An ultrasonic probe for the extraction of microscopic quantities of minerals for X-ray work

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Abstract

The instrumentation and procedure is described for the extraction of very small and pure samples from polished mounts and thin sections of minerals, for X-ray analysis and other micro-analytical techniques. It uses a stand-mounted, tunable ultrasonic probe (400 W/15–50 kHz) inclined at 45° to a microscope stage. The tip of the probe, usually a steel needle, is located on the desired mineral and an appropriate ultrasonic frequency is selected. Very fine powders are produced from grains as small as 0.01–0.005 mm. A distinct advantage of this technique is that, as different minerals powder at different rates and different frequencies, it is possible to separate a single mineral from an intergrowth of two or more minerals.

KEYWORDS: X-ray analysis, Debye Scherrer, ultrasonic probe, mineral separation.

THE extraction of very small quantities of a mineral before identification or analysis by X-ray using the Debye Scherrer powder camera has always been somewhat difficult and something of an art. Now that the electron microprobe has become so widely used to determine the composition of minute grains and intergrowths of minerals the need for a reliable extraction method for confirmatory diagnosis by X-ray diffraction is even more important. Probably the most widely used manual method for extraction is similar to the one described by Hiemstra (1956). This uses a steel needle, scalpel, or diamond-tipped needle to loosen and pre-grind the mineral which is then ground more completely and mixed with an X-ray amorphous gum to form a ball. Other procedures which have been advocated include the use of microhardness testers (Berkovich and Kuritzina, 1949; Lebedeva *et al.*, 1965), and a compound microscope objective with a steel needle (Juschko *et al.*, 1975) to fracture and break the sample. All of these require the separated sample to be ground before mounting and, perhaps more importantly, require that the operator be thoroughly trained in the use of the instrument as well as taking great care in its application.

These problems may be overcome by using an instrument first described by Kovachev and Strashimirov (1979) which uses an ultrasonic probe to prepare an optimal amount of material for examination. Its advantages may be summarized as follows:

a) very precise location of the sampling point to an area certainly as small as a needle point;

b) the need for additional grinding of the sample is eliminated;

c) it becomes possible to extract selectively one of a number of minerals present in an intergrown assemblage, thus providing a very pure sample;

d) the preparation and setting up of the probe is quick, and the procedure is automatic once the equipment is set up.

In essence, the instrument consists of a needle of steel or some other hard material which is set under ultrasonic oscillation, the tip of which fragments or powders the sample which can then be combined with gum to prepare the X-ray mount. The frequency of the ultrasonic oscillations is 15–50 kHz, and this may be tuned to optimise the extraction for different species. Depending on the physical properties of the mineral, the minimum size of grain from which material may be successfully extracted is in the range 0.01 mm to 0.005 mm. In terms of the minimum quantity sufficient for a satisfactory Debye Scherrer powder pattern, Genkin and Korolev (1961) established that

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for galena this would be about 0.003 mg; in terms of volume, a cube supplying this mass would have an edge of 0.035 mm. The ultrasonic probe has the major advantage over microhardness testers in that, when powdering the sample, it is not restricted to surface destruction (Lebedeva *et al.*, 1965, having shown that, to achieve a satisfactory sample, indentations of 0.02 to 0.07 mm are required). Of course, even with the ultrasonic probe, where the grain size of a single grain is too small, powder from two or more grains of the same species may be combined.

In the event that this still provides too little material for a complete powder produced by the probe, a sufficient number of the main reflections are often obtained with which to identify the mineral.

The equipment (Fig. 1) which we use consists of an ultrasonic generator (400 W/15–60 kHz); an emitter with a specially designed tip and a stand to hold, guide and orientate the probe (allowing several degrees of movement). Obviously, sufficient free space must be available between the microscope objective and the sample to permit the introduction of the probe and to site it accurately. This means that long working distance objectives are essential. The tip of the probe/emitter is positioned such that the angle between the tip and specimen surface is about 45° (adjusted by a micrometer on the stand). A certain amount of experimenting is generally required to find the frequency of ultrasonic oscillation most appropriate for the mineral. In operation, the needle produces a small crater in the sample around which the mineral powder collects. The steel needle does not contribute any significant contamination to the powder extracted from the minerals. No specific lines of Fe were found in the patterns checked. Gum may be added to this powder on completion of the operation, or a small drop may be put on the tip of the needle before the generator is switched on. The mineral powder combines with the gum and the ball produced is ready to be mounted for X-ray work. There are no special requirements for sample preparation from thin sections (of course they should be without cover glass on them), or from a block, with or without a flat surface, to be put on the microscope stage.

The method could be used also for extracting individual minerals for isotope analysis. In this case no gum should be added and the collection of the material should be done by the use of a small brush or in any other suitable manner. If the surface is cleared before the operation no contamination of the probe can be expected.

The very short time (10–15 sec) for the destruction of the material could hardly affect the health of the operator but ear defenders should be used, especially if there are any special requirements for work with ultrasonic devices in the country in question. In any case, if a high degree of probing is expected, the specimen should be fixed on the microscope stage and the operators should not handle the specimen by hand, so that the vibrations will not affect their health.

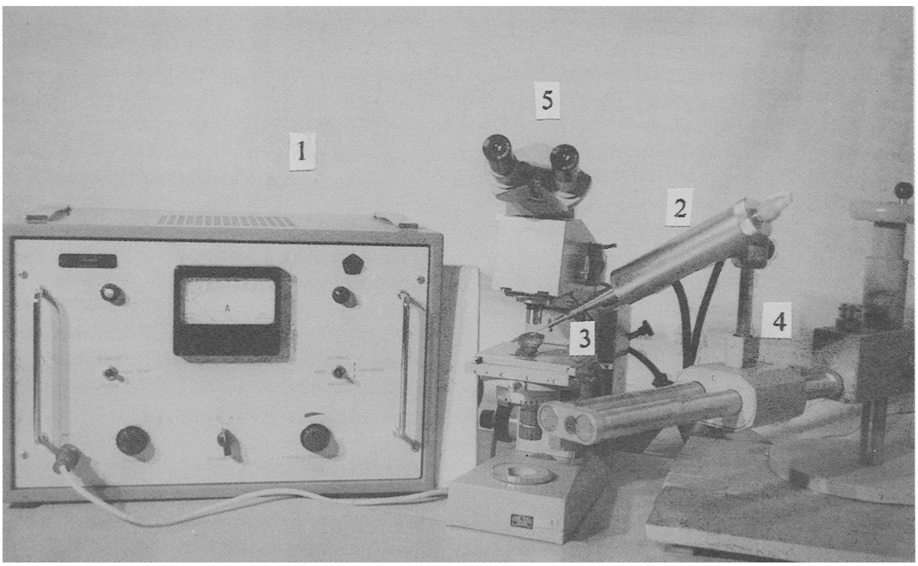

FIG 1. The equipment used by the authors: 1. Ultrasonic generator, 2. Ultrasonic emitter, 3. Probe or working tip, 4. Stand, 5. Microscope.

As noted earlier, a major advantage of this technique derives from the fact that different minerals disintegrate and powder at different ultrasonic frequencies; thus it is often possible to separate a pure powder of a single species from an intergrowth of two or more minerals. This feature renders the technique suitable for the separation of samples for micro-analytical techniques other than Debye Scherrer.

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