Mechanical and Chemical Dissolution Methods Versus Selective Fragmentation for Mineral and Fossil Separation and Concentrates from Select Geologic Materials

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Abstract

Historically, the first steps in mineral and fossil comminution/separation in rocks have involved mechanical force (crushing and grinding) or chemical dissolution. Selective fragmentation is a technology that uses high voltage pulse power fragmentation in an insulating medium (water) to progressively break materials along grain boundaries. Some advantages of selective fragmentation include low dust production, fewer fractured grains, coarser mineral separates, and no hazardous chemical exposure or waste. This study attempts to determine qualitative differences between selective fragmentation and traditional comminution practices in a variety of samples that represent commonly processed rocks at the USGS.

Selective fragmentation of geologic materials was performed on samples including granodiorite, quartz pebble conglomerate, amphibolite, ash flow tuff, and fossiliferous carbonate. Minerals and fossils of interest include zircon (igneous and detrital grains), apatite, amphibole, monazite, titanite, sanidine, and conodont tests. Each sample has corresponding splits reduced by traditional methods (jaw crusher and vertical grinder or acid-buffer dissolution for carbonates). Both the selective fragmentation splits and traditional splits were further processed for mineral and fossil concentrations of interest by using magnetic separation and heavy liquids. Splits of selective fragmentation and traditionally reduced methods were examined visually by binocular scope and in more detail by electron microscopy. Some initial observations for selective fragmentation include rapid comminution (<10 minutes/sample), more composite grains, more fully intact grains, and less fine-grained material (e.g. dust). Traditional sample reduction methods observations include, heavy dust generation, many fractured grains of interest, but few composite grains. Buffered acetic acid digestion of carbonate rocks yields almost complete recovery of conodonts and other residue that can typically be concentrated in magnetic and heavy liquid separation splits relatively easily. The separation of condont by selective fragmentation requires large efforts in post-processing methods including large sample throughput in magnetic separation and heavy liquid separation.

Methods

Each sample was reduced by the two methods mentioned- selective fragmentation and mechanical comminution. Selective fragmentation reduces whole rock to its constituent minerals by inducing high-voltage pulse power that separates the minerals along their natural physical boundaries. The shockwave wave that follows the discharge track (illustrated in Figure 1) acts as a comminutive force by physically separating the constituents. Selective fragmentation of the geologic minerals was undertaken at the SEFRAG Lab in Switzerland, where a lab technician determined the correct operational parameters for selective fragmentation of each individual sample. The following parameters were used:

- Temora 2: 8 cycles @ 190-200kV, 5 Hz, 50 pulses
- 74AR15: 10 cycles @ 195 & 200 kV, 5 Hz, 6 cycles @ 60 pulses, 4 cycles at 100 pulses
- Conglomerate: 8 cycles @ 194 & 200 kV, 5 Hz, 50 pulses

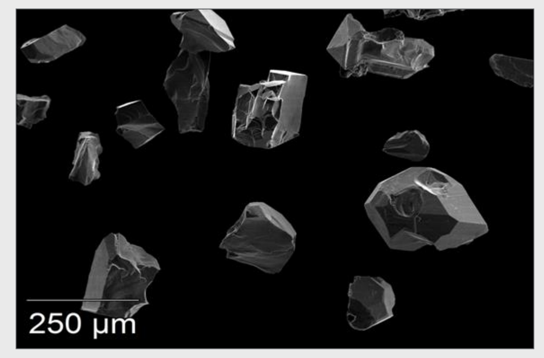
Mechanical comminution was used on representative fractions of each of the geologic samples, as well; chemical dissolution using an acid-buffer solution for the carbonate rock For this reduction process whole rock was first reduced by a jaw crusher, then further reduced by a disk mill, before being sieved to a size range of 44-300 microns (325-50 mesh). Once sieved, the bulk material was then processed on a Wilfley water table, which effectively separates and concentrates heavy minerals (>3.0 SG). Selective fragmentation fractions were also sieved to this size range, but not concentrated by the Wilfley table. Each of the separately-processed fractions were then separated using a Frantz isodynamic magnetic separator, where subsequent paramagnetic charges (0.4A, 0.8A, 1.8A) were used to reduce the bulk material in stages. Once a non-magnetic fraction was obtained for each process (usually containing heavy minerals such as zircon and apatite) it underwent a gravity separation utilizing methylene iodide (~3.32 SG) as a density medium.

