MINIATURE CVD-DIAMOND CORING DRILLS FOR ROBOTIC SAMPLE COLLECTION AND ANALYSIS. D. T. Vaniman[1], V. J. Trava-Airoldi[2], D. L. Bish[1], and S. J. Chipera[1], [1]Hydrology, Geochemistry, and Geology, Los Alamos National Laboratory, MS D469, Los Alamos, NM 87545, USA and [2]Instituto Nacional de Pesquisas Espaciais, Av. Astronautas 1758, BR-12201970 Sao Jose Dos Campos, SP, Brazil vaniman@lanl.gov.

Introduction: Coring tools have been used effectively on the Moon, but to date no such tools have been used on any other extraterrestrial surface. The lunar experience includes both manual (Apollo) and robotic (Luna) systems. These coring systems were concerned primarily with acquiring samples from depth for return to Earth or for the creation of instrument emplacement holes (e.g., heat probes). Current designs for planetary drills differ from the lunar tools primarily in that they are integrated with robotic instrumentation for remote analysis, but the role of the drilling or coring system remains one of acquiring samples that must be extracted from the core barrel for analysis. Missing from current sample collection systems is a tool that can double as both a coring device and a sample holder. This dual utility can minimize the number of motions, the mass, and the power required for several classes of instruments in planetary surface exploration. To be effective, such a system must be durable and simple in operation. Hollow CVD diamond drills possess the hardness, excellent cutting properties, and heat resistance required for drilling into a wide variety of rocks and minerals. Because CVD diamond is also unreactive and transparent to infrared radiation and to X-rays of moderate to high energy, it can be used as a sample holder in various instruments for X-ray diffraction (XRD), X-ray fluorescence (XRF), infrared spectroscopy, Raman spectroscopy, and thermal analysis.

The specific application explored in this study is that of XRD. A small robotic instrument for combined XRD/XRF analysis [1] has been developed and has proved effective in the determination of mineralogy from XRD data on small (<1 mg) powdered samples of rocks and minerals. One of the obstacles to space applications of this instrument, however, is the need to acquire and insert rock powders robotically. Powdered samples are necessary to obtain accurate mineral identifications and quantitative mineral abundances by XRD. The low X-ray absorption coefficient of diamond permits transmission of X-rays with energies typically used in XRD (e.g., Cu Kα at 8.04 keV) with little attenuation. One of the standard sample mounting systems for powder XRD is based on glass capillaries in which small amounts of sample powder are held and rotated to present a large number of random crystal orientations to the X-ray beam. The capillaries used are made of silica glass with typical wall thicknesses of 10 µm and internal bores of 100-500 µm. A miniature, hollow CVD diamond drill can be as effective as a sample holder, exploiting the lower X-ray absorption coefficient of diamond to compensate for its thicker walls and taking advantage of the absence of amorphous X-ray scattering in crystalline diamond.

Hollow CVD diamond drills were produced by the Instituto Nacional de Pesquisas Espaciais (INPE), the national space research institute of Brazil [2]. These drills were produced by chemical-vapor deposition on Fe wires in a methane-plasma furnace. Two types of drills were produced, the first consisting of thick-wall (230 µm) CVD diamond grown over smooth wires of 300 µm diameter. After deposition around the wire forms, these drills could be simply pulled off of the wire for use. The second type was formed by growth of CVD diamond over 475-µm diameter Fe wires in which spiral grooves had been cut. This type of drill has a raised spiral along the inner wall that assists in drawing rock powder into the hollow drill as it turns. For this type of drill the Fe form is removed by acid dissolution. Wall thickness of the spiral drills was reduced to 80-150 µm to improve X-ray transmission.

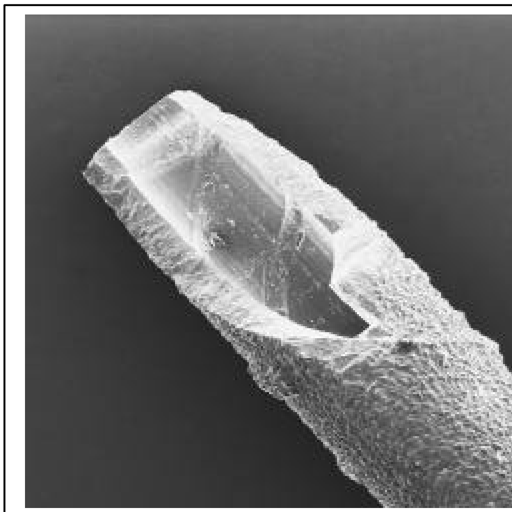

Figure 1: Secondary-electron image of a spiral CVD diamond core, broken open to show spiral grooves. Drill-core wall thickness is 150 µm.

