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# Future lunar mission Active X-ray Spectrometer development: Surface roughness and geometry studies



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#### ABSTRACT

The Active X-ray Spectrometer (AXS) is considered as one of the scientific payload candidates for a future Japanese mission, SELENE-2. The AXS consists of pyroelectric X-ray generators and a Silicon Drift Detector to conduct X-Ray Fluorescence spectroscopy (XRF) on the Moon to measure major elements: Mg, Al, Si, Ca, Ti, and Fe; minor elements: Na, K, P, S, Cr and Mn; and the trace element Ni depending on their concentration. Some factors such as roughness, grain size and porosity of sample, and the geometry of X-ray incidence, emission and energy will affect the XRF measurements precision. Basic studies on the XRF are required to develop the AXS. In this study, fused samples were used to make homogeneous samples free from the effect of grain size and porosity. Experimental and numerical studies on the XRF were conducted to evaluate the effects from incidence and emission angles and surface roughness. Angle geometry and surface roughness will be optimized for the design of the AXS on future missions from the results of the experiment and the numerical simulation.

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# 1. Introduction

Determining the distribution of elements in the lunar surface is essential for lunar science as it characterizes the geochemistry, and also supports the understanding of geology, of lunar surface materials. Great progress on lunar science has been achieved so far by recent observations made by lunar orbiters such as the Lunar Prospector [1], Clementine [2], SELENE (Kaguya) [3], Chang'E-1 [4] and -2 [5], and Chandrayaan-1 [6]. A landing mission will provide more detailed information on the local landing site area, improving the level of information on the Moon obtained by the previous lunar orbiters. In Japan, SELENE-2 is being developed as a follow-on mission of the first lunar orbiter of SELENE (Kaguya). SELENE-2 is a landing mission with a roving vehicle [7].

An Active X-ray Spectrometer (AXS) [8], one of the scientific payload candidates on the rover, is an in situ element composition analyzer for SELENE-2. It will be mounted on the arm head of the

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http://dx.doi.org/10.1016/j.nima.2015.03.083 0168-9002/© 2015 Elsevier B.V. All rights reserved. lunar rover of the SELENE-2 mission together with a Rock Abrasion Tool (RAT). The AXS will determine elemental concentration of various samples: various kinds of rocks and regolith samples at the landing site and along the path of the rover. The AXS measures lunar major elements: Mg, Al, Si, Ca, Ti, and Fe; minor elements: Na, K, P, S, Cr and Mn.

The AXS consists of pyroelectric X-ray generators and a Silicon Drift Detector (SDD) [8]. The AXS performs X-Ray Fluorescence spectroscopy (XRF) to determine elemental composition. The XRF is a well established laboratory technique. In general, an X-ray tube is used as an X-ray source for XRF in laboratory experiments. However, the X-ray tube requires high voltage supply significantly increasing the payload weight. In space missions, the XRF instrument should be light in weight and low in electric power consumption due to the severe restrictions on the payload resources. All the XRF instruments carried on previous lunar and Mars missions such as Chang'E-3 [9], Viking [10], Mars Pathfinder [11], MER [12], and MSL [13] used radioisotopes such as <sup>55</sup>Fe, <sup>109</sup>Cd, and <sup>244</sup>Cm to excite planetary surface material elements. Although the use of those radioisotopes has extraordinary advantage of light weight and no need of high voltage supply, it is not allowed at present for Japanese missions to

bring or send radioisotope sources. There are some alternatives to the radioisotope sources, and the X-ray generator using pyroelectric crystals is one of them [14–16].

Features of the pyroelectric X-ray generator are small size, light weight, low power consumption, no high voltage power supply, and X-ray emission only when needed. Therefore, the AXS is suitable for a Japanese roving mission on the Moon.

Samples for laboratory XRF measurements are finely ground to smooth surfaces, whereas planetary surface is contaminated by micro-meteoroids and other cosmic factors. The weathering effect makes analysis complex. In order to reduce the weathering effect, the uppermost sample surface will be removed by the RAT. Moreover, there are some factors such as sample surface roughness, grain size and porosity, geometry of X-ray incidence, and emission and energy, which might affect the precision of XRF measurements [17–20]. Therefore, we have to investigate the effect of those factors.

