**3D imaging of volcanic ash using the confocal microscope; a comparison of natural fragments and experimentally vesiculated volcanic glass.**

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**Abstract**

Identifying the microstructural characteristics of volcanic ash particles is key in developing our understanding of their production, transport and impact. Volcanic glass in ash clouds can damage aircraft engines, surfaces and coatings, and if inhaled cause harm to humans and animals on which they depend. We present results of 3D imaging of volcanic glass particles from two Icelandic volcanoes together with two samples of experimentally-vesiculated volcanic glass. True colour 3D images were obtained using an Olympus LEXT 3D laser scanning confocal microscope. The images enabled examination of bubble structure, fracture patterns, shape morphology characteristics of grains.  For the two simulated fragments the bubbles had a median long axis lengths of 13.2 and 37.0 µm. For the material from Grimsvötn and Eyjafjallajökul volcanic eruptions the median long axis length was 25.2µm and 23.1 µm respectively. Laser confocal microscopy-based 3D imaging of volcanic glass fragments provides a rapid, non-destructive way to assess 3D particle geometry and quantify internal artefacts responsible for their formation.

Key Words: volcanic ash, glass, bubbles, 3D imaging

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**Introduction**

Volcanic glass can give insights into magma flow behaviour, eruption characteristics, deposition at the surface and cooling (Wadsworth and Tuffen 2016; Wertheim et al. 2017). Where the volcanic glass obsidian is concerned, it is often relatively slowly extruded from a volcano vent as a lava flow, but at the same time it can produce large amounts of volcanic shards due to eruptive events (Tuffen et al. 2013; Wadsworth and Tuffen 2016). All obsidian forms from such explosive events according to the Castro-conjecture (Castro et al. 2014) with the loss of volatiles. These events in fact represent cycles of 'explosive pulverization' where material reforms as a dense glass. The escape of volatiles / excess gases carries with it volcanic ash and glass fragments.

Magmas when they are at the surface have cooled, lost water and may have formed crystals so are often very viscous. Therefore, they may show viscoelastic glass transition properties behaving like glass, cyclically flowing and fracturing into shards.

Predicting eruptions with such magmas and explosive events is particularly difficult, and poses an issue for planners, governments and civil aviation authorities (Harman 2010; Perkins 2011; Alexander 2013).

Carazzo et al. (2014) examined the behaviour of ash clouds and associated volcanoes, the latter producing explosive eruptions of dense mixtures of hot gases and pyroclasts at the vent, which can then rise to a height of several tens of kilometres into the atmosphere if conditions are right (Wilson, 1976; Carazzo et al. 2014; Girault et al. 2014). As a result of turbulent entrainment and the thermal expansion of cold air, the bulk density of the mixture rising can be reduced. The column can then be lifted until it reaches level neutral buoyancy, spreading laterally due to the influence of high-altitude winds when the density of the mixture becomes lower than the density of the atmosphere due to natural convection (Carazzo et al. 2014). The impact of such events on climate and on air traffic can be substantial and is reasonably well documented (Miller and Casedevall 2000), but the details depend strongly on the concentration and distribution of grain sizes in the ash clouds (Girault et al. 2014).

The impact of ash clouds on every-day life can mean minor local issues, to regional to hemispheric or global impacts on climate systems (Robock 2000). Such events can pose a threat to ground infrastructure (Baxter et al. 2005) or pose a minor to major threat to humans more directly (Hornwell 2007; Pennec et al. 2012). The ash itself consists of rock and glass shards that are sharp-edged, hard, abrasive and mildly corrosive which may clump together to form aggregates. Issues surrounding ash clouds for aircraft are associated not only with their highly abrasive nature, but also the sintering / melting process that occurs when particles enter jet engines and stick to surfaces (Swanson and Beget 1991; Grindle and Burcham 2003). There has therefore been some considerable effort utilising techniques such as satellite remote sensing to try to quantify and understand the behaviour and nature of volcanic eruptions that result in explosive injections of ash into the atmosphere. This has been for the most part due to the recent eruptions in 2010 of Eyjafjallajökull and Grimsvötn in 2011. The Eyjafjallajökull eruption cost the global airline industry £130 million per day (International Air Transport Association estimate) and disrupted the travel plans of 8.5 million passengers (Alexander 2013).