Nucleation of CVD diamond on the surface of the wire produces a very smooth surface, in contrast to the exterior of the drill where crystal faces of the deposited diamond produce a rougher cutting surface (Figure 1). The relief on the spiral ridge around the inside of the drill barrel is ~10 µm; a bulge of comparable magnitude can be seen above the inner spiral and around the outer wall of the drill. The relationship between inner and outer spirals indicates that the grooves cut into the wire substrate support thicker diamond growth than the smooth wire surface between the grooves.

Drill bits were prepared by mounting the CVD drills in brass pins that could be inserted into a Dremel® rotary grinding tool. Drilling experiments were performed with single-crystal samples of calcite (Mohs 3) and quartz (Mohs 7). This range of hardness encompasses that of most rocks. X-ray diffraction experiments used an INEL diffractometer with Cu Kα radiation and a 120º 2? position-sensitive detector.

Results: The CVD diamond microdrills cut well and maintained structural integrity even when drilling through minerals as hard as quartz. However, there was a tendency for the drills to throw most of the powder created to the outside, leaving little inside the drill for analysis. This tendency is most pronounced in the smooth-bore drills with thick walls (230 µm). The thinner-wall (80-150 µm) drills with spiral inner ribs retained more powder within the drill barrel.

Drilling times of several minutes in calcite had little effect on the CVD drills. Drilling in quartz resulted in gradual abrasion of the drill tip, principally by plucking of individual diamond crystals. The thinner-walled drills are more fragile. Spiral fractures that follow the spiral ribs on the inner drill wall are problematic but can be avoided if pressure on the drill is kept light.

Diffraction experiments in which the CVD drills served as a sample mount were successful with both types of drills. The thinner wall of the spiral-rib drills provided greater transmission of diffracted X-rays. The diffraction pattern obtained from quartz powder in a thick-wall, smooth-bore drill (Figure 2) shows an excellent match in both position and relative intensities for the quartz peaks. All diffraction peaks above background represent signal from the sample (>9 peaks) or diamond (3 peaks). Although the diamond diffraction peaks are large, diffraction patterns for the quartz and calcite samples were obtained with considerable accuracy. The presence of only three diffraction peaks from diamond within the 2θ range plotted is a consequence of diamond's simple cubic structure. In practice these diffraction peaks will not have serious overlap with most geological samples and can be used to advantage as (1) calibration of the 2θ scale should the instrument drift from alignment and (2) an intensity reference for obtaining quantitative XRD data. The data obtained from the CVD diamond drills compare favorably with data obtained from silica capillaries, where a large amorphous diffraction signal centered at ~24º 2? makes background correction more difficult.

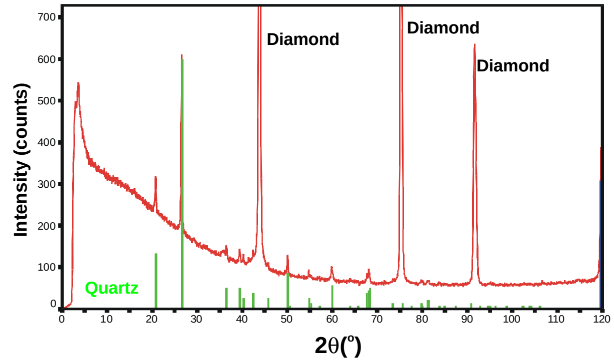

Figure 2: X-ray diffraction pattern of quartz powder in a CVD diamond core. Diamond reflections are marked, and a standard pattern for quartz (ICDD card #33-1161) is superimposed.

Thinner-walled spiral-ribbed drills were tried in an effort to increase the signal from the sample relative to that from diamond. To evaluate the effects of wall thickness, calcite was analyzed in CVD diamond core barrels of both 80 µm and 230 µm wall thickness. The difference in diffraction signal from calcite with these two wall thicknesses is quantified in Table 1 by comparing the measured peak areas of the calcite (104) peak and the diamond (111) peak.

Table 1: Comparative XRD performance of thick-wall and thin-wall hollow CVD diamond drills

Wall thickness of drill barrel	Calcite 104 peak area	Diamond 111 peak area	Calcite/ diamond peak ratio
230µm	36.9	210.5	0.175
80µm	37.4	209.0	0.180

The 65% decrease in drill-barrel wall thickness produced an insignificant increase in sample signal, reflecting the high mass absorption coefficient for Cu Kα X-rays for calcite (70.9) compared with diamond (4.6). Diamond walls with thicknesses that are practical for drilling will not significantly curtail Cu Kα X-ray transmission. However, thick diamond walls will have a very significant effect on transmission of X-rays with lower energy (e.g., Si) that will be produced as secondary fluorescent events from the sample and will need to be accounted for if XRF analysis is pursued.

References: [1] Vaniman D.T. et al. (1998) *JGR, 103*, 31,477-31,489; [2] Trava-Airoldi V. J. et al. (1998) *Surface and Coatings Tech., 108-109*, 437-441.