Faster and More Accurate Processing of Samples for Microtephrochronology

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1 Introduction

Microtephrochronology is a stratigraphic dating method used by archaeologists and Quaternary scientists involving the location and characterisation of volcanic glass particles present in soils and sediments in sizes and amounts invisible to the unaided eye (e.g., Alloway et al. 2007; Turney and Lowe 2001). Accurate determination of the presence or absence of volcanic glass shards in a soil or sediment sample precedes quantitative analysis. This determination is typically accomplished using optical microscopy (Fig. 1) and is one of the most time-consuming and laborious steps in the process, with great potential for misclassification or miscounting error. Here we present a novel method of identifying and counting volcanic glass particles while simultaneously providing a rough geochemical characterisation of all the particles present in the sample (glass and non-glass), using an Aspex PSEM 3025 Particle Analyzer with Automated Feature Analysis^{TM} (AFA) software (Fig. 2).

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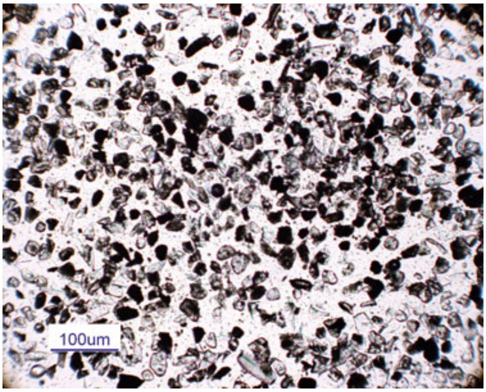

Fig. 1 An example of a sieved tuffaceous sand showing glass and non-glass grains of various morphologies from the flanks of Göllü Dağ, Turkey. Polarised light microscopy is used to identify and count shards of volcanic glass in soil and sediment samples. This is laborious and time consuming, and identification of glass shards can be highly subjective and not always secure. Counting of hundreds of shards, even when samples are spore-seeded, can be inaccurate due to the nature of the sample or operator error. (Photo by Mel Wachowiak, Museum Conservation Institute, Smithsonian Institution.)

2 Background

After extracting the fraction of soil or sediment where distal microtephra is likely to be found, samples are typically mounted on glass slides and visually "scanned" using polarised light microscopy to determine if volcanic glass shards are present (see Gehrels et al. 2008 for a recent review). If found, the shards must be counted in order to determine the number of shards per cm^3, data which is required to estimate relative shard abundance, eruption volume, and post-depositional reworking. Shard morphologies differ within and between eruptions and/or volcanic systems, and methods for optically distinguishing between tephra and non-tephra vary in their usefulness from sample to sample. Hence there is a long training period before a worker becomes skillful in identifying tephra, and even then there is a certain amount of subjectivity involved in identifying shards, especially when seeing a new morphology (or diagenetic state) for the first time.

The Aspex Personal Scanning Electron Microscope (PSEM) is a specially designed SEM paired with an energy dispersive spectrometer (EDS) and particle recognition and statistical software. It was developed for automated imaging and analysis in industrial applications, but it has obvious applications to microtephrochronology. The PSEM analyses upwards of 1,000 particles per hour, can be programmed to distinguish between anything elementally or morphologically distinct, will not make judgment errors, and gives an absolute count of each type of particle it finds, the size of every particle, and an EDS spectrum of major and minor elements (down to about 1% by weight) of all particles, making it