The UN’s International Civil Aviation Organisation standing instructions at the time (ICAO 2007 Manual on Volcanic Ash and associated Volcanic Ash Contingency Plan (EUR Region)) was to avoid all contact with ash clouds and the UK Civil Aviation Authority adopted this no risk approach. The Grimsvötn eruption by comparison was responsible for a much-reduced number of flight cancellations than Eyjafjallajökul, but still significant in terms of impact of cancellations. This was partly due to improved understanding of ash clouds but also partly due to different dispersion conditions, although the volume of material ejected was greater. Grimsvötn is the most frequently active volcano in Iceland after Hekla. It lies beneath Europe’s largest glacier Vatnajökull, in the eastern part of the island and erupted 21$^{st}$ – 25$^{th}$ May 2011. The eruption plume rapidly rose to 20 km at the start of the eruption and was the most powerful in Iceland for over 50 years.

Jordan et al. (2014) highlight that the unique morphologies and shapes of volcanic ash particles are directly related to the fracture mechanisms operating at the time of eruption and that these relationships can be used to identify the type of fragmentation that has taken place. Bubbles then affect crack propagation and grain fragmentation as noted by van Otterloo (2015). Hence the significance of studying bubble shape and formation to understand the morphology of ejected volcanic ash and glass particles.

**Man-made volcanic glass.**

The eruption of ash particles at the Earth's surface represents a final stage in the evolution of magma as it ascends through the crust. Decompression during ascent leads to the nucleation and growth of bubbles as volatiles such as $H_2O$ and $CO_2$ exsolve from the melt, creating increased buoyancy that further drives magma towards the surface while simultaneously increasing melt viscosity as the remaining dissolved $H_2O$ content decreases (Sparks 1978). The rate and dynamics of this bubble growth exert a key control on the explosivity of the eruption – and thus the associated hazard. By creating man-made glasses of volcanic composition under the high pressure and temperature conditions relevant to the volcanic conduit, it is possible to replicate key processes of volcanic eruptions such as bubble growth (Castro et al. 2012; McIntosh et al. 2014) and fragmentation (Romano et al. 1996; Okumura et al. 2010). By comparing the physical characteristics (e.g. size and shape of bubbles) of these glasses created under controlled conditions with those of naturally erupted glasses it becomes possible to interpret the eruptive processes that lead to the formation of ash and other pyroclasts.

**3D Imaging Techniques**

There are several other methods of 3D imaging of rock textures and associated materials that have been employed in recent years (Jerram and Higgins, 2007). Techniques such as serial sectioning, grinding and lapping as well as x-ray tomography together with advanced image analysis could be used to create virtual 3D models of rock textures (Mock and Jerram 2005, Jerram and Higgins 2007, Pankhurst et al. 2014). With serial sectioning, material is lost. With serial grinding and lapping resolution of the order of 40 microns is possible (Marschallinger 1998).

Methods such as those based on the attenuation of beams that penetrate the sample or use a series of parallel sections such as serial sectioning (Bryon et al. 1995) or confocal scanning laser microscopy can be utilised with computer reconstruction. Stereological methods can take 2D sections and extrapolate the data into 3D textures based on certain grain shape assumptions (Morgan and Jerram, 2006).

A major advantage with our scanning laser confocal technique is that it is non-destructive and requires very little preparation for analysis and allows colour with 3D imaging: this material is then still available for other techniques such as geochemical analysis. A scanning laser technique was used by Petford et al. (2001) to illustrate porosity and grains in a North Sea sandstone. With x-ray tomography there may be difficulty in assessing touching crystals of the same material (Jerram and Higgins, 2007) and a superimposed true colour image is unlikely to be available. We are able to image small particles even of size about 2 µm using our confocal microscopy methods. We have used this technique to measure and illustrate alpha tracks in CR39 based solid state nuclear track detectors (Wertheim et al., 2010), typically, such tracks are 20 µm across and 20 µm deep.

