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### Incipient melt segregation in subaqueous pyroclasts

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### Analytical Conditions and Selected Results

#### *X-ray Computed Microtomography ( $\mu$ -cT)*

The X-ray computed microtomography ( $\mu$ -cT) imaging was performed with a Phoenix Nanotom 180, using a tungsten filament and molybdenum target. Operating voltages were in the range 90 to 120 keV, with currents of 50-90 nA. In order to obtain the highest resolution scans possible, lapilli were broken into small pieces, ~1 mm in diameter. A total of 2300 scans were collected during 360° rotation, with voxel edge length of ~1.5  $\mu$ m. Raw scans were reconstructed into stacks of greyscale images with an offline PC microcluster running Phoenix reconstruction software. Manual segmentation of intra-vesicular extrusions and adjacent connected void spaces was performed with the Blob3D software (Ketcham, 2005), allowing their respective volumes ( $V_{IVE}$  and  $V_{void}$ ) to be measured directly. Other extrusion geometric parameters, including length ( $L$ ) and diameter ( $d$ ) of flow pathways were measured on greyscale images using the ImageJ software (Table DR1).

Table DR1. Measured IVE parameters

no.	$V_{IVE}$ ( $\mu\text{m}^3$ )	$V_{void}$ ( $\mu\text{m}^3$ )	$L$ ( $\mu\text{m}$ )	$d$ ( $\mu\text{m}$ )
1	2.4E+05	6.4E+04	43	31
2	1.6E+05	1.8E+05	61	25
3	3.5E+05	2.6E+05	67	26
4	6.4E+04	7.1E+04	99	25
5	6.2E+04	1.3E+05	34	27
6	5.9E+04	3.2E+04	49	14
7	1.7E+05	1.1E+05	55	28
8	2.8E+05	2.9E+05	62	24
9	3.2E+05	2.6E+05	49	63
10	8.9E+05	7.3E+05	98	67
11	4.2E+05	2.6E+05	107	70
12	1.8E+05	1.5E+05	70	30
13	3.8E+05	2.9E+05	71	48
14	3.6E+05	4.3E+05	64	61
15	3.2E+05	1.9E+05	79	30
16	9.2E+04	3.2E+04	41	22
17	9.9E+05	3.1E+05	146	46

All measurements made using the Blob3D (Ketcham, 2005) and Image J software packages.  $V_{IVE}$  is volume of individual intra-vesicular extrusions (IVE), excluding their internal void spaces.  $V_{void}$  is volume of connected void space in groundmass adjacent to each IVE,  $L$  and  $d$  are the length and diameters, respectively, of the connected void spaces.

#### *Fourier Transform Infrared (FTIR) Spectroscopic Imaging*

Images of the distribution of total  $\text{H}_2\text{O}$  in areas of the samples were obtained by Fourier-transform infrared (FTIR) spectroscopic imaging using a Varian FTS Stingray 700 Micro Image Analyser spectrometer with an attached UMA 600 microscope fitted with a Varian Inc. Lancer Focal Plane Array (FPA) camera, at IFREE, JAMSTEC. Preparation of chips of sample for analysis involved first impregnating them in low-viscosity epoxy to preserve delicate features, and then grinding them down into free-standing wafers using silica carbide paper, progressing from a grade of 600 to 1200, and polishing both sides using 1  $\mu\text{m}$  diamond paste. The FPA camera consists of a liquid-nitrogen cooled  $\text{HgCdTe}_2$  (MCT) array detector that can collect an image  $350 \times 350 \mu\text{m}$  across 4096 channels (arranged  $64 \times 64$ ) giving a channel, or spectral, resolution of 5.5  $\mu\text{m}$ . A heated ceramic (globalar) infrared source and a Ge-coated KBr beamsplitter were used to collect the spectroscopic images over the mid-IR range of  $6000 - 400 \text{ cm}^{-1}$  at a resolution of  $8 \text{ cm}^{-1}$ . Images were averaged from 512 scans. The free-standing wafer, polished on both sides, was placed on an IR-transparent KBr window and the UMA 600 microscope was used to select areas to image. Initially a background image on a clean area of the KBr window was collected, which was subtracted from the image of the sample resting on the KBr window. Images were treated using Varian's Win-IR Pro software (v3.3.1.014). The images presented here are originally of the absorbance (height) of the vibration band at around  $3500 \text{ cm}^{-1}$ , caused by the fundamental OH-stretching vibration, above a linear background between  $3800$  and  $2520 \text{ cm}^{-1}$ . Measured absorbances were converted to  $\text{H}_2\text{O}$  concentration using the Beer-Lambert law (Stolper, 1982), with molar absorptivity of  $63 \pm 3 \text{ mol}^{-1} \text{ cm}$  (Dixon et al., 1995) and glass density of  $2.9 \text{ g.cm}^{-3}$  (Lange and Carmichael, 1990). Sample thickness, or beam pathlength,

