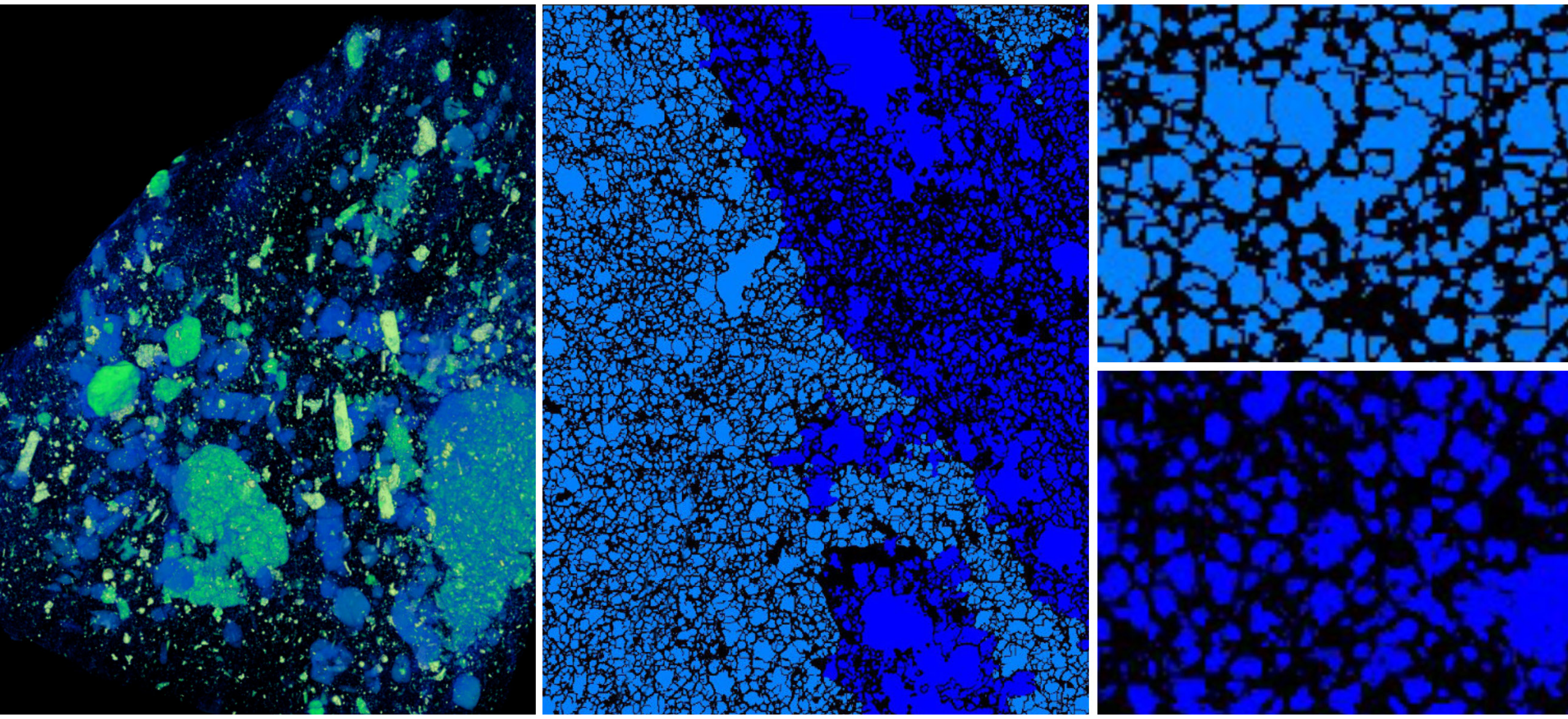
Application Note

Bursting Bubbles: Investigating Magma Ascent Dynamics

Correlative Microscopy



Seeing beyond

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Understanding geological processes is often a multi-scale problem within heterogeneous materials. Petrological studies are often correlative, linking field observations to hand samples, along with textural and geochemical information from whole rock scale and micro-to-nano scale analysis. Correlative microscopy workflows form an integral part of these studies: not just linking multi-modal and multi-scale information, but also guiding our research to create the most efficient and effective workflows to describe our sample.

