

CRYSTAL SIZE DISTRIBUTION ANALYSIS OF APOLLO 15 MARE BASALTS: NEW METHODS AND SOME RECOMMENDATIONS. S. K. Bell¹, M. E. Hartley¹, K. H. Joy¹ and J. F. Pernet-Fisher¹, School of Earth and Environmental Sciences, University of Manchester, Oxford Road, Manchester, M19 3PL, UK (samantha.bell@manchester.ac.uk).

Introduction: Crystal size distribution (CSD) analysis is a non-destructive, quantitative method of understanding the crystallization history of a magma [1,2,3]; from which, signatures of magma mixing, accumulation, fractionation and multi-stage cooling histories can be identified and crystal residence times can be calculated [2]. As a result, CSD analysis enables examination of the magmatic and thermal history of planetary-scale processes.

Traditional CSD data collection is often time-intensive, requiring the manual tracing of crystals within a sample from a digital image e.g., [4,5]. QEMSCAN is an automated quantitative petrology system that combines scanning electron microscopy with energy-dispersive spectrometry to produce high-resolution mineral maps.

Here, we have used Apollo 15 mare basalt samples to investigate the feasibility of using QEMSCAN for semi-automated crystal size distribution analysis. This work is part of a wider project to understand the petrogenetic relationship between the Apollo 15 quartz-normative and olivine-normative suites [6]. We present plagioclase, pyroxene and olivine CSD data for five Apollo 15 mare basalt thin sections (15597,12, 15597,18, 15125,6, 15475,15 and 15555,209), gathered using both manual and QEMSCAN methods. To fully assess the potential of QEMSCAN analysis, samples were selected to incorporate as many potential CSD trends as possible and reflect the range of crystal shapes and sizes within the Apollo 15 mare basalt collection.

Manual data collection: Backscattered electron (BSE) maps were collected using an FEI QUANTA 650 field emission gun (FEG) scanning electron microscope (SEM) at the University of Manchester. Data were collected at a resolution of 0.98 μ /pixel. The crystal edges of the mineral(s) plagioclase, pyroxene and olivine were manually traced from the BSE maps in CorelDraw X6. Crystal shapes were then analysed using ImageJ version 1.51 [7] to attain properties such as major and minor axis length. The 2D crystal data were processed using *CSDslice* [8] to estimate true 3D crystal shape, before stereologically converting into 3D crystal lengths using *CSDcorrections* [4]. The natural logarithm of the crystal population density was then plotted against corrected crystal length to produce a CSD graph.

QEMSCAN data collection: The FEI QEMSCAN at the University of Manchester operates using a

QUANTA 650 FEG SEM, equipped with a single Bruker XFlash energy dispersive X-ray spectrometer (EDS). The QEMSCAN software, iExplorer, uses a combination of EDS spectra and BSE brightness to assign a mineral to each pixel using a Species Identification Protocol (SIP) list. The SIP list is a series of pre-defined mineral definitions that can be entered by the user. Samples were classified using a SIP list specifically especially set up for lunar samples. Mineral phase maps of each sample were acquired using step sizes (i.e. pixel sizes) of 5, 10 and 20 μ m to understand the effect of resolution on the final CSD analysis. All mineral phase maps were analyzed using processors found in iExplorer. The granulator processor was used to extract crystals of a specified mineral from the mineral map. The touching particles processor was used to separate touching crystals. When used in combination, these two processors allow crystals of a particular mineral to be separated and displayed. Crystals from each sample were then analyzed using ImageJ, *CSDslice* and *CSDcorrections* in the same way as the manually collected data (Fig.1).

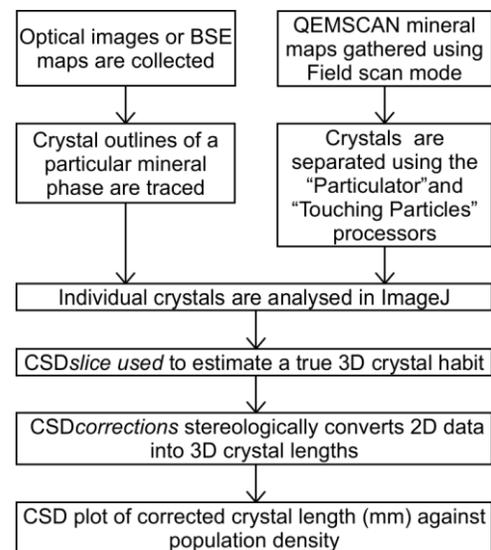


Figure 1. Flow diagram summarising the steps involved to produce CSD plots using manual and QEMSCAN methods.

Results: CSD plots of population density against crystal length were produced for pyroxene crystals in 15597,12, 15597,18, 15125,6 and 15555,209; plagioclase crystals in 15475,15; and olivine crystals in

15555,209. In all samples except 15555,209, we observe differences between the CSD trends produced from manual and QEMSCAN-derived data (Fig.2).

