MÖSSBAUER SPECTROSCOPY ON MARS: EFFECTS OF SAMPLE STRUCTURE ON SPECTRAL INTERPRETATION; T.D. Shelfer1, R.V. Morris2. 1Mail Code C23, Lockheed Martin SMSS, 2400 NASA Rd. 1, Houston, TX, 77058, 2Mail Code SN3, NASA Johnson Space Center, Houston, TX, 77058.

Summary: Mössbauer spectroscopy is a powerful diagnostic tool for investigating iron mineralogy, and when applied in backscatter geometry, the technique is ideal for use in the field. The potential of Mössbauer spectroscopy has been recognized by mission planners and miniature backscatter Mössbauer instrumentation is scheduled for use on upcoming Mars lander payloads [1, 2]. The interpretation of backscatter Mössbauer spectra of rock samples is significantly different from standard laboratory transmission Mössbauer spectra in which randomly-oriented homogeneous powder samples are typically used. It is for this reason that we have investigated a number of iron-bearing rocks with a backscatter Mössbauer spectrometer (BaMS) developed at NASA/JSC. We previously reported the potential effects of weathering rinds on spectral interpretation [3]. In this work we will investigate the effects of crystal orientation and sample texture on Mössbauer spectra. This data will prove useful for interpreting spectra returned from the surface of Mars.

Introduction and Background: Based on the relatively high abundance of iron found in nature, its wide range of oxidation states (primarily 0, +2, and +3), and the variety of minerals in which it is found, the element iron is a particularly important source of information about geologic materials. In terms of investigating the oxidation state and mineralogy of iron in a material, there is perhaps no better investigative tool than iron Mössbauer spectroscopy (FeMS). FeMS is a nuclear resonance technique that provides detailed information about the local electromagnetic environment of individual $^{57}\text{Fe}$ nuclei in a sample. The resulting resonance pattern (Mössbauer spectrum) provides diagnostic information about the oxidation state of iron and its distribution among iron-bearing mineralogies, which tightly constrains the types of materials present. For remote use, backscatter geometry (Mössbauer source and detector(s) on same side of sample) is preferable over transmission geometry (Mössbauer source and detector on opposite sides of sample, commonly used in the laboratory) because no sample preparation is required.

The Mössbauer spectra used in this study were collected on a four-detector BaMS system developed at NASA/JSC. The BaMS system uses commercial-off-the-shelf (COTS) components integrated into a custom laboratory instrument. We used an Elscint model MVT-4 velocity transducer (mounted vertically) in conjunction with a model MDF-N-5 driver/generator and model MFG-N-5 function generator also manufactured by Elscint. The radiation detectors were mounted in backscatter geometry at a 45° angle with the incident Mössbauer radiation. We used four XR-100CR detector systems from AMPTEK Inc., with 13 mm² active area. The signals from each detector system were added using a precision unity-gain non-inverting summing amplifier built in-house. Data were collected with an Oxford Instruments Inc., PCAII multichannel analyzer card installed in a standard laboratory PC. Source intensity was ~45 mCi and the percent effect was typically 35-45% with ~8-30K counts in the baseline. A schematic illustration of the BaMS instrument used in this work is shown in Figure 1.

Enstatite, hematite, jarosite, and basalt samples used in this work were either small hand specimens placed directly in the instrument or similarly-sized pieces (~2x2x1 cm) of rock cut using a saw. For orientation studies rocks were cut in orthogonal directions.

Figure 1. Schematic illustration of the backscatter Mössbauer spectrometer (BaMS) used in this work.

Results and Discussion: Figure 2 shows BaMS spectra for enstatite, hematite and jarosite rocks. The differences in peak intensities in the upper and lower spectrum of each pair illustrate the effect of different crystalline orientations with respect to the incident Mössbauer radiation. The enstatite samples were cut from a small hand sample at orthogonal angles. Least-squares fitting procedures show that sample ENB2-R2 has a ratio of left to right peak areas of 1:3:1, where as sample ENB2-R1 has a ratio of 1:1:6. The theoretical ratio of areas assuming a thin random powder in transmission geometry for enstatite is 1:1.

The hematite samples were also cut from a rock in orthogonal directions. The theoretical ratio of the peak areas is 3:2:1:1:2:3. Sample HMRE2-R1 has a ratio of peak areas of 2.9:2.9:1.2:1:2.9:2.8, and sample HMRE2-R2 has a ratio of peak areas of 2.5:1.5:1:1:4:2.3.
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Figure 2. BaMS spectra of three rock specimens illustrating the effect of crystalline orientation.

The last set of orientation samples were two small jarosite rocks. LNVJAR1-R1 had a visible crystalline orientation and the sample was positioned such that a flat surface was exposed to the Mössbauer radiation. Sample GCJAR1-R1 was a jarosite rock with no discernable crystal orientation that was positioned in a similar fashion. The ratio of areas for LNVJAR1-R1 was 1:1.4 and for GCJAR1-R1 the ratio was 1.1:1. Again, the theoretical ratio of the two peaks is 1:1.

Figure 3 shows the results of BaMS spectra taken from a basalt sample with large (~0.5 cm) olivine phenocrysts. OB1-R1 was taken with a large olivine crystal centered in the Mössbauer beam, and OB1-R2 was in an area visibly free of olivine phenocrysts. Fitting results for sample OB1-R2 indicate an olivine to pyroxene ratio of 1.8:1. Sample OB1-R1 has an olivine to pyroxene ratio of 1:2. These significantly different spectra are strictly due to the placement of the BaMS instrument with respect to large mineral structures.

These results show that crystal orientation and textures approaching the size of the Mössbauer beam can significantly effect the peak intensities of the resulting spectrum. Careful analysis will be needed in order to take these effects in account when trying to determine the proportions of minerals that are in a rock. Least-squares fitting models using standard equal area or 3:2:1:1:2:3 area constraints can not necessarily be used, making the fitting procedure more difficult. The benefit of not using equal area or 3:2:1:1:2:3 constraints in the fitting models is that information concerning crystal orientation can be obtained based on peak area data. Additionally, if imaging or other data indicate texture features on rocks, then the Mössbauer instrument could be used to measure mineralogical differences between the features.