A volcanic eruption is a prime example of a multi-scale process that poses a hazard to both local and global communities. The key to effective hazard assessment in these regions is understanding what happens within the magma as it ascends and erupts - one of the grand challenges of volcanology. During volcanic eruptions, ascending magmas ex-solve gases, form crystals and are heterogeneously stressed. The resultant mixture of bubbles, melt and crystals impacts and is impacted by, the rheology of the melt. Ultimately, micro-scale interactions between these phases control the ability of an ascending magma to outgas, determining the explosivity of the resultant eruption.

The sample described in these workflows is a basalt-rhyolite mingled fall from Ascension Island, South Atlantic. The rhyolite magma was intruded by a hot basaltic melt, triggering rapid ascent and eruption (Chamberlain et al., 2020). We investigate micro-scale changes in vesicle texture and composition in 2D and 3D to explore how and why bubble-crystal-melt interactions evolved differently for eruptions with different initial conditions.

Sample Selection and Methodology

Clasts were selected based on their representative nature of the field observations, and key textural characteristics.

Sampled clasts were all between 16 and 32 mm diameter, as these are least likely to have experienced post-eruption textural maturation (Shea et al., 2012) and are more easily imaged using X-ray computed micro-tomography.

For non-destructive 3D analysis clasts were scanned using ZEISS 620 Versa X-ray microscope (XRM) at the Carl Zeiss X-ray Microscopy facility in Pleasanton, CA. Backscattered electron (BSE) images were obtained for each sample on ZEISS Sigma 300 VP at the ZEISS facility in Cambridge, UK. In order to combine the textural and geochemical analysis, a workflow was devised (Figure 1) that began with non-destructive 3D XRM analysis used to both classify samples and guide the selection of regions for thin sections. These 2D thin sections could be used for ground truth 3D observations, generate additional information and provide polished surfaces for *in situ* microanalysis by secondary ion mass spectrometry (SIMS) and electron probe microanalysis (EPMA).

X-ray Microscopy (XRM) Study

In order to identify key locations within the sample, two scans were performed, starting with an initial “scout” of the entire clast with a voxel size of ~27 µm. Key regions of interest within the clast were then scanned with a “zoom” at ~1 µm voxel size. This two tiered Scout-and-Zoom approach using a lower, then higher resolution scan of the sample allows for maximum workflow efficiency while combining full sample context with key sample information. The low-resolution scan was examined to identify large-scale textural and compositional variations and allowed identification of “problem” regions within clasts that would be unsuitable for high resolution imaging, e.g., regions shielded by large crystals that may generate additional noise in the high resolution data. High resolution scans were collected from analytically suitable, texturally representative regions, while also making sure to capture any features of interest, e.g., boundaries between basalt and rhyolite compositions in mingled clasts. High and low-resolution volumes were correlated using image processing software (Avizo) to allow comparison of features captured by each scan type. We also examined the orientation of any finer-scale fabrics revealed in the high-resolution scans, e.g., elongation of vesicles.

