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## High Resolution 3D Confocal Microscope Imaging of Volcanic Ash Particles

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### Abstract

We present initial results from a novel high resolution confocal microscopy study of the 3D surface structure of volcanic ash particles from two recent explosive basaltic eruptions, Eyjafjallajökull (2010) and Grimsvötn (2011), in Iceland. The majority of particles imaged are less than 100 µm in size and include PM<sub>10S</sub>, known to be harmful to humans if inhaled. Previous studies have mainly used 2D microscopy to examine volcanic particles. The aim of this study was to test the potential of 3D laser scanning confocal microscopy as a reliable analysis tool for these materials and if so to what degree high resolution surface and volume data could be obtained that would further aid in their classification. First results obtained using an Olympus LEXT scanning confocal microscope with a x50 and x100 objective lens are highly encouraging. They reveal a range of discrete particle types characterised by sharp or concave edges consistent with explosive formation and sudden rupture of magma. Initial surface area/volume ratios are given that may prove useful in subsequent modelling of damage to aircraft engines and human tissue where inhalation has occurred.

### Introduction

Volcanic ash clouds pose significant hazard to human activities. The 2010 VEI (Volcano Explosivity Index) 4 eruption of Eyjafjallajökull, Iceland caused significant disruption to air travel over Europe and the North Atlantic, with an estimated (negative) economic impact on airline ‘passenger and cargo services’ of US\$1.7 billion (IATA Economic briefing, 2010). Ash particles can also cause potential health conditions in humans where particles are inhaled (Lombardo et al. 2013). Explosive eruptions can send plumes of volcanic ash thousands of metres into the atmosphere that whilst buoyant can travel expansive distances in suspension (Gudmundsson et al. 2012, Stevenson et al. 2015). What is less well understood are the underlying reasons behind the damage to aircraft and health effects associated with volcanic ash in the wider environment (Moore et al. 2002, Clarkson et al. 2016, Oudin et al. 2013, Lombardo et al. 2013). Integral to mitigating these effects is a better understanding of the nature such as shape, size and roughness of the particles themselves. Furthermore measurements of volcanic particle morphology are considered important in understanding fragmentation as well as particle behaviour (Liu et al. 2015).

It has been known for several decades that volcanic eruptions could disrupt air travel locally. In 1982 two aircraft flying through an ash plume suffered engine malfunctions resulting in emergency landings (Gourgauda et al. 2000). However, since the 2010 Eyjafjallajökull eruption, awareness of this specific hazard has been heightened in part due to the fear of potentially catastrophic damage to airframes and aircraft engines (Casadevall 1994, Clarkson et al. 2016). From a respiratory health aspect, it is particles less than 10 µm in size (PM<sub>10</sub>) that cause most concern as larger particles may

39 be caught by the nose, mouth or throat (Baxter et al. 1999, Forbes et al. 2003). These small particles  
40 are also those likely to remain in suspension longest after a volcanic plume enters the atmosphere.

41 In view of the size of the particles, microscopy techniques form an essential component in the study  
42 of volcanic ash. Previous work in this area used conventional light microscopy (Stevenson et al.  
43 2015) and scanning electron microscopy (Hillman et al. 2012, Fitzsimmons et al. 2013) to examine  
44 the 2D appearance of volcanic ash particles. Stereoscopic SEM techniques as well as micro-CT  
45 imaging have been applied to study the surface area to volume ratio of 63 and 125  $\mu\text{m}$  ash particles  
46 (Ersoy 2010, Ersoy et al. 2010); the lateral resolution of the micro-CT imaging was 6  $\mu\text{m}$  and although  
47 the resolution in stereoscopic SEM was much higher than in the micro-CT studies it could be difficult  
48 to image the complete surface with the SEM method described. We have previously applied  
49 reflection confocal microscopy to study of rock forming minerals including apatite (Petford and  
50 Miller 1993) and the 3D appearance of solid state nuclear track detectors (Wertheim et al. 2010,  
51 Gillmore et al. 2017). These studies suggest 3D confocal imaging may help to characterise and  
52 quantify the physical nature of volcanic ash particles in ways not amenable to more traditional 2D  
53 microscope techniques. Table 1 lists some of the possible methods that could be used to image  
54 volcanic dust particles. Two major advantages of using confocal microscopy in such studies are the  
55 ability to overlay a colour image and the relative ease of sample preparation not requiring coating or  
56 cutting.