**Aims.**

The aims of this study were to investigate the structure of volcanic ash and glass particles using 3D confocal microscopy imaging and to use the techniques for 3D imaging of bubbles in thick sections of natural and artificial material.

**Methodology.**

Volcanic particles.

Two sets of samples from the Eyjafjallajökull 14th April – 20th May 2010 eruptions and one set from the 21st-25th May 2011 Grimsvötn event were examined.

Artifical volcanic glass.

Two samples of artificially produced rhyolitic volcanic glass were examined. These were quenched at higher temperatures than the natural ash particles and were not under conditions of strain or quench fragmentation.

Strew Slides

Three sets of samples were obtained and assessed from Eyjafjallajökull (2010) where eruptions contained basaltic, intermediate and silicic material and the basaltic Grimsvötn (2011) eruptions (Gudmundsson et al. 2012; Thorkelsson 2012). The samples were sieved which was important to do so because of the working distances of the lenses used and washed with propan-2-ol in an ultrasonic bath to image clean material. Using a strew slide approach particles were distributed on double sided tape onto glass microscope slides using a similar technique to that previously described (Wertheim et al. 2017; Miyashita et al. 2021).

Thick Sections

Icelandic volcanic ash material was mounted (unfiltered) in resin attached to a glass slide and cut and polished to a 50 μm thick section to facilitate external and internal bubble imaging. Section thickness was chosen in order to image variation in bubble shape. Ash was placed in a polypropylene mould measuring 25mm internal diameter and to a depth of 3-4mm. A small volume of Epo-Tek 301 epoxy resin, only enough to wet the sample and to allow air bubbles to escape was added. This was to ensure that the ash sample is prevented from floating thus obtaining an even distribution of particles throughout the mix. The section was allowed to cure and the sample lapped (that is, two surfaces are rubbed together with an abrasive between them) with 600 grit silicon carbide self-adhesive paper, the surface being ground to expose the grains. Subsequent lapping was carried out with 9 μm aluminium oxide on a rotating metal lap to flatten the surface. The sample was then cleaned in an ultrasonic bath. After bonding to a pre-lapped glass slide, excess material was removed with a CS-1 cut-off saw to leave a 500 μm thick block. This was finally lapped with a Logitech LP50 to a 50-55 μm thickness and polished on a Struers Abramin Polishing machine using 6, 3 and 1 μm diamonds. A similar technique was used to mount the chips of artificial volcanic (McIntosh et al. 2014).

Artificial Samples

The artificial volcanic glass chips were embedded in epoxy resin (Specifix-20) and ground down to expose a continuous flat surface with exposed vesicles. Final polishing was done using 1 μm diamond paste.

Imaging

The unmodified particles in the strew slides and the thick section material were imaged on an Olympus LEXT 4100 (Olympus Corporation, Japan) 3D measurement scanning laser confocal microscope in a similar manner to that described by Wertheim et al. (2009, 2010) and Gillmore et al. (2017). 30 bubbles were imaged in fine mode setting using the LEXT microscope's x50 or x100 lenses. The numerical aperture of the lenses is 0.95. The procedure we followed for the strew slides was to focus just below the level of the mounting tape with the upper level being set just above the maximum detected height on laser imaging. Linear measurements were made using the propriety software for the LEXT microscope. Where the bubbles reached the cut surface we were able to image the bubble form.

**Results.**

Strew Slides

After mounting on tape we obtained images of the shape and texture of ash grains from the volcanic eruptions (Figure 1). Confocal 3D laser scanning microscopy was used to image bubble formation, using intensity images, colour height data and combinations of height, intensity and optical data. As can be seen from Figure 1, intensity, height and 3D combined image data, shows the morphology of fragments of various sizes. The smaller fragments were often angular, elongate and bladed forms (Krumbein and Sloss 1965; Folk 1965; Boggs 2001). Larger ash particles again demonstrated sharp angles, with the overall form being angular to sub-angular (Figure 1). Some fragments were closer to a spheroid in shape but all showed shapes that were consistent in their form with the existence of broken bubbles or vesicles.

