

Preparation Techniques for Microanalysis of Fine-Grain Tephra that Incorporates Imaging Tephra Particles Prior to Quantitative Analysis: Examples from WDC06A Core

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Geochemical analysis on fine grained (<20 μ m) volcanic ash found in ice cores are inherently difficult. Samples are difficult to manipulate due to their fine grain size and low abundance of particles. Typically, samples are too sparse for bulk analysis and therefore microanalytical techniques, like electron microprobe (EMP) analysis (major elements); ion microprobe (IMP) and laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) (trace elements) are required. These methods all require samples to be polished and completely flat to provide precise and accurate geochemical analyses. A variety of sample preparation methods have been used to achieve this polished flat surface, but few provide the flexibility to look at individual particle morphology prior to polishing for quantitative analysis, this technique was first described by Kuehn et al. (2010).

We have developed an updated sample preparation procedure that has been implemented for englacial tephra from the WAIS Divide core (WDC06A). Samples are prepared using a combination of methods from Dunbar et al. (2003) and Kuehn et al. (2010) (Fig. 1). Samples can be prepared in parallel utilizing the best aspect of both methods. Samples are first filtered through a 0.2 μ m nucleopore polycarbonate membrane. Once the sample is adhered to the filter, Scotch tape is wiped along the filter to pick up fine grained particles. The tape is then pressed to a vitreous carbon planchet (VCP) that has been painted with a light adhesive. Multiple samples, typically 4, can be adhered to the same planchet, reducing the time needed for sample change. The tape is peeled back leaving some of the particles on the tape and some on the VCP (Fig1B). The VCP can then be imaged without carbon coating by SEM. After the samples have been imaged a tape dam is adhered around the VCP and is back filled with EpoThin epoxy (Fig. 1C). After curing, the new EpoThin round is separated from the VCP using a hammer and a razor blade (Fig 1D). Particles that are still adhered to the tape are attached to the bottom of a 4-hole epoxy round. The round is then back filled with Spurr low viscosity epoxy and cured under a lead weight for 12hrs (Fig. 1E). Both rounds are then polished with a solution of water and diamond grit powder. The samples can then be used for quantitative analysis by EMP, LA-ICP-MS and IMP. The parallel sample preparation method allows for multiple microanalytical methods to be performed on a single sample, as well as providing back-up sample to be archived.

The updated sample preparation technique discussed above provides the ability to qualitatively and quantitatively analyze the same tephra particles. SEM images are a qualitative way to assess what processes the tephra underwent during fragmentation, transport and deposition, which can be helpful in determine eruptions type (Fig. 2). The top images found in Fig. 2A and 2B were carbon coated and show more detail than non-carbon coated images (2C and 2D). The non-carbon coated samples show the type of imaging capabilities that this sample preparation method can produce. Carbon coat is used to reduce charge build-up on the shards during imaging. Fig. 2C and 2D were not carbon coated but still provide useful images because the particles are small and isolated from other tephra. Some charging can be seen as bright white spots at the edges. Large clusters of particles tend to have more charging because they are more poorly grounded. The tephra in Fig. 2A and 2C are platy shards with truncated vesicles (broken bubble walls) defining their edges. The small size (~30 μ m), thin bladed appearance and large surface area allow these particles to be transported long distance, whereas particles in Fig. 2B and 2D would travel

less distances because of their larger size ($\sim 75\mu\text{m}$) and more blocky shape. All of these tephra shards are typical of explosive volcanism where the driving force is bubble expansion.

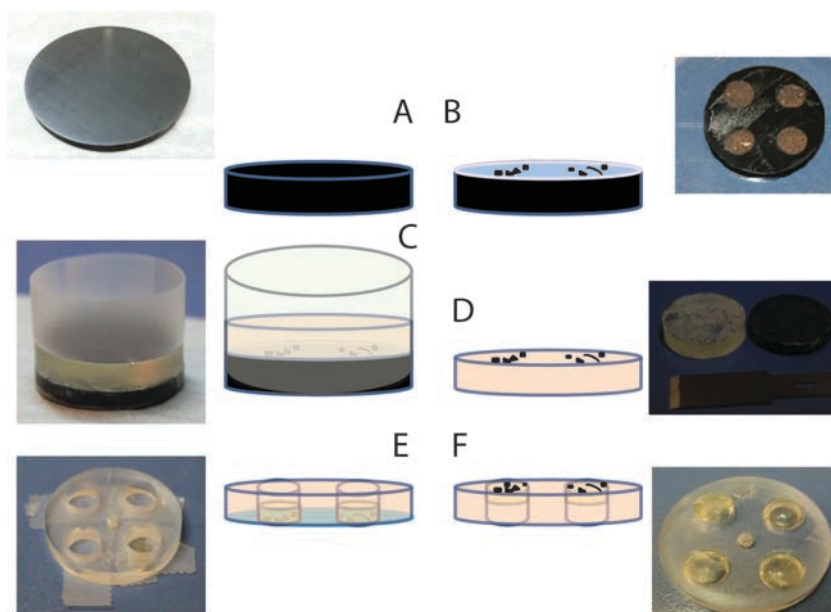


Figure 1: Cartoon and pictures of the sample preparation technique discussed in text. Black cylinder= Vitreous Carbon Planchet, Peach cylinder= EpoThin round, Peach cylinder with holes= Spurr low viscosity epoxy round, Blue= light adhesive (B) and Scotch tape (E)

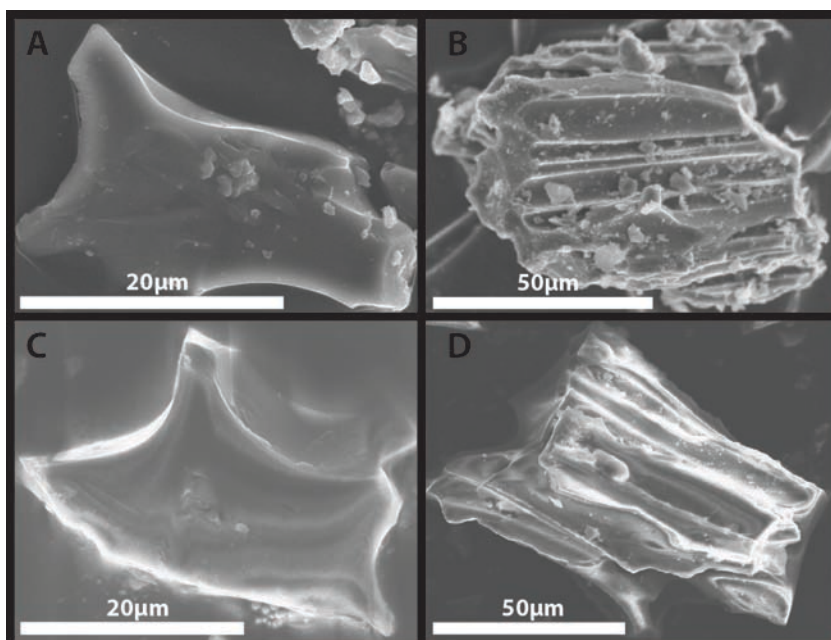


Figure 2: SEM images of tephra particles with (A and B) and without (C and D) carbon coat.