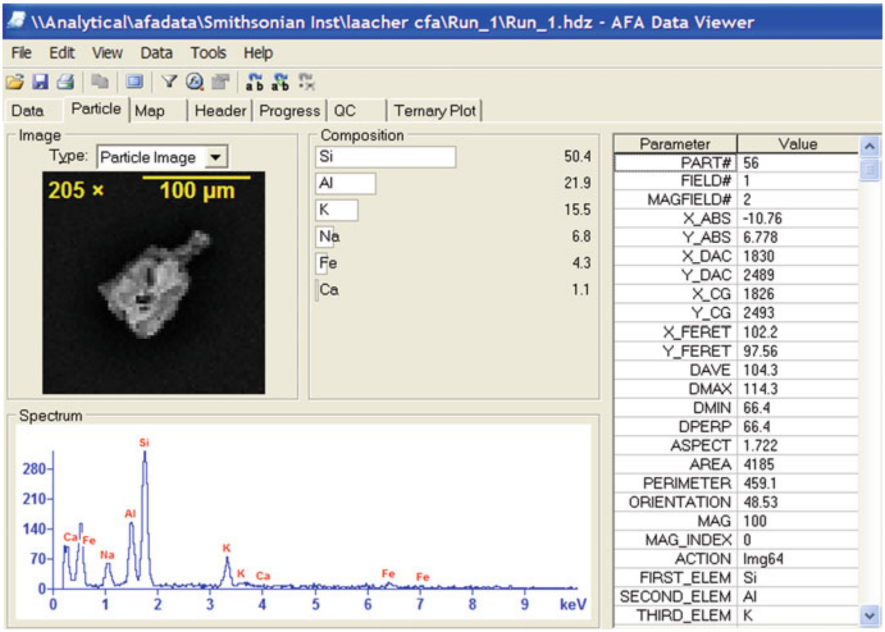

Fig. 2 Screenshot of the Automated Feature Analysis™ (AFA) Dataviewer. In this tab are displayed a low resolution image of the particle (64 × 64), EDS spectrum (acquisition time 2 s), histogram and quantitative results, and dimensional data. Low resolution settings were selected for quick scanning

possible to look for patterns or trends without any additional investment of time. In addition, it takes an image of each particle and records its coordinates on the SEM stub as described below (see Fig. 2), allowing rapid re-location of specific shards for further analysis (at a longer acquisition time for quantitative analysis if desired).

Identification of particles is accomplished using one of two algorithms. When a particle is identified, its image is captured, an EDS spectrum is collected, and morphological information is calculated and recorded, along with its exact position on the sample stub. The PSEM 3025 systems dynamically scan the sample. Rather than capturing a high-resolution image of the field, which is time consuming and inefficient, the PSEM 3025 instead moves the beam across this field in an array of fairly coarse steps. At each point, the brightness of the backscattered electron signal is noted. If the signal is bright enough to indicate that a particle is present at this position, the software initiates a particle-sizing sequence. There are several algorithms that can be used for this purpose, but for simple particle shapes, the rotating chord algorithm is both accurate and exceptionally fast. Once the coarse scanning identifies a particle, the center is identified and chords are drawn on the particle to define the particle's size and shape. The major reason for this improvement in speed is because the PSEM 3025 only spends time collecting detailed data where particles are known to be present, rather than capturing and transferring vast numbers of empty pixels.

3 Methods

Three morphologically and chemically distinct tephra samples were analysed; proximal samples from Laacher See and Hekla (1947), as well as an obsidian sample from Romania (Limba) which was ground using a mortar and pestle (Watson 2006). Dry samples which had been extracted by heavy liquid flotation using sodium polytungstate and sieved to between 25 and 90 μm were dispersed onto weighing paper. Samples were transferred onto 15 mm SEM stubs bearing double sided conductive carbon adhesive discs by pressing the exposed surface of the carbon discs onto the weighing paper containing the sample. These stubs were placed (without coating) into the sample stage and introduced into the chamber. Samples were run at full vacuum at 15–25 kV. Each particle was analysed for 1–4 s. Experimental conditions for the study of highly vesicular shards were optimized to avoid double-counting, as the automated analysis is based on backscattered electron signal, and pores and voids within the shard produce a similar signal to the substrate (carbon tape).

4 Results and Discussion

The PSEM was able to identify, count, and geochemically characterise tephra shards of varying morphologies, including highly vesicular shards. Less than 4% of the total counts mistook two particles for one when particles were overlapping. This is expected to improve with experience, and there are other methods of sample dispersion which would eliminate the problem.

While the PSEM 3025 identifies all particles present in a sample by means of characterisation by EDS, a separate step is necessary for quantitative chemical analysis, as the EDS analysis for the scanning process is less than 5 s long. The separate step for quantitative analysis can be done using this same instrumentation, either specifying ahead of time that all particles matching a certain chemical profile be analysed for longer (e.g. 100 s), or by resubmitting the same stub to be analysed later, with the instrument programmed to go to specified particles for a longer acquisition time. The location of each analysis is stored so that the stage can be returned to any particle. If WDS analysis is desired, the stub could be embedded, polished, and carbon coated. In this case, analysis on a different instrument would be aided by a map of the location of volcanic glass particles which is produced by the PSEM 3025.

Further work is required to establish ideal analytical parameters for both scanning and quantitative analysis, and it is likely that these will vary somewhat with different samples.

5 Conclusion

By providing the ability to accurately distinguish between glass and non-glass at a rate of up to 3,600 particles per hour, the PSEM 3025 offers a cost-effective method of scanning soil and sediment samples for the presence of microtephra. This method is an improvement over polarised light microscopy in that it provides a total particle count broken down by material and avoids classification and counting errors.

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