous energy information, each pixel must only record one

X-ray event [5]. Once all the events are recorded, the diffraction information and the fluorescent information are extracted with the use of sorting algorithms [5,6].

The major factors that affect the energy resolution of the CCD are temperature and exposure time. Cooling the CCD and shortening the exposure time increase the energy resolution. The CCD is sensitive to X-rays in the region of 1.5 keV to 12 keV. The depletion region of the CCD limits detection of energies higher than 12 keV and the gate structure on the CCD limits energy detection below 1.5 keV. With the CCD cooled to -50° C with five-second exposures' the energy resolution of the CCD is 185 eV at 5.89 keV. Figure 3 shows an XRF spectrum collected with the CCD camera from a powder KCl sample. In the figure, chlorine Kα and potassium Kα and Kβ emission from the sample can be seen. A copper Kα diffraction peak can also be seen that is caused by X-ray diffraction scattering.

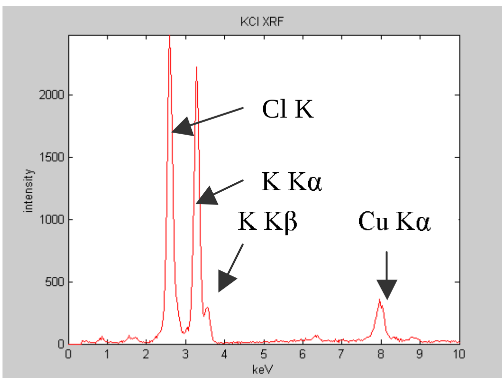

Figure 3. The XRF spectrum from a KCl sample.

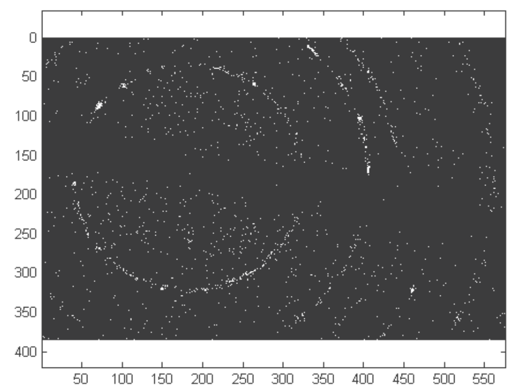

Figure 4. The XRD imaga KCl sample.

The angular resolution of the test bench instrument is limited by the X-ray spot size of the incident beam and the beam divergence. By using various pinhole configurations the spot size can be varied from 60 µm to 150 µm when viewed on the CCD. This provides an angular resolution of 0.4° to 0.7° FWHM in a 2θ diffraction plot**.** With the present configuration, resolution of the diffraction patterns may be slightly improved by using smaller pinholes, but smaller pinholes also reduce the flux of X-rays impinging upon the sample [5].

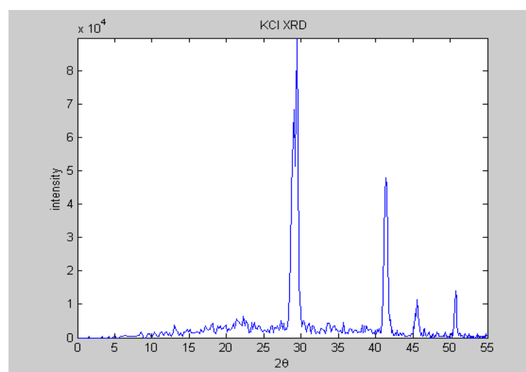

Figure 5. The XRD 2θ plot from a KCl sample.

The angular range of X-ray diffraction of the instrument is based primarily on the distance from the sample to the detector and the size of the CCD. The angular resolution of the instrument could be greatly improved by moving the CCD further away from the sample, but this would decrease the range of angles the CCD could observe. The range of the X-ray diffraction peaks captured is ~10° to ~50° 2θ if the beam spot is placed in the middle of the CCD X-ray window. The range of diffraction peaks can be increased to about 65° 2θ if the beam spot is placed at the edge of the CCD, but under these conditions full diffraction rings will not be captured. Figure 4 shows the rings in diffraction image captured from a KCl sample. Figure 5 shows a plot generated from the image that gives the intensity of the rings as a function of angle.