In our previous study [21], we used fused samples to make homogeneous samples and remove the effect from grain size and porosity of surface and considered the effect of roughness, incidence angle, and emission angle. Various surface roughness of material samples is produced by grinding samples with different hones. In this work, we performed a more detailed measurement related to the surface roughness below 2  $\mu$ m, and numerical calculations to simulate the experiments. The experiments in this work provide important information to design the configuration of X-ray generator, SDD and sample position in the AXS. The results obtained by the experiments and simulations reported in this work are related with the surface roughness of samples and incidence angle of X-ray.

# 2. Methods

# 2.1. Experimental setup

The experimental setup is described in detail in Ref. [21]. Here, we briefly describe the experimental apparatus and sample preparation.

In this study, the X-ray fluorescence analysis was performed in vacuum at a pressure of about 4 Pa. An X-ray tube with a silver target (Amptek Mini-X Silver (Ag)) was used as an X-ray source, and an SDD (Amptek XR-100SDD) with a detector size of 25 mm<sup>2</sup> × 500  $\mu$ m and a Be window of 8  $\mu$ m in thickness was used as an X-ray detector. Fig. 1 shows the energy spectrum of X-rays emitted from the Mini-X through a 1 mm $\emptyset$  hole Pb collimator



**Fig. 1.** Energy spectrum of the X-rays emitted from the Mini-X measured by the SDD. A sum peak of Ag  $L_{\alpha}$  (2.98 keV) and  $L_{\beta 1}$  (3.15 keV) around 3 kev, and high energy tail of bremsstrahlung were detected. X-ray counts are normalized at Ag  $L_{\alpha}$ .

measured by the SDD. The applied voltage and current to Mini-X were 20 kV and 100  $\mu$ A for all measurements, respectively. The X-ray beam was collimated with brass and acrylic collimators. The distance between the collimator and sample surface was 5 cm, and that between the sample surface and SDD was 2.2 cm. In this study, two kinds of angle geometry were investigated as shown in Fig. 2. In the geometry A, the emission angle  $\theta_e$  was fixed at 0°, thus the sample surface is parallel to the SDD window. In the geometry B, on the other hand, the incidence angle  $\theta_i = \theta_e$ . The incidence angle  $\theta_i$  was set at 15°, 30°, 45°, and 60° in each geometry.

The glass samples used in this study were made from the geochemical reference samples of JGb-1 (Gabbro) and JP-1 (Peridotite) [22,23] supplied by National Institute of Advanced Industrial Science and Technology, Japan. The powder of each reference sample was mixed with Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub> in the weight ratio of 1:2 and heated at 1200 °C to fuse, and then cooled down to glass. The glass samples have the size of 36 mm $\emptyset \times 3$  mm and density of 1.53 g/cm<sup>3</sup> (JGb-1) and 1.76 g/cm<sup>3</sup> (JP-1). This thickness is much larger than the attenuation length of Ag L<sub>α</sub>. The chemical composition of major elements in these samples is shown in Table 1. The K<sub>α</sub> X-ray energy of these elements is also listed. We selected the reference samples of JGb-1 and JP-1 in order to measure a variety of major elements. The measured elements were Al, Si, Ca, and Fe for JGb-1, and Mg, Si, and Fe for JP-1.

The sample surface was ground by hones to investigate the effect of sample surface roughnesses. We used seven kinds of hones with different mesh numbers, #100, #180, #400, #800, #1500, #3000, and #6000, to obtain different surface roughness. The mesh number is related to the grain size on hones, and the sample surface becomes smooth when using hone with large mesh number. Here, the surface roughness is evaluated by the arithmetic average roughness  $R_a$ , which is defined by

$$R_a = \frac{1}{l} \int_0^l |h(x)| \, dx \tag{1}$$

where *l* is the measured length, *x* the distance from the origin, and h(x) the deviation of surface height from the mean line at the



**Fig. 2.** Schematic drawing of the two angle geometry of experiments: (a) geometry A and (b) geometry B.

Table 1

The chemical composition of major elements in geochemical reference samples of JGb-1 and JP-1, and the  $K_{\alpha}$  X-ray energy of each element [22,23].

Elements	JGb-1 (at%)	JP-1 (at%)	$K_{\alpha}$ X-ray energy (keV)
Mg	4.15	21.6	1.25
Al	7.31	0.25	1.49
Si	15.5	13.8	1.74
Ca	4.52	0.19	3.69
Ti	0.43	-	4.51
Fe	4.02	2.05	6.40
0	59.7	55.9	
Others	4.34	6.19	

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