

ELECTRON MICROSCOPY OF APOLLO 12 GLASS SPHERULES. J. Levine¹, R. A. Muller^{1,2}, and P. R. Renne^{3,4}, ¹Department of Physics, University of California, Berkeley, California 94720, USA (jlevine@socrates.berkeley.edu), ²Lawrence Berkeley National Laboratory, Berkeley, California 94720, USA, ³Berkeley Geochronology Center, 2455 Ridge Road, Berkeley, California 94709, USA, ⁴Department of Earth and Planetary Science, University of California, Berkeley, California 94720, USA.

Introduction: We are continuing our investigation of the meteoroid bombardment history of the Moon, recorded in the formation ages of impact-generated glass spherules in the lunar regolith. We earlier reported [1] the ages, measured by the ⁴⁰Ar/³⁹Ar isochron technique, of 155 spherules from sample 14163, returned by the Apollo 14 mission to the Fra Mauro formation. From the distribution of ages, we identified an increase in the rate of spherule production by a factor of 3.7 ± 1.2 in the last 400 Myr. We presently seek to determine whether this indicates an increase in the meteoroid bombardment of the Moon, or whether it is the result of a local change at the Apollo 14 landing site (e.g. [2]). As in [1], we chose spherules from a regolith sample mixed by a recent impact (sample 12023,151 comes from the rim of Sharp Crater in the Procellarum Basin, visited by the Apollo 12 mission), whose ages we therefore believe are representative of the impact history of the Moon. Details of our sample selection are in [3].

Improving our technique from [1], we have thoroughly documented each spherule in advance of the radioisotopic dating experiment. We used the scanning electron microscope to image our 179 spherules, each $>180 \mu\text{m}$ in diameter. Elemental analyses were made on 2-5 spots that appeared representative of most of the surface of each spherule and on ≤ 5 small grains welded to the surface. Here we report the results of this electron microscopy, favoring an impact origin for nearly every spherule.

Analysis: We made measurements using a JEOL 6340F field-emission scanning electron microscope at the National Center for Electron Microscopy. Secondary electron images were taken of each spherule, and elemental analyses were made by energy-dispersive x-ray spectroscopy using an EDAX x-ray detector and EDAX Genesis software. The specimen was held 12 kV above the electron gun so that beam-specimen interactions could excite transitions as energetic as the 6.4 keV K_{α} x-ray of iron, while keeping the excited volume of the specimen as shallow as possible. Indeed, absorption within the specimen of x-rays as soft as the 1.3 keV K_{α} x-ray of magnesium was inconsequential.

To prevent electrostatic charging of the specimens and resulting diminution of the beam energy, the spherules were mounted on electrically conductive carbon pads that were in turn mounted on brass microscope stubs. The prepared stubs were coated with a thin layer of silver using a Denton Desk II sputter-coater. The sputtering was performed in a ~ 120 mtorr atmosphere of 99.998% pure nitrogen. Alongside the spherules, we silver-coated shards of multiply-degassed and homogenized basaltic glass. These “zero-age glass” samples enable us to determine if any argon was adsorbed to the spherules during sputtering which would compromise our ability to measure ⁴⁰Ar/³⁹Ar ages. We also used the “zero-age glass” samples as standards for elemental analysis, determining the reproducibility of our analyses from 38 measurements on different glass fragments (Table 1).

Abundances in “zero-age glass” (wt. %)	
SiO ₂	53.63 ± 1.27
TiO ₂	1.78 ± 0.39
Al ₂ O ₃	17.02 ± 1.01
FeO	8.91 ± 1.05
MnO	0.51 ± 0.32
MgO	7.36 ± 0.96
CaO	7.57 ± 0.67
Na ₂ O	2.56 ± 1.06
K ₂ O	0.66 ± 0.28

Table 1: Composition of the basaltic “zero-age glass” determined by analyses on 38 shards. The uncertainties (root-mean-square deviations of the measurements) approximate the uncertainties in our analyses of the lunar glasses.