57

## 58 **Method**

59 Three sets of volcanic ash from, the first days of the eruption in April 2010 of Eyjafjallajökull (A),  
60 another sample from Eyjafjallajökull Island mountain glacier 2010 (B) and Grimsvötn May 2011 (C)  
61 were assessed.

62 From each sample, a portion was sieved and washed in order to select a clean and similar particle  
63 size, thus allowing for ease of focusing. The preparation was similar to the 'Strew' slide technique  
64 and involved distributing the sieved particles on double sided tape mounted on a glass slide. Each  
65 sample was washed with propan-2-ol in an ultrasonic bath. The solution was poured onto a filter  
66 paper and allowed to dry on a hotplate at 40°C. A small strip of double sided tape was placed on  
67 glass slides and each sample spread evenly across the surface. Crucially, sample preparation did not  
68 involve any grinding or cutting of the material, meaning the ash particles are intact and unmodified  
69 prior to imaging.

70 Twenty volcanic dust particles were imaged using a LEXT OLS4100 laser scanning confocal  
71 microscope (Olympus Corporation, Japan) with a 405 nm laser, in a similar fashion to that previously  
72 described (Wertheim et al. 2010). In the present studies images were taken using a x50 or x100  
73 objective lens both having a Numerical aperture of 0.95; for 3D surface examination data were  
74 collected using the fine mode setting (60nm z-axis pitch spacing). The sieving procedure in the slide  
75 preparation was an important step in order to allow focussing in view of the working distance of the  
76 lenses (350  $\mu\text{m}$  for the x50 and x100 objective lenses); it was crucial to avoid large particles greater  
77 than the working distance of both lenses. During imaging the lowest level was set by focussing  
78 slightly below the level of the double sided tape; the upper level was set at slightly above the  
79 maximum detected height on laser imaging. In this study the size of dust particles examined was

80 similar to those expected to be seen in volcanic ash clouds and hence the maximum major axis  
81 length was about 120  $\mu\text{m}$ . Particle surface area and volume measurements were made using  
82 Olympus OLS4100 software version 3.1.8 (Olympus Corporation, Japan).

### 83 Results

84 Volcanic ash particles were imaged successfully in 3D using the confocal microscopy technique. The  
85 appearance of particles revealed frequent, sharp, knife-like edges with some particles showing  
86 concave surfaces suggestive of remains from gas bubble inclusions (Figures 1 and 2). The true colour  
87 image which is superimposed gives further insight into the nature of the particles; the colour image  
88 shows the surface features including colour thus enabling for example crystalline structure to be  
89 detected. Examples below illustrate scatter from crystals within particles (Figures 1 and 3) as well as  
90 concave morphology reflecting breakup of bubble texture leaving apparent sharp edges (Figure 3).

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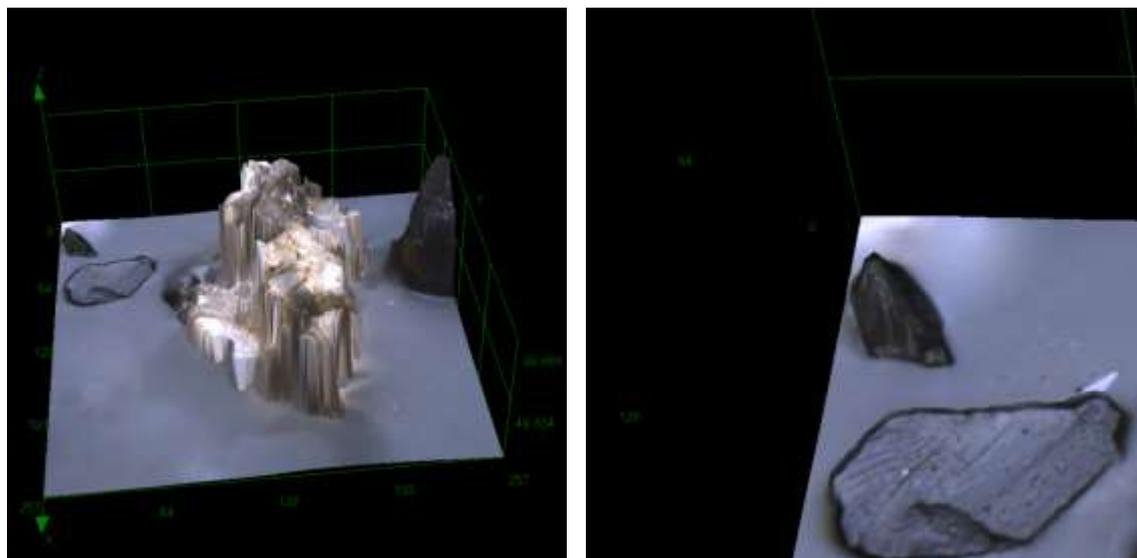
### 92 Size range above 20 $\mu\text{m}$

