

## Liberation of Fossils Using High Voltage Electric Pulses

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## INTRODUCTION

The availability of a complete and efficient method for separating a rock's constituents is of interest to a wide spectrum of earth scientists, ranging from those interested in large-scale ore processing, to mineralogists or geochemists wishing to separate single crystals for crystal structure determinations or isotopic measurements, and to paleontologists wishing to examine individual microfossils. The process of physically disaggregating well-lithified sedimentary rocks to recover minerals or fossils of interest is hampered by breakage or production of multiminerale fragments (composite grains) during crushing. Selective dissolution of whole rocks using acids is also used to separate soluble components from insoluble matrices.

Hutchison (1974) reviewed techniques for crushing whole rock specimens to achieve good mineral separation. He stated that care must be taken not to crush the rock too finely, but enough to ensure that the fragments will be monomineralic. He also noted that the rock dust released during the crushing is a health hazard. Desliming, or removal of the dust from the surfaces of the mineral grains, must be done before magnetic or heavy liquid separation. Crushing commonly produces multiminerale grains, which hampers efficient separation of different minerals.

Kummel and Raup (1965) described paleontological preparation methods, including heat treatment, miniature sand-blasting, and mineral dissolution. For the latter method, common acids used are hydrochloric, acetic, formic, and hydrofluoric. Often, the sample must be soaked for many days, and care must be taken that the specimen itself is not lost to dissolution.

Over the past few decades, the feasibility of Electric Pulse Disaggregation (EPD) for mineral separation has been investigated by researchers in the former Soviet Union (Maurer, 1968, Kurets et al., 1989; Finkelshtein et al., 1989; Shuloyakov et al., 1989; Rudashevsky et al., 1995a), Europe (Andres, 1977, 1989; Andres and Bialecki, 1986; Bialecki et al., 1992; Saini-Eidukat et al., 1993) and the U.S. (Touryan et al., 1992; Weiblen, 1994; Rudashevsky et al., 1995b). EPD uses high voltage pulses to fragment a rock sample into its constituents. These independent investigations have shown that EPD has several advantages over conventional

crushing and grinding methods, including: a) breaking of minerals into their natural size distribution, as present in the rock and regardless of the grain size, b) lower production of rock dust, and c) formation of fewer multiminerale grains. Disadvantages include gaining access to specialized equipment not yet commercially available, the use of high voltage, and the need for the sample to be immersed in or saturated with a suitable dielectric liquid such as water or oil.

## THE EPD INSTRUMENT

The basic components of the unit are a high voltage power source fed to a voltage-increasing circuit, which discharges the current to the sample chamber (see Rudashevsky et al., 1995a, for details). With our instrument, a marx generator is used for increasing voltage, allowing the build-up of a voltage much greater than 100 kV; the exact voltage depends on the number of capacitors in the circuit. The water bath in which the sample rests actually acts as the last capacitor in the system.

At voltages below approximately 100 kV, the fluid discharges before the rock and the electrons circumvent the sample. At higher voltages, however, the rock electrically breaks down before the fluid. A plasma is formed inside the sample as a result of the breakdown, resulting in an explosion which preferentially takes place along zones of weakness in the solid. In the case of rocks, these zones of weakness seem to be along grain boundaries of mineral phases, particularly when the minerals have different dielectric constants. The discharge may move through the rock preferentially along grain boundaries because of the presence of better conducting paths there, perhaps in the form of interstitial fluids or ions.

The process of electric-pulse disaggregation may produce small amounts of contamination in the recovered product. This may consist of spheres or fragments of melted or spalled electrode material, i.e., steel, copper or aluminum used as electrodes. Less commonly, spheres or fragments formed by melting of the sample may be found in the finest grain-size fraction.

## APPLICATIONS IN PALEONTOLOGY

Here we present examples of EPD experiments made on fossiliferous sediments from North Dakota and South Dakota. Run products were dried, size classified, and examined using optical and scanning electron microscopy.

The first example is from the Carlile shale (Upper Cretaceous). These shallow inland sea sediments are exposed as a thin veneer lying unconformably on Precambrian granites in quarries of northeastern South Dakota. The Milbank site probably represents a bedrock high in the Cretaceous sea, which formed islands or steep shorelines. Our samples are from the Cold Spring granite quarry near the town of Milbank (outcrop B of Merewether (1983), sec. 18, T. 120 N., R 46 W., Grant County). The base of the shale is a conglomerate, which includes pieces of weathered granite, belemnites, shark teeth, and other components all cemented by carbonate.