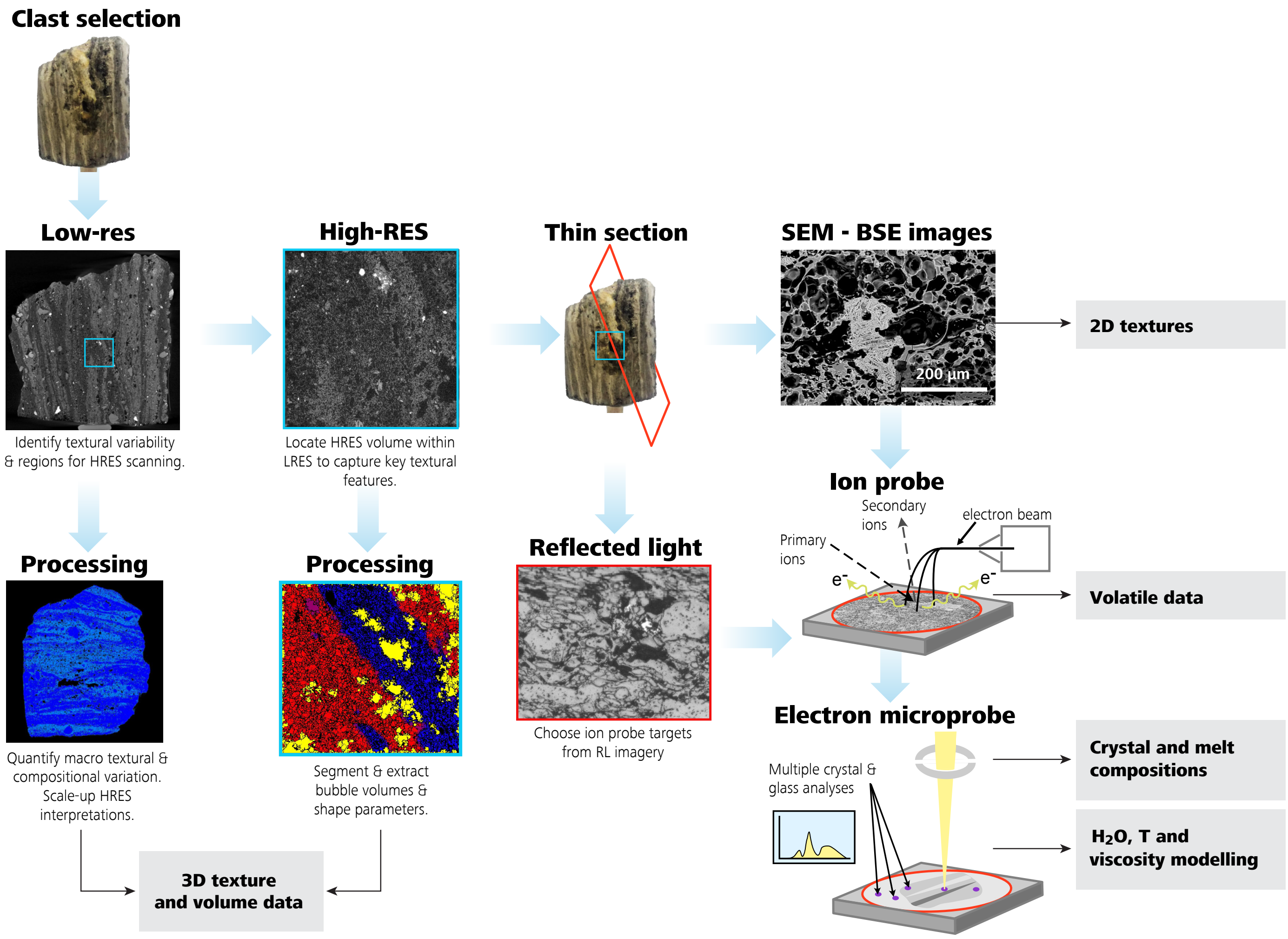

Figure 1 Full correlative sample workflow for the project. Initial XRM scans highlight key areas for higher resolution imaging and target locations for thin section orientation within the volume. Subsequent 2D analysis includes electron and light microscopy, leading to correlation with in situ *microanalytical data.*

Scout-and-Zoom greyscale volumes were then processed to extract a range of textural information. High density objects such as large crystals or plutonic clast inclusions are easily extracted by simple thresholding techniques and their 3D volume can be rendered to show their locations in each sample. A critical feature to observe are glass walls of the bubbles, as these are used to define their size and shape characteristics. Scoria (basalt) clasts have thicker glass walls and fewer thin melt films and so fewer corrections for unresolved melt films are required. Pumice clasts, however, typically have a more heterogeneous bubble population and abundant thin melt films, and so noise in the data must be more carefully evaluated to reconstruct broken or unresolved films, ensuring the bubble population is correctly represented (Figure 2). A combination of manual and algorithm-based segmentation were applied in an iterative process to achieve the best results. Once bubble volumes have been appropriately defined, volume,