Upon acquiring non-magnetic, heavy mineral fractions for each process qualitative observations were made via visual discrepancy and comparison of the fractions under binocular microscope and scanning electron microscopy (SEM). The binocular microscope afforded a general overview of the effectiveness of each process, as well as an understanding of the behavior of the mineral phases. SEM imagery, both backscatter electron (BSE) and secondary electron imaging (SEI), provided a visual test of the surface expressions of mineral grains (SEI) and contrast of mineral grains and inclusions (BSE). When BSE imagery indicated inclusions at fracture planes electron dispersive X-ray spectroscopy (EDS) was used in order to obtain a qualitative elemental composition of the inclusion.

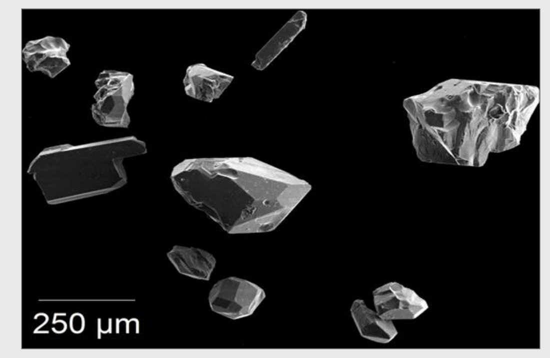
Images (selective fragmentation): Top left - Image of the Selfrag Lab used to reduce samples. Top right - cross-sectional view of the Selfrag Lab showing high-voltage pulse being introduced to the processing vessel. Bottom - Illustration showing the discharge track as it travels through the material following natural boundaries.

Images (traditional methods): Top left - Jaw crusher. Top middle - Disk mill. Top right - Sieve shaker. Bottom left - Wilfley table. Bottom middle - Frantz magnetic separator. Bottom right - Gravity separation via methylene iodide (heavy liquid).

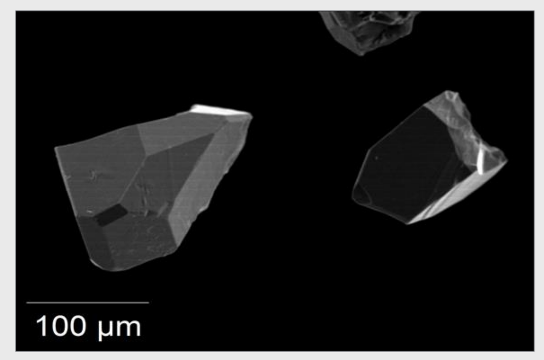
Figures



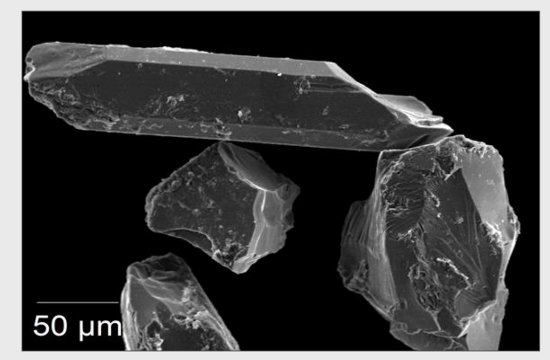
Selective fragmentation fraction (Temora 2): SEI image of typical zircon grains liberated.



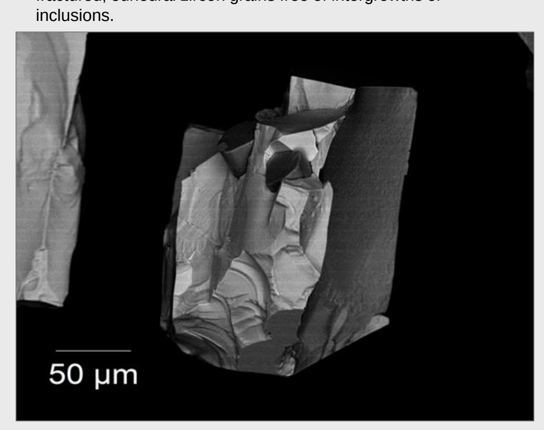
Traditional fraction (Temora 2): SEI image of typical zircon grains liberated.



Selective fragmentation fraction (Temora 2): SEI image of fractured, euhedral zircon grains free of intergrowths or inclusions.



Traditional fraction (Temora 2): SEI image of fractured zircon grains with plagioclase intergrowths at the surfaces.



Selective fragmentation fraction (Temora 2): BSE image of zircon grain fractured at the contact of two XX inclusions. See illustration (right).

