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An image-based technique to determine the freezing temperature T_f of vesicle volumes in decompressed, synthetic melt samples

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The non-in-situ analysis of H_2O degassing of silicate melt at high temperature and pressure is conducted using synthetic, decompressed melt samples quenched to glass. Interpretations regarding the degassing behavior are based on the number of H_2O filled vesicles and the porosity of the vitrified samples. These properties of the glass samples may not represent the vesiculation at experiment temperature T_{exp} and target pressure P_{final}. Even at high quench rates q, a decrease of vesicle volumes during cooling occurs, facilitated by resorption of H_2O fluid back into the melt (McIntosh et al., 2014) and by the decrease of molar volume of H_2O (Marxer et al., 2015) in the vesicles. This vesicle shrinkage introduces uncertainty regarding the true q-dependent "freezing" temperature T_f, at which shrinkage stops, represented by the vesiculated glass sample. While often neglected, knowledge of T_f is useful for improved sample interpretation.

McIntosh et al. (2015) developed a computer tomography (CT) based method to determine T_f. This approach infers T_f from the volume fraction of liquid H_2O in vesicles (whose volumes are comprised of a liquid and a gaseous H_2O phase) which decreases for increasing T_f.

Using their theoretical foundations, we developed a simple, transmitted light microscopy (TLM) image-based approach for the determination of this intra-vesicle phase ratio, applying two different model calculations: 1) Approximation of phase boundaries using polynomial functions. 2) Calculation of total vesicle and gas-phase volumes from ellipsoid axes measurements, approximating the vesicle and gas-phase volumes with symmetrical spheroids. In our analyzed hydrous, haplogranitic samples, we found mean T_f's up to ~250 to ~300 K lower than T_{exp}, at which quench was initiated, for q's of ~40 and ~90 K/s. These values are close to the estimated T_f's obtained using an independent glass porosity equation (Gardner et al., 1999). The large scatter of volume fractions and thus T_f for individual vesicles cannot be attributed to our image-based approach as data obtained from phonolitic samples using the CT method (Allabar et al., 2020) depict a similar scatter. At present, no correlation of T_f with vesicle size or position within the sample could be made. The method is, for the range of vesicle sizes investigated (20 to 50 µm in diameter), limited to liquid volume fractions larger than ~10 vol% as a distinction between phases is limited by optical resolution.

Nevertheless, our TLM based approach provides a simple, readily available method to constrain T_f of vitrified vesiculated samples which significantly improves the quality and comparability of

derived interpretations. Our method uses standard polished sections for FTIR, making it even applicable to already existing samples.

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