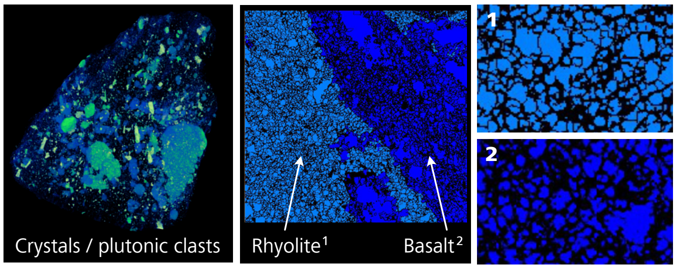

Figure 2 Careful segmentation of 3D datasets allow for comparison of textures in regions of different composition. Statistically relevant datasets can be obtained by extracting bubble geometry in 3 dimensions without damaging the sample.

shape, connectivity, and orientation data can be extracted for hundreds of thousands of bubbles at a time, making this technique ideal for collecting statistically robust textural data in 3D. This technique is non-destructive and generates a 3D record of textural and, to an extent, compositional heterogeneity within clasts.

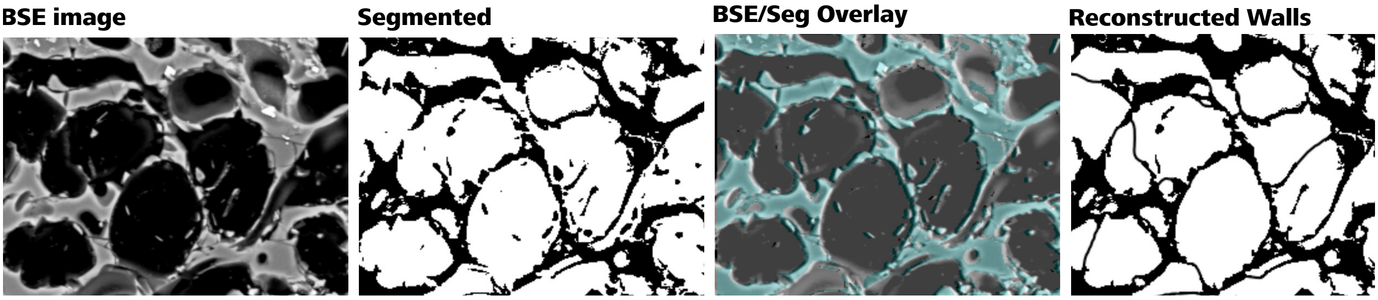

Figure 3 A combination of electron and light microscopy means that the most delicate structures can be manually identified, improving the process of segmentation and the resulting analysis.

Thin Section SEM Imaging and Analysis

In order to examine crystal textures and carry out the *in situ* geochemical analysis of volatile, major and trace element concentrations, 2D thin sections must be produced. It can be difficult to ensure a sectioned plane is representative of the internal structure of the clast. By starting with the 3D study (Figure 1), we were able to use the 3D scans of each clast to ensure our sections did the following:

1. Sampled a texturally representative region
2. Intersected some larger crystals for future analysis
3. Plane of section was suitably oriented relative to internal fabrics (depending on desired outcome)
4. Allow comparison between 3D and 2D textural observations and analyses

This approach allowed greater control over which textures were sampled and later analyzed in thin section. It also allows for later testing and comparison of 2D vs 3D textural quantification techniques as the intersected region in 2D should closely match that imaged in 3D.

Samples were imaged at a range of scales with images collected to capture regions of specific textural interest, e.g., phenocryst textures and compositional boundaries. Whole-section maps were produced onto which higher magnification, correlated image locations were registered, key to ensuring a good spread of image collection and for later reference.

