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A technique for extracting small crystals from thin sections

PETER C. RICKWOOD[1]

Nordic Volcanological Institute
University of Iceland
Reykjavik, Iceland

Abstract

Micromanipulators used by life scientists have been evaluated for extracting minute grains (< 20 μm) from petrological thin sections. A pantograph design with a single control for all motion was found to be the most convenient, and sharpened hypodermic needles made ideal probes.

Unambiguous identification of minerals using petrological thin sections and optical techniques is often difficult, particularly when the available grains are small (< 30 μm) or so few in number that the opportunity of finding one in a suitable orientation is slim. Recourse can be made to X-ray techniques, but although the literature contains several methods for preparing extracted grains for X-ray powder photography, little attention has been given to the method of extraction. In their encyclopaedic work, McCrone and Delly (1973) noted that for X-ray diffraction studies the minimum size of grain is about 5 μm in diameter (*ca.* 10^{-10}g) but "very few experts in X-ray diffraction, electron microscopy, or microprobe analysis are adept at handling single particles near the limit of sensitivity of their instruments" (p. 222). Although they optimistically claimed some microscopists to be capable of "picking-up and delivering" grains smaller than 1 μm in diameter, this is a very different attribute from being able to extract such grains from thin sections.

Few petrologists appear to have recognized that polarizing stereomicroscopes which give an erect, laterally correct, stereoimage are particularly advantageous for manipulating thin sections. The optical magnification required is not great, but Barer and Saunders-Singer (1951) drew attention to the involuntary random motion of the human hand which becomes apparent under these conditions and which is commonly as large as 100 μm. To reduce such motion biologists have developed mechanical aids, and it is these micromanipulators that have been investigated for mineralogical work.

There are three main designs:

(a) Micromanipulators which offer controlled motion along three independent axes. Two instruments with orthogonal micrometer screws, but of different design and manufacture, were tried. Both lacked "feel sensitivity" which often led to loss of the extracted material because of a flicking motion of the needle at the moment of grain release. In addition, it was cumbersome to have to adjust three separate controls to transport extracted grains to a desired location.

(b) Micromanipulators which have two controls, one of which governs movement in two of the three orthogonal directions. Devices of this type are more convenient to use than those of type (a) [see Buchtal and Persson (1936)], but trials with one instrument revealed little improvement in "feel sensitivity."

(c) Micromanipulators which permit a probe to be moved by a single control in any desired direction in space. De Fonbrune (1932) designed a pneumatic instrument with an X–Y "joystick" control which can be rotated to obtain motion along the Z axis. A currently manufactured model is too sensitive for mineralogical work, permitting only 2.5 mm of probe-tip movement and a range of 40:1 to 2000:1 for the ratio of control-lever motion to probe motion.

Barer and Saunders-Singer (1948) were the first to propose a mechanical pantograph design which they

[1] Present address: School of Applied Geology, University of New South Wales, Australia

subsequently developed and described (1951). It has the following attributes which make it a very useful mineralogical tool: (a) linear motion in any spatial direction; (b) separate twisting motion of the point of the needle probe in a circle of small radius; (c) arrestment of the needle in any spatial position without subsequent creep under gravitational force; (d) scale reduction of hand motion in a ratio of 5:1, with consequent similar reduction of uncontrollable random hand motion from 100 μm to about 20 μm (average); (e) appreciable "feel sensitivity"; (f) either right- or left-hand operation.

The instrument that is used by the author is the Oxford model made by Micro-Techniques (Oxford) Ltd., U.K.[2] (Fig. 1) which embodies a few minor improvements on the original design of Barer and Saunders-Singer (1951). This micromanipulator can be fitted with many types of probe holders, but these have been designed for life scientists who commonly manipulate their soft specimens with glass probes which can be drawn to points as small as 0.05 μm diameter. However, the mineralogist must use a more robust metal probe and manufacturers of micromanipulators could offer nothing suitable.

Sewing needles are too flexible and inadequately sharp (size 12 had tip diameters *ca.* 24 μm), and dental seekers although more rigid were still too blunt. The tubular form of hypodermic needles gives reasonable rigidity, and size no. 30 have tip diameters usually < 10 μm [finer needles (higher size numbers) are not often used in the medical and dental professions but can be ordered from manufacturers]. No. 30 needles are easily sharpened to point diameters of about 5 μm, using either chromic oxide on a rotating

[2] Available in U.K. from Micro Techniques (Oxford) Ltd., 7 Little Clarendon St., Oxford, and in Australia from Wild (Australia) Pty. Ltd., P.O. Box 21, North Ryde, N.S.W. 2113, Australia.