93 Figures 1 to 3 show examples of images from confocal microscopy of larger particles above 20  $\mu\text{m}$ .  
94 The 3D appearance of the volcanic ash particles was examined. Some particles for example showed  
95 crystalline patterns, uneven surfaces, sharp appearing surfaces or concave surfaces suggestive of  
96 reflecting breakup of bubble texture.

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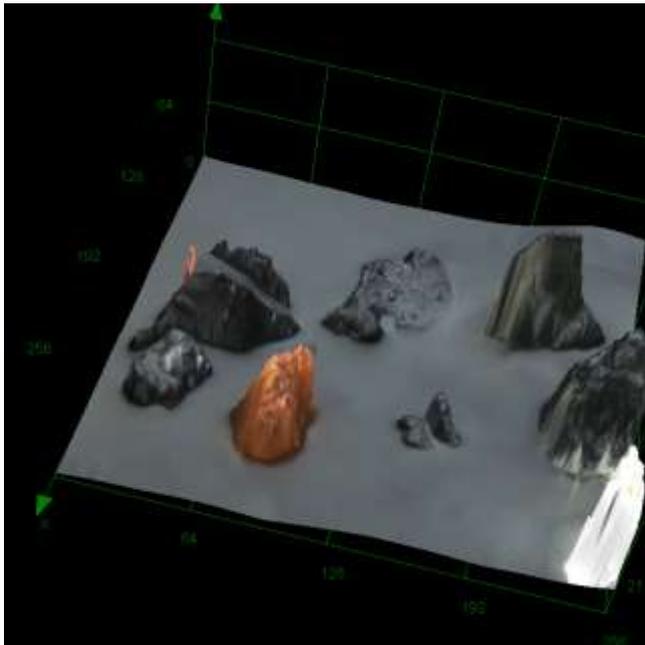
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101 Figure 1: 3D confocal microscope image of volcanic ash (glass) particles from sample C Grimsvötn  
102 eruption with centre particle showing light scatter from inclusions within in Figure 1a. The concave  
103 morphology reflects breakup of bubbly texture leaving apparent sharp edges. One small fragment of  
104 about 16 x 28 x 10  $\mu\text{m}$  is also seen in the upper left corner. Objective lens x50, image size 260 by 260  
105 by 100  $\mu\text{m}$ . A zoomed image of the small sharp appearing fragment is shown on right.

106



107

108 Figure 2a: 3D view showing a diverse range of particle sizes and colours, x50 objective lens, image  
109 size 260 x 260 x 128  $\mu\text{m}$  from sample A (Eyjafjallajökull eruption).

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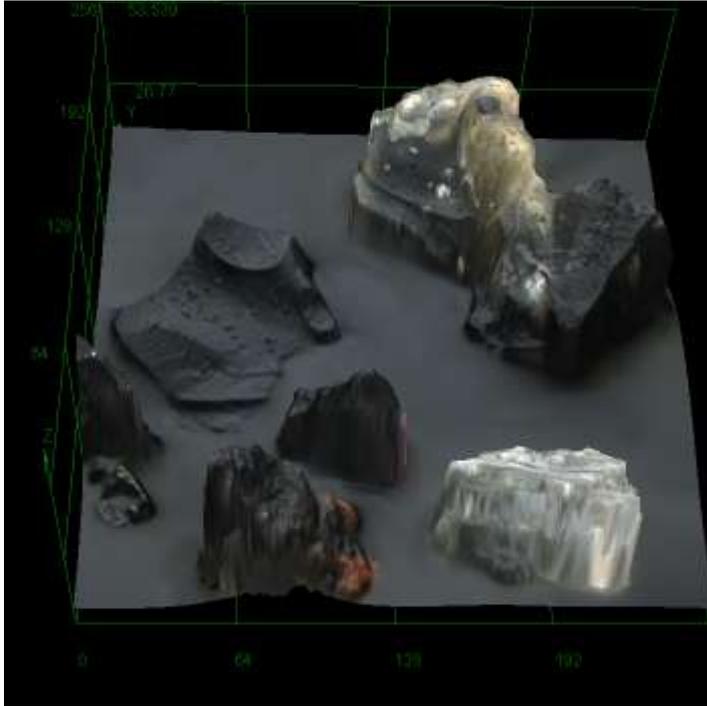


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113 Figure 2b: Close-up view of specimen in Figure 2a showing sharp edges associated with various sizes  
114 of fragments including small particles of sample A (Eyjafjallajökull eruption).