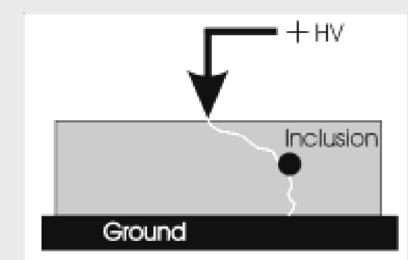
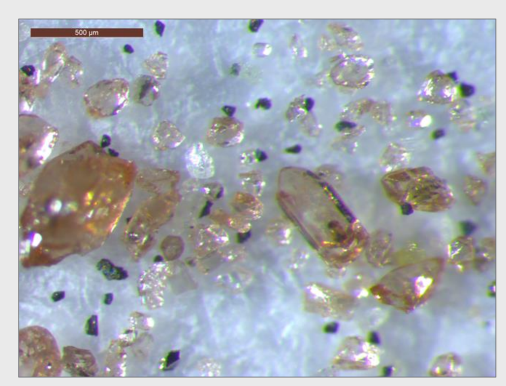
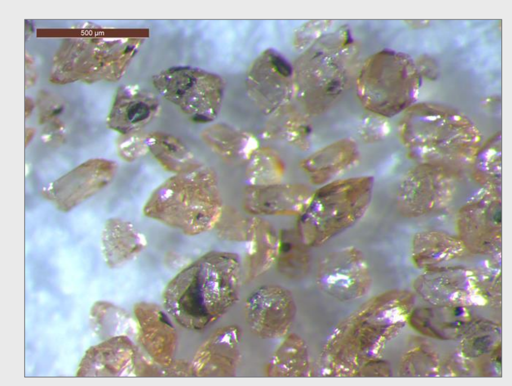


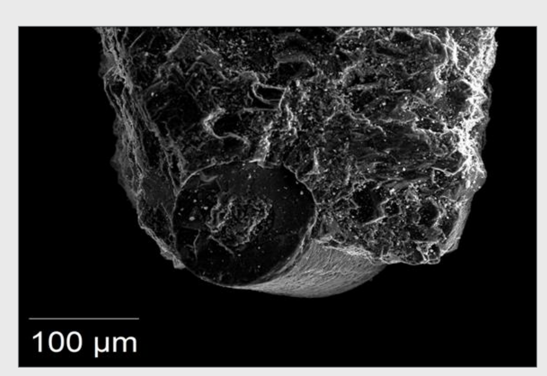
Illustration of the preferential attraction of the discharge track to metallic, or high dielectric constant, inclusions (as evidenced by the image to the left.)



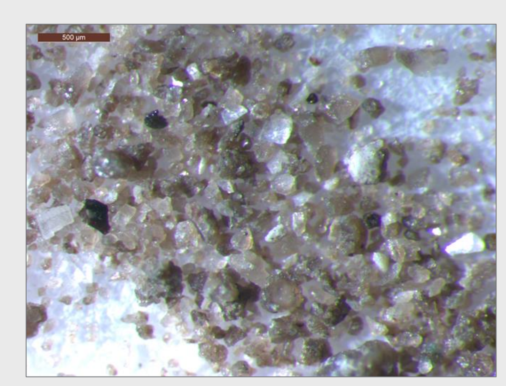
Selective fragmentation fraction (Temora 2): Reflected light image of zircons liberated. Note the near absence of in-place inclusions, of which can be seen lying around the zircons.



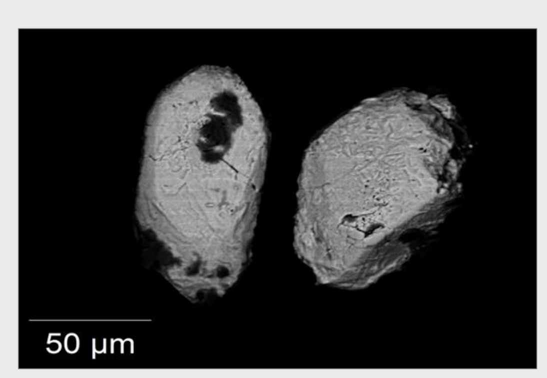
Traditional fraction (Temora 2): Reflected light image of liberated zircons. Note that nearly all inclusions have remained within the zircon grains.



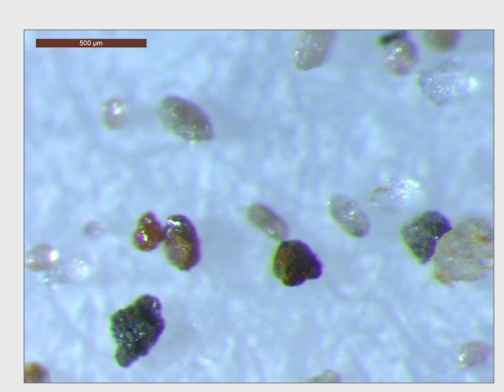
Selective fragmentation fraction (CO-ML-L1): SEI image of fractured, in situ microfossil.