These 2D images were segmented to extract bubbles from glass and crystals. Particularly thin films are often visible in BSE imagery, but some may have been broken during sample prep, or poorly captured by the segmentation process (Figure 3). Here the user can reconstruct films manually using the BSE image as a reference. Once fully segmented and with films reconstructed, the user can extract measurements such as bubble perimeters, long and short axes of the best fit ellipse, orientation, and area. These values can then be used to calculate textural descriptors such as regularity, circularity and roundness, and construct vesicle size distributions (VSDs) using 2D – 3D stereological conversion (Sahagian and Proussevitch, 1998).

Further geochemical analysis

The 3D and 2D textural analysis of bubble populations provides key information on the rheology of the magma during ascent. The correlative approach beginning with XRM provides a textural platform upon which additional geochemical measurements can be made and directly liked to the textural information. Several techniques can be applied to the 2D thin sections to obtain key geochemical information including energy dispersive spectroscopy (EDS), which can rapidly ascertain mineral and glass compositions, to act as reference for more detailed analyses by EPMA and SIMS.

Secondary Ion Mass Spectrometry (SIMS)

Reflected light (RL) imagery of each section was collected and suitable locations for SIMS analyses identified and mapped. Reflected light imagery was used as this is the view seen when using the ion probe. Comparisons of BSE and RL imagery allowed suitable regions, e.g., microlite free and across a range of textural properties, to be more easily identified. Cross-correlating BSE images to RL images meant SIMS analysis sites could be accurately located in the sample. Matrix glass SIMS analyses for H_2O, CO_2, Cl and F were then carried out using the Cameca IMS 4f ion Probe at the NERC Ion Micro-Probe facility in

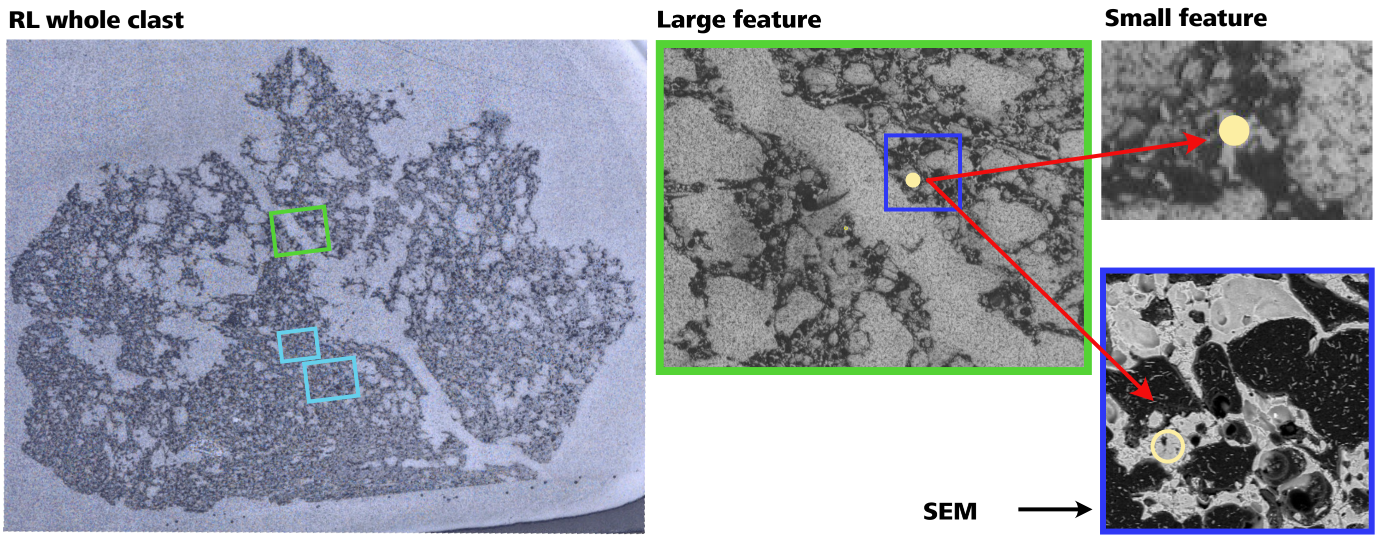

Figure 4 Correlation of XRM and EM data with reflected light images of polished samples allows for easy identification of targets for microanalysis in systems (e.g., large sector SIMS) that use reflected light cameras for targeting

Edinburgh.