is an important parameter in the Beer-Lambert law. During collection of transmission FTIR spectra, reflectance spectra were also collected at each point, with interference fringes on the reflectance spectra used to calculate thickness as described by Nichols and Wysoczanski (2007). For the total H<sub>2</sub>O image presented in Figure 2, the average thickness, 53±5 µm, across the sample was used to calibrate the colour scalebar.

### *Electron Microprobe (EMP) Element Mapping*

X-ray distribution maps of Na-ka, Al-ka, K-ka, Ca-ka and Fe-ka were obtained simultaneously by WDS-EPMA on a JEOL JXA-8500F electron probe microanalyzer. Operating conditions were 15 keV acceleration potential, 25 nA probe current, focused beam (~50 nm), 0.2-0.3 micron step size, and a dwell time of 50 ms per pixel. Quantitative point analyses on far-field sideromelane, within intra-vesicular extrusions, and on tiny pockets of residual melt in interstices within tachylite (Table DR2), were used to calibrate the colour scalebars.

Table DE2. Point EMP analyses

SAMPLE	Sideromelane			IVE			Residual Melt		
	SID_1	SID_2	SID_3	IVE_1	IVE_2	IVE_3	Res_1	Res_2	Res_3
SiO <sub>2</sub>	50.1	50.2	49.9	49.9	50.1	50.1	54.0	54.3	55.3
TiO <sub>2</sub>	2.89	2.90	2.87	2.91	2.94	2.85	3.57	3.74	3.34
Al <sub>2</sub> O <sub>3</sub>	12.2	12.4	12.2	12.2	12.4	12.2	16.8	17.0	17.8
FeO	12.3	12.4	12.4	12.4	12.5	12.2	10.7	10.1	9.24
MnO	0.19	0.18	0.17	0.17	0.18	0.17	0.16	0.15	0.15
MgO	8.56	8.49	8.42	8.37	8.38	8.30	1.97	1.87	2.27
CaO	11.7	11.6	11.7	11.5	11.5	11.4	9.51	9.68	7.81
Na <sub>2</sub> O	2.27	2.35	2.32	2.19	2.09	1.96	2.63	2.60	2.74
K <sub>2</sub> O	0.77	0.72	0.77	0.73	0.72	0.68	0.90	0.95	1.17
P <sub>2</sub> O <sub>5</sub>	0.25	0.26	0.30	0.28	0.25	0.22	0.28	0.31	0.29
TOTAL	101.2	101.5	101.1	100.6	101.1	100.1	100.5	100.7	100.1

## References

- Ketcham, R.A., 2005, Computational methods for quantitative analysis of three-dimensional features in geological specimens: *Geosphere*, v. 1, p. 32-41.
- Dixon, J.E., Stolper, E.M., and Holloway, J.R., 1995, An experimental study of water and carbon dioxide solubilities in mid-ocean ridge basaltic liquids. Part I Calibration and solubility models: *Journal of Petrology*, v. 30, p. 1607-1631.
- Lange, R.L., and Carmichael, I.S.E., 1990, Thermodynamic properties of silicate liquids with emphasis on density, thermal expansion and compressibility: *Reviews in Mineralogy and Geochemistry*, v. 24, p. 25-64.
- Nichols, A.R.L., and Wysoczanski, R.J., 2007, Using micro-FTIR spectroscopy to measure volatile contents in small and unexposed inclusions hosted in olivine crystals: *Chemical Geology*, v. 242, p. 371-384. doi: 10.1016/j.chemgeo.2007.04.007
- Stolper, E.M., 1982, Water in silicate glasses: an infrared spectroscopic study: *Contributions to Mineralogy and Petrology*, v. 81, p. 1-17.