Selective fragmentation fraction (CO-ML-L1): Reflected light image of carbonate rock material. Note the fragments of microfossil (dark grains) and euhedral calcite grains.

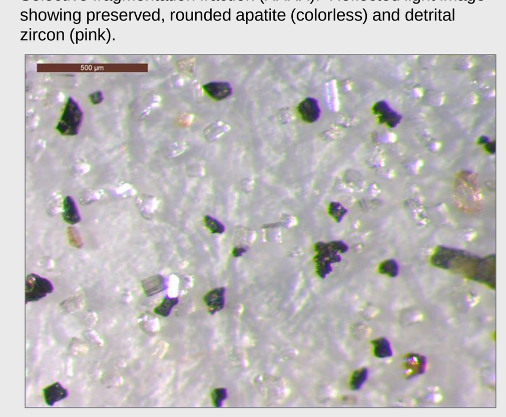


Selective fragmentation fraction (XXXX): BSE image of zircons with preserved inclusions and provenance features (rounding, surface pitting.)



Selective fragmentation fraction (XXXX): Reflected light image showing preserved, rounded apatite (colorless) and detrital zircon (pink).

74AR15



Selective fragmentation fraction (74AR15): Reflected light image showing euhedral apatite and fragments liberated.

Observations

Diorite Sample (**Temora 2**): Selective fragmentation fractions produced zircon grains free of mineral intergrowth and often free of inclusions. However, the presence of metallic inclusions attracted the discharge track, fracturing zircon grains. The traditional fraction did not fracture, in general, at inclusion sites, but produced many small fragments through mechanical comminution.

Carbonate rock (**CO-ML-L1**): Selective fragmentation wielded many small fragments of micro-fossils, as well as fragments in situ. Traditional methods, obversely, wield intact micro-fossils and has a near 100% recovery.

Quartz-pebble conglomerate (**CON-X**): Selective fragmentation preserves detrital zircon and apatite grains, as well as surficial provenance features that can be utilized during myriad analyses. Traditional methods can preserve the features noted in the selective fragmentation fraction, but to a lesser degree (i.e. some grains are broken/fractured).

74AR15: Selective fragmentation fractions show preserved apatite and zircon grains, as well as other heavy minerals, and a retention of provenance features. Traditional methods yielded similar results, yet there were a greater degree of broken or fractured grains.

Conclusions

The use of selective fragmentation to reduce geologic samples has many advantages that may or may not be found in traditional methods. Where traditional methods produce dust and fine material, fractured or broken grains, sample loss, and take several days to finish the process, selective fragmentation shines. Selective fragmentation produces very little dust, is quite fast (<10 minutes), and produces intact grains while retaining all material. The number of processes used traditionally can add to the risk of sample contamination, whereas selective fragmentation lessens and nearly omits the chance of contamination.

SEM imagery shows that grains liberated by selective fragmentation are virtually free of intergrowths and surficial remnants, while traditional methods produce grains with intergrowths and a greater degree of mineral fragments. Due to the highly inclusive zircon grains of the Temora 2 sample, several fragments were produced by selective fragmentation's preferential attraction to metallic inclusions (which may be quite useful for large-scale comminution of ore rock). This was not seen in selective fragmentation fractions where zircon inclusions were non-metallic.

The traditional method of chemical dissolution of the carbonate rock using an acid-buffer solution proves much more effective at liberating intact microfossils than selective fragmentation. Selective fragmentation did not adequately preserve or separate micro fossils from the host rock; many of the microfossils were found to be in small fragments, and/or in situ.

Overall, selective fragmentation proves particularly effective as a reduction method, especially when dealing with a small sample and material retention is critical, but also on larger samples in which the mineral(s) of interest may be scarce. Further research can provide a qualitative and quantitative analysis comparing the two processes among other types of material, geologic or otherwise.

Future Research

- Quantitative analysis of heavy mineral size and c-axis length.
- Determination of selective fragmentation as a means for reducing samples to undergo fluid inclusion analysis.
- Determine the effectiveness of selective fragmentation on a greater variety of geologic samples.
- Quantitative analysis of mineral fractures propagated or exploited by selective fragmentation.
- Determine the effectiveness of selective fragmentation in ridding glassy material from mineral grains, such as sanidine and other felsic minerals.

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