The characteristics of the CCD make it an excellent detector for the instrument. It is able to capture both the energy and the position of X-ray events. This allows the X-ray events to be sorted by algorithms; thus, we do not attenuate the events with filters to get the desired X-ray energy. Also, the CCD captures a large solid angle, i.e., every event within 35° 2θ will hit the detector. For a low-power instrument this is ideal because there will be few scattered events with a low power X-ray source. The CCD allows the instrument to maximize the detection of every scattered event.

LOW-POWER TUBE CHARACTERISTICS

The low-power MOXTEK tubes have a grounded anode with a tungsten filament cathode. Figure 6 shows the construction of the side-window tube design with all major components identified. The ceramic tube body isolates the anode and the cathode and is sealed so the inside of the tube maintains a vacuum environment. The silicone potting allows the tube to operate at higher voltages by eliminating airborne arcs between the anode and the cathode on the outside of the tube. The anode and cathode optics direct and focus the electron beam onto the copper anode.

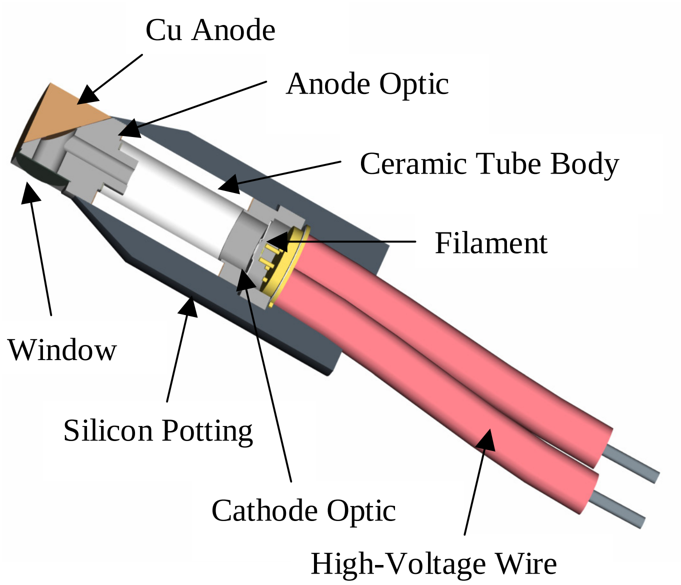

Figure 6. Schematic of the copper anode MOXTEK side-window tube.

The important features of the MOXTEK tube are that it is small in size and consumes very little power compared to conventional tubes. The tube is 42 mm and 15 mm in diameter (Fig. 7). The high-voltage power supply is 3x7x17 cm and requires a controller box and a 10 Volt DC input to operate. The maximum voltage is 20 kV with an emission current of 100 μA. The maximum input power required to run both the tube, the high-voltage power supply is five watts. For the data taken in the next section, the tube operated at 17.5 kV and 95 μA with a total power consumption of 4.4 watts. The MOXTEK tube is substantially smaller and consumes a great deal less power than the

RU-200 rotating anode tube. The rotating anode tube is rated for 60 kV, 200mA, and is housed on a large table that is 1.75x1.75x1 m. The MOXTEK tube is more compact than the Svetlana transmission-anode tube. The Svetlana tube is rated for 45 kV, 200 µA, and is 400 mm long and 40 mm in diameter.

Figure 7. Picture of MOXTEK low-power tube.

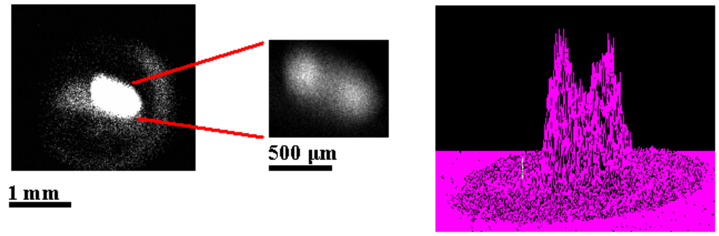

Figure 9. Image and 3D plot of the spot on the side-window tube.