Thick sections

The rock thick section method allowed grain edges well to be imaged (Figure 2; Figure 3a) and illustrated the curving and angular form of these grains as well as observation of Internal bubble structures (Figure 2; 3a-b). The bubbles varied in size and shape (Table 1) with a median of short versus long axes for the Eyjafjallajökull eruption of 23.1 and 12.9 µm with depth of 7.8 µm as well as median of 21.0 and 25.2 µm with a depth of 5.7 µm for Grimsvötn. In some cases, the bubbles appear to be aligned along the long axes (Figure 3a).

Simulated Volcanic Bubbles

Figure 4 demonstrates an example of similarity between bubbles in natural volcanic glass with the artificial glass. Bubble size varied but were similar compared to the natural glass depending on which sample was being viewed. It should be noted that data generated depends on the nature of the intersection between the cut of the polished thick section surface and the bubbles. Details about the range for each sample are given in Tables 1-3. For the data in Table 2 the median of short versus long axes was 11.1 x 13.2 µm with a depth of 2.6 µm and for Table 3 the median of short versus long axes was 31.0 x 37.0 µm with a depth of 20.8 µm.

Figure 5a for artificial volcanic glass indicates a linear relation between length and width of bubbles. A similar relationship can be observed for natural volcanic glass in the Grimsvötn sample examined (Figure 5b).

## Discussion

Icelandic cryptotephra (or microtephra / glass shards) deposited in NW Europe have lognormal Particle Size Distributions (PSDs) of 20 -70 µm (length) (Stevenson et al., 2015). An ash cloud can transport particles less than 80 µm diameter as far as 850km in 24h. Even moderately sized Icelandic eruptions can deposit cryptotephra on mainland Europe. Newnham et al. (1999) highlight that the hazard represented by distal rhyolitic and andesitic volcanic sources have been significantly underestimated in the past whilst Durand and Grattan (2001) noted that there is increasing evidence that near-surface volcanic air pollution may occur 1400 km downwind of an eruption. Direct sampling of ash clouds and satellite remote sensing have also indicated finer material in clouds than that deposited (satellite data suggests that 95% of the grains in clouds are < 17 μm diameter). Remote sensing observations cannot give the necessary detailed information on PSD (particle concentration, size spectrum, composition) that are essential for assessing such a hazard for aircraft (Zehner 2010). Typically such glass shards have highly irregular shapes and are often vesicular or bubbly in texture. Their size is described by their long axis which is typically between 20 to more than 125 μm. These are similar to the long axes measurements we have observed in material and will be the largest in a depositing cloud but may have formed a significant portion of the cloud closer to the volcano. Typically, such glass shards have highly irregular shapes and are often vesicular or bubbly in texture (Lautze et al 2012). In the 2010 Eyjafjallajökull and 2011 Grimsvötn eruptions grains that were up to 110 and 80 μm long respectively were deposited 800-1500 km from the source in the UK. in UK respectively. Studies suggested that the finer cryptotephra are mostly greater than 20 μm long with most less than 60 μm long (see Stevenson et al. 2015). There is a large discrepancy between the size of volcanic ash particles recorded on the ground at least 500km from their original source and those measured via techniques such as satellite remote sensing in the ash cloud (Stevenson et al. 2015).

Since the size and distribution of bubbles in magma is a key control on the size and shape of resulting particle fragments (van Otterloo 2015), we compared the bubble characteristics of the naturally erupted ash particles with those of the artificial glasses created under pressure and temperature conditions relevant to magma ascending in the volcanic conduit. The fragmentation of particles is one of the primary reasons why we have elected to examine the particles from these two eruptions in detail and to compare these fragments with man-made volcanic glass.

The artificial glass bears some resemblance to natural materials. The two slides examined varied in terms of the character of their bubbles with one containing bubbles that were very rounded / spherical in form, and generally smaller than the natural materials examined. However, the second sample contained vesicles / bubbles that were more elongated – not as elongated as the natural materials as a general rule, but also often larger than the natural glass. 3D confocal microscope imaging of bubbles provided similar morphologic data for both artificial and natural volcanic glass. There was some difference on the median bubble dimensions of the samples with the natural volcanic material having dimensions in a corresponding range.