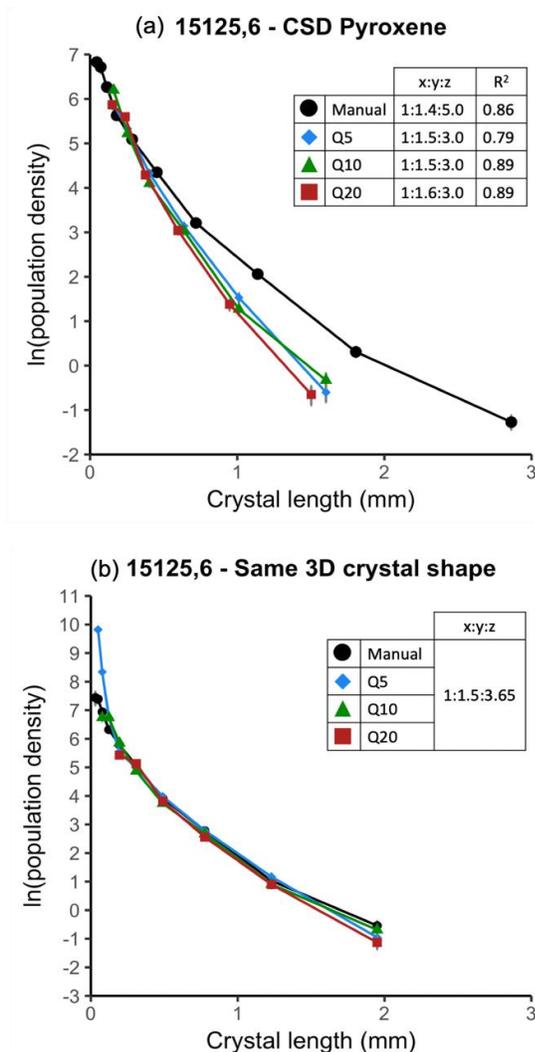


Figure 2. CSD plots for sample 15125,6. (a) CSD plots where Q5, Q10 and Q20 correspond to QEMSCAN step sizes of 5, 10 and 20 μm , respectively. The short-, intermediate- and long-axis ratios (x, y, z) and R² value for the 3D crystal shape estimates are also shown for each plot. (b) Reprocessing of the data sets in (a) using the same 3D crystal shape.

In most cases, the manual data result in CSD trends with shallower gradients that extend to larger crystal lengths than the QEMSCAN CSD data. The 3D crystal shape estimates from CSDslice are near-identical for QEMSCAN data collected at different step sizes; however, the 3D crystal shape estimates for manually derived CSD data often have larger long-axis values compared to QEMSCAN data. Discrepancies in the

CSD plots between manual and QEMSCAN-derived data can be resolved by reprocessing all data sets with the same 3D crystal shape estimate (Fig. 2a). The 3D crystal shape estimate, in this case, was calculated by taking a weighted average (based on R² values) of 3D crystal shape estimate from the QEMSCAN data at 5, 10 and 20 μm step-sizes. However, whilst such processing masks an underlying segmentation issue that most strongly affects the longest crystals in the sample. We find that the iExplorer touching particles processor often fails to identify and separate elongate crystals that are in contact with other crystals. This inefficient segmentation causes long crystals to be split into multiple smaller-length crystals, and therefore artificially reduces the long (z) axis portion of the 3D crystal shape estimate calculated in CSDslice. The underestimation of the number of long crystals, combined with the underestimation of crystal aspect ratio, results in QEMSCAN-derived CSD plots with artificially steepened gradients that extend to shorter crystal lengths (Fig. 2a). Not only does this effect the ability to recognize signatures of certain magmatic processes, but calculations of crystal residence times can also differ by up to a factor of ~8. In the case of 15555,209, the two methods produced CSD plots which fell within error of each other at step-sizes of 5 and 10 μm . This is due to 15555,209 consisting of coarser, rounded crystals touching only in a few places, which are more easily separated by the touching particles processor.

Conclusion: Differences between data collected by manual and QEMSCAN methods were seen in four out of five of the Apollo 15 mare basalt samples in this study. Such differences have the potential to influence petrologically important signatures of crystal accumulation and/or fractionation and calculations of crystal residence times. The texture and crystal habit of the sample plays an important role as to how well the touching particles processor performs, affecting the overall reliability of the QEMSCAN data. QEMSCAN should only be used for CSD analysis after careful consideration of the suitability of the samples texture and average crystal habit.

References: [1] Cashman, K.V. and Marsh, B.D. (1988) *Contributions to Mineralogy and Petrology*, 99(3), 292-305. [2] Marsh, B.D. (1988) *Contrib Mineral Petrol*, 99, 277-291. [3] Marsh, B.D. (1998) *Journal of Petrology*, 39, 553-599. [4] Higgins, M.D. (2000) *American Mineralogist*, 85, 1105-1116. [5] Neal, C.R. et al. (2015) *Geochimica et Cosmochimica Acta*, 148, 62-80. [6] Chappell, B.W. and Green, D.H. (1973) *EPSL*, 18, 237-246. [7] Abramoff, M.D. et al. (2004) *Biophotonics International*, 11, 36-42. [8] Morgan D. J. and Jerram, D.A. (2006) *Journal of Volcanology and Geothermal Research*, 154, 1-7.