1. Introduction

KARLE (Potassium-Argon Laser Experiment) has been developed for use in situ planetary geochronology using the K-Ar (potassium-argon) isotope system [1,2], where material ablated by LIBS (Laser-Induced Breakdown Spectroscopy) is used to calculate isotope abundances. We are determining the accuracy and precision of volume measurements of these pits using stereo and laser microscopy data to better understand the ablation process for isotope abundance calculations. If a characteristic volume can be determined with sufficient accuracy and precision for specific rock types, KARLE will prove to be a useful instrument for future planetary rover missions.

2. Methodology

- 11 samples with 5 possible Martian analog compositions (basalt, jarosite, rhyolite, microcline, and tuff) were prepared by cutting an analysis surface and polishing to 1 μm.
- These compositions provide a range of hardness, heterogeneity, porosity, and grain size.
- We created a series of pits in each sample by firing a 1064nm Nd:YAG (neodymium-doped yttrium aluminum garnet) laser for 50 to 1350 shots per pit.
- Pit geometry and volumes were determined using a Keyence VK-X100 laser scanning microscope, utilizing both laser scanning and optical imaging techniques.
- Platinum tubes manufactured by Johnson Matthey Medical were used to test volume measurement error of the Keyence and operator, resulting in an average error of 5%.
- We conducted optical image analysis of several pits using the optical mode of the Keyence microscope and the Olympus SXZ16 stereomicroscope to understand how pit volume could be reconstructed using the z-stacking method.

3. Approach

- **KARLE Setup (Figure 2)**
  - LIBS uses high energy laser pulses to ablate a sample, creating a pit and producing a vapor cloud with excited atoms and ions that emit light at wavelengths specific to certain elements. This spectrum is used to estimate the elemental composition of the ablated sample.
  - We use LIBS to calculate the relative K abundance in wt% (weight percent).
  - The OMS (Quadropole Mass Spectrometer) measures absolute Ar abundance, in mols, and is dependent on the mass that is ablated.
  - To relate the OMS Ar measurement (mols) to the LIBS K measurement (wt%), we need to calculate the total mass of the ablated sample.
  - Some material may not ablate, so calculating accurate mass may be difficult.
  - Instead, we use average volume and density to back calculate mass.
  - Bulk mineralogy or elemental composition can be used to calculate density.
  - This study is aimed towards developing a method to accurately and precisely determine volume.

5. Discussion

A. Pit Volume

- Jarosite and rhyolite are heterogeneous and/or porous samples and display nonlinear volume increase. Linear volume increases are observed for basalt and microcline.
- Although some samples are heterogeneous (like some basalt), they still behave fairly linearly because their heterogeneity is on a similar scale as the laser pit.
- Slopes of best fit lines for basalt and microcline (Figure 2) are near those for jarosite and rhyolite and exhibit greater R² values, possibly suggesting similarities between materials.

B. Variations in Pit Morphology

- Assuming a symmetric pit may be adequate for homogeneous samples with a large number of LIBS shots per pit (1000 LIBS shots on Microcline 2, Figure 7) but cannot generate error for heterogeneous samples with less LIBS shots per pit.
- Figure 8 is data for the Basalt Sill 2 pit generated with 350 LIBS shots. The top image shows the pit was created along a grain boundary between plagioclase and pyroxenes/epidote.
- The lower images are topographic views (left is plan, right is a cross section) of the same pit showing the asymmetry in diameter with depth as a result of a change in mineralogy. This asymmetry is particularly apparent when compared with Figure 7.

C. Pit Reconstruction

- A best-fit function is a Gaussian when considering only pit depth and width, but underestimates pit volume and introduces a volume uncertainty of about 10%.
- Z-stacking
  - With the Keyence microscope, a total of seven layers over a depth of 1200 μm were stacked to reconstruct a pit (Figure 4) with a calculated volume of 8.345E+07 mm³.
  - Both calculated volumes agree within 20% of the reference volume (7.51E+07 mm³).

D. Compositional Effects

- Pits were generated with 200 LIBS shots on samples of jarosite (porphyritic), basalt sill (magnetohysall, crystals 1-4 mm), and a core of Fish Canyon Tuff (macrystalline, crystals less than 2 mm).
  - Larger volumes ablated per shot are observed for jarosite and decrease for Fish Canyon Tuff Core, with Basalt Sill 2 having the smallest.
  - Larger variations in volume ablation occur for Jarosite 1 and Fish Canyon Tuff Core while Basalt Sill 2 exhibits a smaller range.

Critical to the success of the KARLE experiment, or any LIBS-KAr geochronology investigation (e.g., 3-4), is the accurate measurement of the LIBS-ablated pit. This study shows that either a z-stacking or stereo imaging using available micro-imaging cameras are suitable methods for determining the volume of LIBS pits in flight designs (Figure 9). In a pinch, material properties (hardness, heterogeneity, porosity, and grain size) can be used to estimate the likely range of pit volume per shot and a functional fit using pit width and depth can estimate the pit volume within a larger uncertainty.

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8. References