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116

117 Figure 3: Further examples showing the wide and striking variation in shape, colour, transparency  
 118 and surface roughness of volcanic ash particles, x50 objective lens, image size 260 x 260 x 54  $\mu\text{m}$   
 119 from sample A (Eyjafjallajökull eruption). The top left particle shows a concave structure which is  
 120 suggestive of the remains of a bubble.

121

122 **Size range  $\text{PM}_{10}$**

123 Six small particles with longest axis between 5 and 15  $\mu\text{m}$  were imaged using the x100 objective lens.  
 124 Overall two patterns of 3D surface appearance were observed: 1. scatter type undulating uneven  
 125 surface (Figure 4) and 2: an appearance characterised by sharp edges (Figure 5).

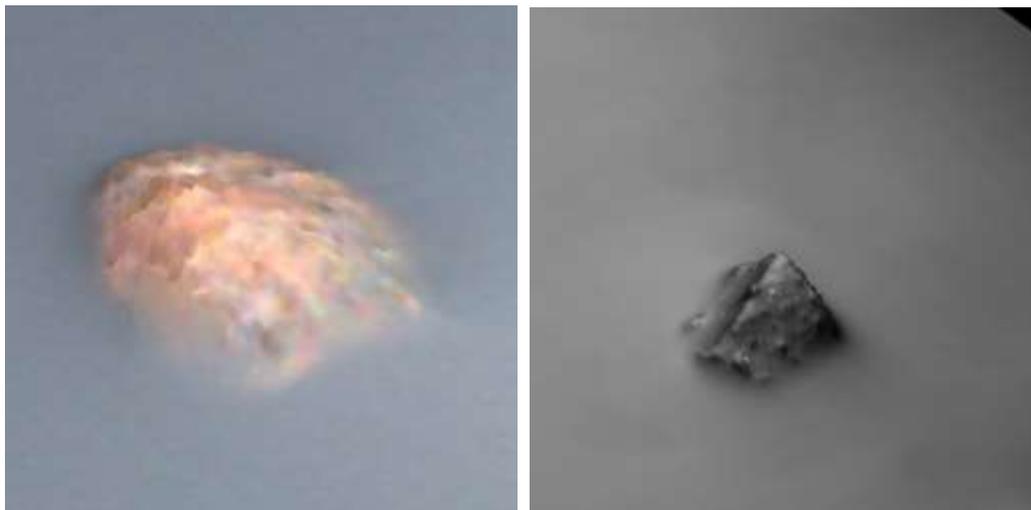
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127 **Surface area/volume ratios**

128 For the right hand particle in Figure 4 the volume was 112  $\mu\text{m}^3$ , the surface area (excluding the  
 129 lower surface) was 135  $\mu\text{m}^2$  with the lower surface having an area of approximately of 112  $\mu\text{m}^2$ .  
 130 Thus the surface area to volume ratio was 2.2  $\mu\text{m}^{-1}$  with an equivalent diameter for the lower surface  
 131 area of 12  $\mu\text{m}$ . The particle in Figure 5 had a volume of 38  $\mu\text{m}^3$ , the surface area (excluding the  
 132 lower surface) was 71  $\mu\text{m}^2$  with the lower surface having an area of approximately of 53  $\mu\text{m}^2$ . Thus  
 133 the surface area to volume ratio was 3.3  $\mu\text{m}^{-1}$ . The surface area to volume ratios are different as the  
 134 sharp surface in figure 5 has a higher relative surface area to that in figure 4, however the difference  
 135 is not as much as might be expected as the surface in figure 4 is not smooth.

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139 Figure 4. Examples of particles in the general PM10 range showing a highly undulating surface (left  
140 image) and a more polygonal structure with sharp sides (right image) sample B (Eyjafjallajökull  
141 eruption Island mountain glacier)..

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148 Figure 5. Example showing two views of the same sharp volcanic fragment (6 by 12  $\mu\text{m}$ ) from sample  
149 C Grimsvötn eruption.