Electron Microprobe Analysis (EMPA)

Using the BSE and RL imagery as a guide, high precision major and trace element analyses of matrix glasses and crystals can now be applied to each sample in regions of differing texture. This allows us to trace compositional changes not only during each eruption, but also identify whether key textural features are associated with micro-scale compositional variations. Compositional analyses also provide a basis for reconstructions of temperature and pre-eruptive volatile contents using Fe-Ti thermometry and crystal-melt hygrometers (Ghiorso & Evans, 2008; Mollo et al., 2015). These analyses also identify which crystal populations are in equilibrium with the host melt. This is important for considerations of pre- vs. syn-eruptive crystallization. Constraining these parameters is key as they strongly influence the viscosity and outgassing of the magma.

Outcomes

This example of the Ascension Island mingled fall to show how the described techniques provide key insights into factors controlling ascent dynamics and eruptive behavior. Differences in the processing of XRM data required for basaltic regions vs rhyolitic regions within the mingled clasts highlighted the contrasting behavior of these melts. While this difference was unsurprising, it was not as pronounced as would be expected when comparing the scoriaceous and pumaceous end-member clasts from this deposit. VSDs for the two regions are likely to be only subtly different, with implications for equilibrium vs non-equilibrium degassing, supersaturation and permeability during the dominantly mingled phase of the eruption. VSDs from the early pumaceous phase and late scoriaceous phase will reflect distinctly different gas-melt-crystal interactions.

Summary

Petrological studies in the geosciences often involve a wide range of techniques that must be correlated to provide meaningful insights. Here we see how non-destructive 3D analysis doesn't just provide textural insight, but can be used to guide the next stage of the project, where 2D thin sections must be made for detailed observations and *in situ* microanalysis. This correlated, multimodal, multi-scale approach provides the most efficient workflows for geoscience research. Importantly it also allows those workflows to be adaptable, with observations of each stage of the process guiding exactly how the workflow progresses, creating the most complete and relevant datasets.

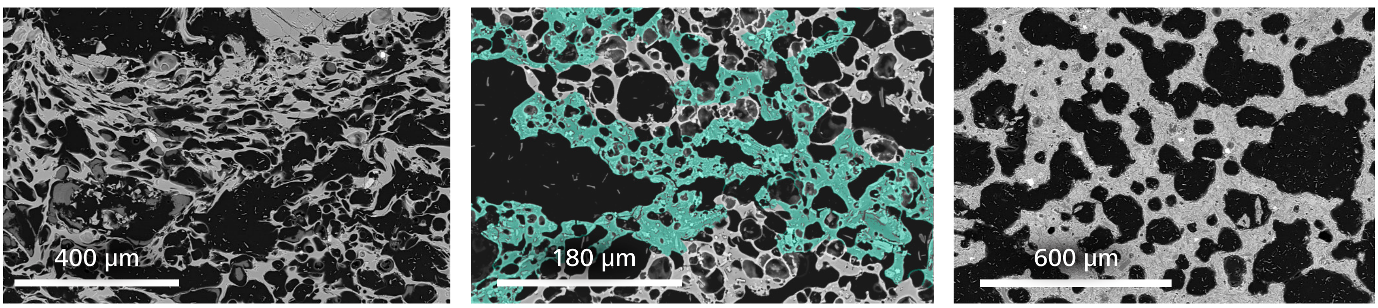

Figure 5 Contrasting textures of both the bubble size and density, as well as bubble wall (glass) thickness is readily highlighted by the correlative XRM-EM-LM workflow. Allowing for large accurate datasets to be used to interrogate rheological factors in eruption dynamics.

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