Mock and Jerram (2005) reconstructed and modelled shapes and sizes based on extrapolating between measurement points, which also involved calculating the true 3D long axis of crystals and using 3D sphere models whereas our technique allows direct measurement of shapes and sizes. We plan to develop this work further by analysing a greater number of samples to increase the population of 3D data for both natural and artificial samples.

**Health impacts**

In terms of health impacts of volcanic ash, Horwell (2007) has characterised Grain Size Distributions in terms of health-relevant size fractions. Horwell (2007) found a strong correlation between the amount of 4-10 μm material for all ash types they examined with eruption style and distance from the source. They also point out that the dominant phase in volcanic ash is silicate glass (20-80%). Ash is of health concern because it contains respirable crystalline silica (a Class 1 human carcinogen responsible for cases of silicosis and lung cancer), plus the presence of surface transition metals such as $Fe2^{+}$ can generate hydroxyl radicals which damage DNA, and bio-toxic elements and compounds such as Hg, Cd and As (Horwell, 2007). The Grimsvötn ash was classified by Olsson et al. (2013) as glassy tholeiitic basalt with less than 10 mass% crystalline plagioclase and pyroxene. Olsson et al. (2013) also stated that the particles were small (less than 125 μm) and elongated with sharp edges, with around 50% of the particle mass being fine ash (less than 63 microns), which, unless curtailed by aggregation (Wallace et al. 2013), could travel long distances, with around 8% being less than 10 μm.

The bubbles can break up creating small fragments, the shape and size of bubbles and fragments therefore being closely related. Health issues arise from inhalation of respirable fragments due to their chemical characteristics, size and morphology (Rose and Durrant 2009; Damby et al. 2013). Smaller fragments, $PM_{10}$ and below, are of particular interest in connection with human health as for example they are thought to be able to penetrate the lungs (Wertheim et al. 2017, Miyashita et al. 2021).

**Conclusions**

This study has shown that laser confocal microscope-based 3D analysis of volcanic ash particles can be used effectively in imaging the morphology of volcanic glass particles. This research has also clearly demonstrated that there is a similarity between simulated volcanic glass, albeit quenched at higher pressures than natural glass, and the bubbles contained therein and imaged at the surface, from samples of naturally occurring volcanic glass ejected. Lastly, the images gained from examining both the artificial glass and natural ash, together with finer fragmented particles, show how bubble formation can control the morphology and size of particles fine enough to be transported by ash clouds.  This work may help in developing models to better understand the formation and break up of bubbles in glass, and hence assess the impact of such particles on aircraft and human health.

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Figures and Captions

**Figure 1**. Example of views of strew slide from Eyjafjallajökull sample. 256x256x47µm (x50 objective). The grains are on the surface of double-sided tape. Images x50 objective.

**Figure 2**. Examples of volcanic ash (Eyjafjallajökull) thick view section showing grain edges, some internal crystal forms and bubbles. Top view and view from underneath the top surface. 128x128x26µm (x100 objective).

**Figure 3a.** Images of bubbles in Grimsvötn volcanic ash grains from a thick section. Deepest structures are blue and violet and highest structures are orange / red. X100 objective Image size 86 µm by 86 µm. X50 objective and 3X zoom.

**Figure 3b.** Note bubbles imaged at the surface of a thick section (Grimsvötn), the 3D view of a cross sectional line and the large size of the bubbles. Some bubbles are seen in the optical image but not in the 3D image because they do not emanate to the surface. X100 objective. Image size 129 µm by 129 µm.

**Figure 4.** Example images of bubbles in artificial glass. The gold colour within the bubbles in the colour image is a result of residual coating from SEM microscopy. Colour image, intensity and height data together with a zoomed 3D image. X100 Objective. Image size 129 µm x 129 µm.

**Figure 5a.** Scatterplot of Length versus width of bubbles / vesicles in two artificial volcanic glass samples. Blue circles sample 1 (MC1 B11), red squares sample 2 (MCN13), showing an approximately linear relationship.

**Figure 5b.** Graph showing Length versus width of vesicles in Grimsvötn and Eyjafjallajökull natural volcanic glass samples.