We originally attempted EPD on these rocks to separate the abundant shark teeth. Some teeth were successfully freed, but the method charred and broke many of the larger specimens. However, EPD successfully liberated large numbers of microfossils, which were excellently preserved as pyrite molds.

EPD liberated a diverse and large number of microfossils at this location; Figure 1 shows a selection of microfossils recovered. Sloan (1964) reported similar pyrite molds of microfossils recovered using acetic acid dissolution. No post-EPD cleaning was done on these samples; even so, the separation from matrix and preservation of surface detail is excellent. Mineral overgrowths on the ostracode molds obscure details and may make precise identification difficult (F. Swain, personal communication, 1994).

The observation that ammonitellas found at this site (Fig. 1.2) match the size of those found elsewhere supports the idea that ammonite hatchlings were planktonic as opposed to the larger hatchlings of contemporaneous nautiloids (about 12 mm) that probably swam in deeper water (Landman, 1984; N.H. Landman, personal communication, 1995). The large numbers of ammonitellas found support Landman's idea that the reproductive strategy of ammonites was to release large numbers of eggs as opposed to the strategy of modern nautiloids of laying 5 or 6

eggs per year. Sedimentologically, this outcrop is interesting because there are non-pyritized fossils (belemnites, shark teeth) mixed in with pyritized fossils in a high energy environment. This observation could imply that the microfossils were pyritized elsewhere and transported to this site.

Figure 1.5 and 1.6 show spheroidal objects recovered from the Milbank basal conglomerate. Hundreds of these were collected from the fine fraction of the EPD sample and separated using a Frantz magnetic separator. The spheroids are intimately associated with the pyrite microfossil molds; some have smooth surfaces (Fig. 1.6) while others have ornamented surfaces (Fig. 1.5). They do not appear to be artifacts such as melted pyritized fossil molds or melted pyrite concretions, because they can be observed in situ. Also, casts of spheroids are observed in the calcite matrix. The origin of these spheroidal objects is enigmatic. Their approximate diameter is 300-500  $\mu\text{m}$ , an order of magnitude larger than typical framboids reported in, e.g., Rickard (1970) or Sawlowicz (1993). Whether they are biogenic, perhaps megaspores, or deposited by geochemical processes is equivocal and awaits further characterization.

A second sample which was investigated was from the Minnelusa Fm (Pennsylvanian-Permian) sampled from Elk Creek, 12 miles N. of Rapid City, South Dakota (Allan Ashworth and Joe Martinetti, collectors). This rock is a fine-grained sandstone, from which we recovered conodont fragments (Fig. 2). Again, this sample has not been cleaned after EPD.

A third sample is from a drill core of the Mowry Formation (Upper Cretaceous) from a well in Burleigh County, North Dakota. (John Hoganson, collector). Macrofossils which were recovered include a belemnite tip, fish scales and fish teeth (Figure 3). EPD also preserved the cast of a fish scale (Fig. 3.4), which might have been destroyed in conventional processing.

Further experiments were made on a Cretaceous age tar sand, and showed that nautiloids can be cleanly liberated from the asphalt mineralized matrix.

## CONCLUSIONS

For the samples in this study, EPD allowed us to separate hundreds of microfossil molds quickly, efficiently and cleanly. Perhaps the greatest advantage of EPD is that the breakage usually occurs along natural boundaries, resulting in whole, unbroken components. This pattern of breakage allows petrologists, geochemists, mineral engineers, or paleontologists to examine items of interest, recovered from the rock with minimal damage, and with surface features preserved. EPD can be used as a method of fossil liberation. However, its suitability for a particular sample must be determined on an individual case basis. A well-defined interface between minerals of contrasting dielectric constant should be present for the physical process to operate. The high voltage apparatus can be lethal and instrument construction must be undertaken with care. For EPD to become a routine fossil or mineral separation method, considerable method development is required.

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## FIGURE CAPTIONS

Figure 1. Scanning electron micrographs of microfossils recovered from the Carlile shale at Milbank, South Dakota. 1. juvenile clam, 2. ammonitella, 3. foraminifera, 4. ostracode, 5. spheroid, 6. spheroid.

Figure 2. A conodont fragment from the Minnelusa Fm (Pennsylvanian-Permian). Scale bar represents 100  $\mu\text{m}$ .

Figure 3. Fossils recovered from the Mowry Fm., North Dakota, sampled from well Kleven #1A, NDGS Well #4592, at 2,887 feet (880.0 m). Scale bar represents 100  $\mu\text{m}$ . 1. fish tooth, 2. belemnite tip, 3. fish scale, 4. impression of a fish scale.