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### 153 **Conclusions**

154 Previous studies of volcanic ash particles have mostly used light microscopy or SEM imaging which  
155 mainly only provide 2D visualisation and data of 3D structures. Stereoscopic SEM imaging can be of  
156 use but the technique could be limited in its imaging capacity because of occlusions (Ersoy 2010).  
157 Micro-CT is another possible imaging modality that has been applied to give the outline appearance  
158 of volcanic particles (Ersoy et al. 2010). Atomic Force Microscopy (AFM) has been used in  
159 atmospheric aerosol particle studies (Li et al. 2016) however, the study suggests this approach is  
160 more applicable to particles less than about 2  $\mu\text{m}$ . AFM has also been used together with  
161 transmission electron microscopy (TEM) to study sea salt aerosols (Chi et al. 2015).

162 To obtain a true record of particle geometry in 3D quickly and non-destructively, confocal scanning  
163 laser microscopy provides an optimal solution. This non-destructive technique maintaining original  
164 sample geometry, not only provides high resolution image height data it also allows a colour optical  
165 image to be superimposed. Incorporating a colour image with height allows the identification of  
166 structures on the surface of particles to be easily made and as seen detection of crystalline structure  
167 as well as helping to detect concave morphology reflecting breakup of bubble texture leaving  
168 apparent sharp edges. Applying this method, it is apparent the particle edges are frequently sharp  
169 across all size ranges investigated. Our approach also allows calculation of volume, surface area and  
170 shape thus enabling calculation of surface area/volume ratio.

171 Particles of the order of 10  $\mu\text{m}$  ( $\text{PM}_{10}$  size) with sharp edges were also observed. These images may  
172 help understand better the increase in respiratory and cardiovascular symptoms associated with  
173 inhalation of air contaminated with volcanic ash particles (Gudmundsson 2011, Lombardo et al.  
174 2013, Hlodversdottir et al. 2016).

### 175 **Further Work**

176 Damage to aircraft engines in flight that inject volcanic ash can result from melting of particles in  
177 the temperature range of aircraft engines 1200 to 2000  $^{\circ}\text{C}$  (Song et al. 2016, Dean et al. 2016).  
178 Particle size and its relationship to melting point has an important bearing here as experimental  
179 work on grain size and melting point show (Volcanic Ashfall Impacts Working Group, 2014). Our  
180 results suggest damage may also occur as a result of mechanical abrasion by sharp particles,  
181 consistent with observations of turbine blade damage as well as paint being stripped from one plane  
182 travelling through a volcanic ash cloud (Casadevall 1994, Weinzierl et al. 2012). The surface  
183 area/volume ratio of ash particles is considered of value when assessing volcanic plume dynamics,  
184 shape and particle interactions, transportation and deposition of ash particles (Ersoy, 2010).  
185 Potentially the confocal technique could be complemented with SEM to also provide information  
186 about the composition of particles; such an approach may facilitate understanding the underlying  
187 reasons for different particle colours and appearances that we observed. The confocal technique  
188 outlined here can obtain these data easily and offers promise to further advance this important  
189 aspect of modelling volcano plume-atmosphere interactions. Finally, further studies are required to

190 see if such sharp features are present in other types of PM<sub>10</sub> particles that impact negatively on  
191 human health when inhaled.

192

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195 commercial, or not-for-profit sectors.

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277 Table 1: Comparison of expected potential advantages and suitability ( ✓ ) of different techniques  
 278 for imaging volcanic dust particles. The standard implementation of techniques listed are for 2D  
 279 brightfield light microscopy, micro-CT, scanning electron microscopy (SEM), atomic force microscopy  
 280 (AFM) and confocal microscopy. Chemical composition refers to the ability in certain techniques to  
 281 obtain details of chemical composition, e.g. for AFM in combination with infrared spectroscopy.  
 282 Suitable scanning area and height refers to whether these are likely to be appropriate for imaging  
 283 the wide range of particle sizes seen with volcanic dust particles. Colour imaging refers to the ability  
 284 of the technique to provide a visual colour image of the particles' appearance.

Method	3D imaging	Chemical composition	Suitable scanning area	Suitable scanning height	Colour image
<b>2D Brightfield light microscopy</b>			✓		✓
<b>Micro-CT</b>	✓		✓	✓	
<b>SEM</b>	stereo ✓	✓	✓	✓	
<b>AFM</b>	✓	✓			
<b>Confocal microscopy</b>	✓		✓	✓	✓

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