Sample Genesis: Most lunar glass spherules form by impact melting [4], but some are due to volcanic fire-fountaining events [5]. Delano and Livi [6] argue that impact glasses may be (but need not be) chemically heterogeneous, but that volcanic glasses must be homogeneous. From the analyses we made on spots texturally representative of the surface of each spherule (rather than on surface grains), we find that 68 of the spherules are heterogeneous in Mg, Al, Si, Ca, or Fe, with spot-to-spot variations greater than we would expect from replicate analyses.

Because impacts can also create homogeneous spherules, this number underestimates the fraction of our spherules produced this way.

On 13 of our spherules, we detected small surface grains (typically $<5\ \mu\text{m}$ across) that are very rich in iron, have smaller amounts of nickel, and occasionally contain sulfur, phosphorus, or chromium. The grains typically have Fe/Si weight ratios ~ 20 . We suggest that such iron-rich surface grains indicate an impact origin for a host spherule, with material from an iron-rich impactor becoming welded to the spherule surface in the aftermath of an impact. Not all impact spherules would have iron-rich grains because some might be created in impacts of stony meteoroids. Furthermore, because of the small number of spot analyses we performed on each spherule, we expect that additional spherules have iron-rich grains that we did not detect.

Schonfeld and Bielefeld [7] correlate volcanic dark mantle deposits with high Mg/Al ratios in glass spherules, and Delano and Livi [6] also use Mg/Al ratios to distinguish impact spherules from volcanic ones. In [6], they found that only impact glasses have Mg/Al weight ratios <1.5 . Averaging together all the compositionally representative analyses for each spherule (i.e. those not taken on surface grains), we find that 176 of our spherules have mean Mg/Al weight ratios <1.5 , indicating that the vast majority of our spherules are impact-generated. Though 3 spherules were found with Mg/Al ratios >1.5 , approximately 1/60 of the impact glasses studied in [6] also had Mg/Al ratios above this level, so it is possible that even these 3 spherules formed in impacts. (The 3 spherules with Mg/Al >1.5 are all chemically homogeneous and on none have we detected iron-rich grains.)

Other observations: In our electron microscope images, we see that some of our spherules are completely glassy, some are wholly crystalline, and others are partly so (Figures 1,2). It has been suggested (e.g. by [8]) that $^{40}\text{Ar}/^{39}\text{Ar}$ ages of glass spherules might be corrupted by incomplete degassing of argon during the brief period in which the spherule is melted. Such “inherited argon” would introduce bias toward older ages, but our results in [1] are surprising for the abundance of young ages. In the present experiment, we will test whether $^{40}\text{Ar}/^{39}\text{Ar}$ ages of crystalline spherules are systematically different from those of glass spherules.

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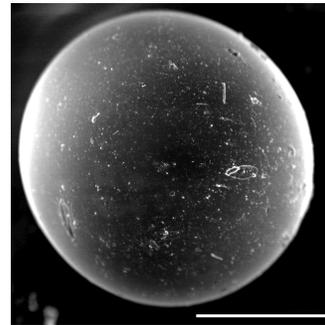


Figure 1: Secondary electron image of a glassy spherule. Note the smooth texture over most of the surface, punctuated by small welded grains. Scale bar is $100\ \mu\text{m}$.

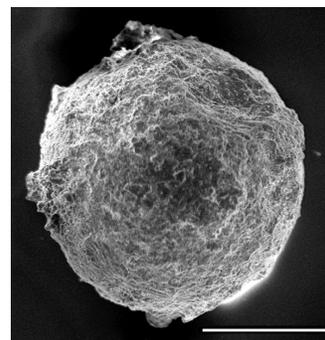


Figure 2: Secondary electron image of a crystalline spherule. Scale bar is $100\ \mu\text{m}$. Though crystalline, this grain must once have been melted to pull itself into a sphere.

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