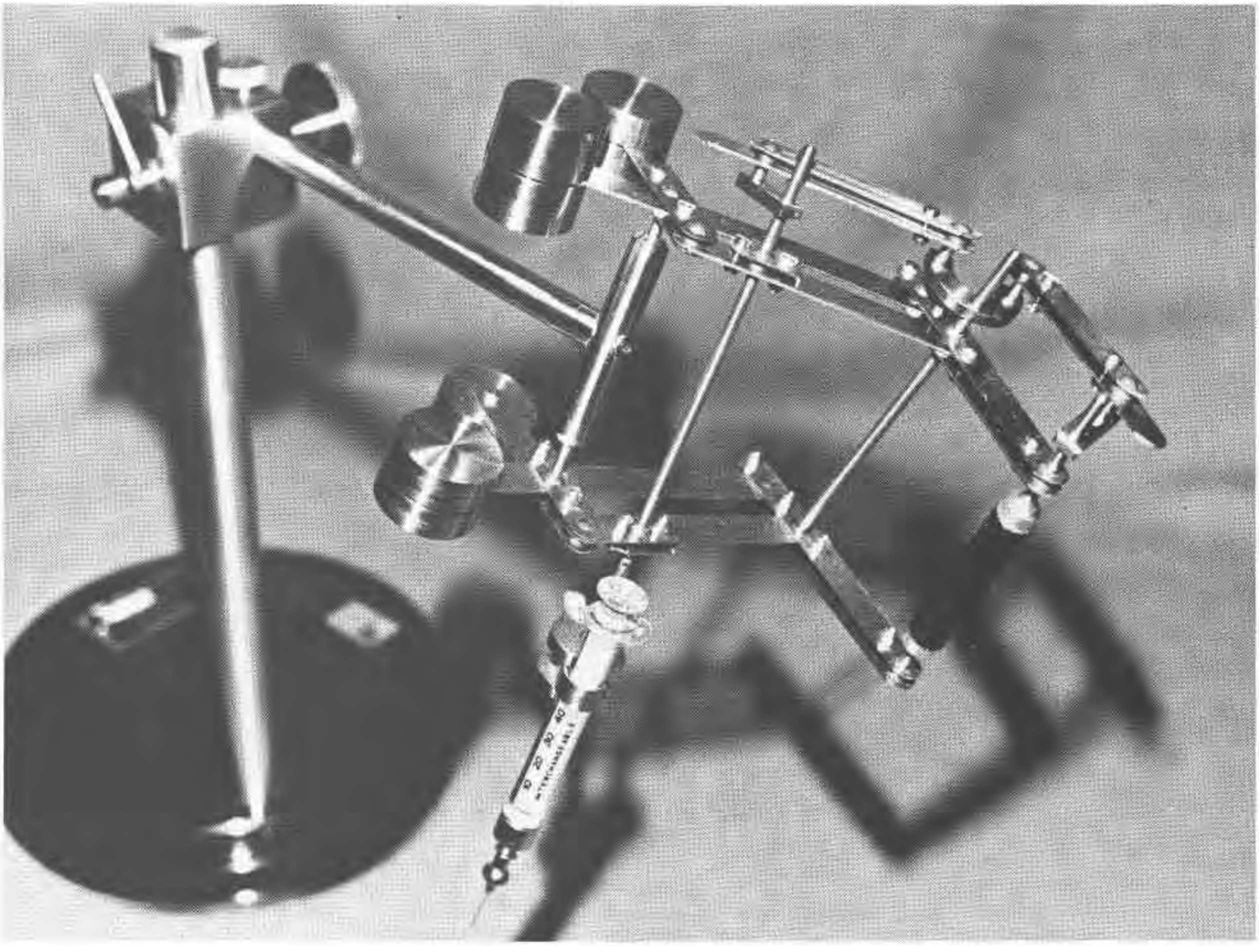

Fig. 1. Pantograph micromanipulator with 1 ml hypodermic syringe (calibrations are insulin units) All movement made by gripping the grooved black handle, at the extreme right, is duplicated by the needle point at one-fifth scale.

metallurgical polishing disc or by honing on an oiled carborundum stone, and when attached to a syringe of ≤ 5 cm^3 volume, they can be readily fitted to the micromanipulator. With this equipment it is not difficult to extract grains of 20 μm diameter, and persons with a steady hand have found 10 μm quite possible. Often the grain breaks during extraction and affords opportunity to select a much smaller portion, free of particles of neighboring minerals.

Small indentations made around the desired crystal assist in its removal, and softening of the thin section adhesive with a solvent (Wallace, 1955) was not found to be necessary, nor is it desirable to so damage the slide. Nevertheless, the force required to remove the grain may still cause the needle to bend, but with this instrument one can feel when the grain is about to come free and can then lessen the applied force, thereby largely preventing flicking. The surface tension of a small amount of moisture can be advantageously exploited to help retain the extracted grain, and McCrone and Delly (1973) even advocated use of a drop of collodion/amyl acetate solution.

As an alternative to a simple needle probe, Gray (1968 a, b) described the use of an ultrasonic (29 khz magnetostrictor) chisel. However, 15 μm diameter was the smallest grain that he mentioned as being extractable from metals, and it has not been possible to examine this device.

An X-ray method for identifying single uncrushed grains using a Debye-Scherrer camera has been described by Kittrick and Hope (1967), but a Gandolfi camera is more convenient. However, crushed material is most commonly used, and techniques described by Sorem (1960), using a gelatin fiber, and by Hiemstra (1956), using a rubber ball, are noteworthy for the small amount of material that they require. The latter procedure is recommended, but whereas Hiemstra advocated crushing the grain before the solvent evaporated, it has proved better to wait, for capillarity causes the mixture to be spread over too great an area. Extracted fragments > 10 μm diameter can be readily prepared with this technique, and the 5 μm minimum size claimed by McCrone and Delly (1974) is quite within the scope of some people. Spherical quartz grains of these sizes weigh only 1.4×10^{-9}g and 1.7×10^{-10}g respectively, much less than the 3-4 $\times 10^{-6}$g that Donnay (1946) used.

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