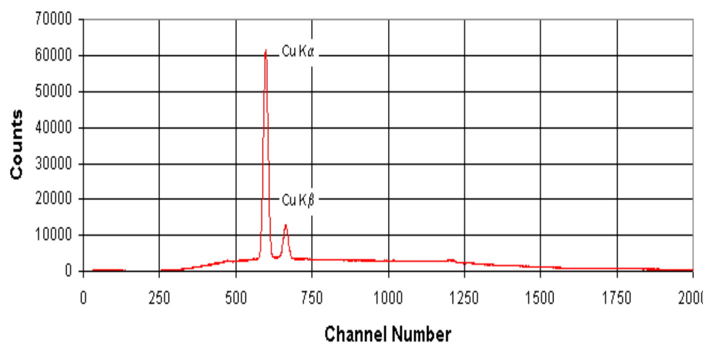

Figure 8. Emission spectrum of the low-power tube.

For X-ray diffraction the main considerations are the characteristic emission lines and the size of the beam spot on the anode. The low-power MOXTEK tube has a copper anode so the primary excitation radiation will be the copper Kα emission line. Figure 8 shows the emission spectrum measured with a Si(Li) detector with the tube operating at 20 kV and 4 µA. The copper Kα radiation consists of 27 % of the emission spectrum of the tube. The beam spot on the tube is approximately 500x750 µm (Fig. 9). Two bright lobes within the spot can be seen in the image. The three-dimensional intensity profile shows the intensity distribution across the anode. Each tube we investigated with the side-window design had a similar spot size and shape.

RESULTS

Table 1 contains data produced by the different X-ray sources employed. The optics control the diffraction angular resolution and the flux irradiating the sample. There is a trade-off between the flux and the angular resolution. If the flux on the sample is increased the angular resolution is decreased and vice versa. In some runs we wanted to get a higher flux while in others we wanted to improve angular resolution. On each run we used a KCl sample to eliminate any variations from the sample.

One key comparison within the table is between the Svetlana tube and the MOXTEK tube 72. The Svetlana tube was used before the MOXTEK tubes were built to test the feasibility of using a low-power X-ray source for XRD. In this comparison the optical setup and the tube emission current are exactly the same for both tubes. The differences are that the tubes have different spot sizes and that the Svetlana tube has a transmission anode while the MOXTEK tube has a reflection anode. The XRD/XRF instrument was able to collect at about four times the count rate with the MOXTEK tube over the Svetlana tube.

Source	RU-200	Svetlana tube	MOXTEK tube72	MOXTEK tube79
tube characteristics				
spot size	.1mmx1mm	300 μm	360x600 μm	500x750 μm
tube current	30 mA	50 μA	50 μA	95 μA
tube voltage	30 kV	30 kV	16 kV	17.5 kV
Optics				
pinhole size near source	300 μm	300 μm	300 μm	75 μm
pinhole size near sample	100 μm	200 μm	200 μm	50 μm
distance between pinholes	~9 cm	~6 cm	~6 cm	1.38 cm
divergence of beam	0.13°	0.24°	0.24°	0.25°
CCD angular resolution	~0.6°	~1.3°	~1.3°	~0.5°
Data count rate				
Cl K alpha (2.622 keV)	12.8 cps	1.5 cps	7.6 cps	4.4* cps
K K alpha (3.312 keV)	18.3 cps	2.1 cps	6.4 cps	3.4* cps
Cu K alpha (8.041 keV) XRD events	4.7 cps	0.33 cps	0.83 cps	0.98* cps

* Contains data from both the single and split events.

Table 1. A comparison of data collection rates of a KCl sample taken with different x-ray tubes and optical setups.

The second key comparison within the table is between the RU-200 tube and the MOXTEK tube 79. With the high-power tube we were able to get four times the count rate, but at a cost of more than 900 watts in just the emission current alone. With the MOXTEK tube the cost in total power input was 4.4 watts. The comparison is not direct in that a different optical setup was used with each tube, but it does show that with the low-power tube we are able to get data at a comparable rate. The data capture rate can be slightly increased with better optical setups and can be greatly increased with a smaller spot on the low-power X-ray tube anode.

CONCLUSION

The testing setup has been successful in proving the feasibility of a compact portable XRD/XRF instrument with the potential to be used in future planetary applications and in terrestrial applications. We were able to capture XRD and XRF data using the MOXTEK tube. The total power requirements for the instrument have been minimized and, thus, a battery can supply the power required to capture X-ray diffraction and X-ray fluorescence. Future work will include maximizing the data collection rate so that enough data can be collected to do quantitative analysis in a timely manner.

ACKNOWLEDGMENTS

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