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A microscope-mounted drill to isolate microgram quantities of mineral material from thin and polished sections

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SUMMARY. Several types of microscope-mounted drills have been described in literature to obtain pure mineral material from thin sections or polished sections (e.g. Granigg, 1915; Moritz, 1929; Haycock, 1931; Russanow, 1937; Machairas, 1967), but all of them appear to be of limited mechanical stability and reliability. This paper describes an improved drill to extract under the microscope minute quantities of pure material from thin or polished sections. The drilling is performed in a liquid (silicon-oil) to minimize loss of the drilled-out mineral powder. With the help of a micromanipulator the liquid, together with the borings, is drawn up into a microcapillary tube (less than 30 μm in diameter) made of Lindemann glass. For identification of the mineral powder the microcapillary is mounted in a Debye-Scherrer X-ray diffraction camera.

Description. The instrument (fig. 1) consists of an optical and a mechanical part that can both be attached to a polarization microscope (fig. 2). The optical part is a short cylinder (fig. 3.1) provided with screw-thread at either end. It can be mounted between a centrable objective holder[1] of the microscope and an objective with a large clearance (e.g. Carl Zeiss, Oberkochen, UD 16 ×, 0·17 N.A. or UD 40 ×, 0·65 N.A.; fig. 3.2). The mechanical part of the drill consists of a basal plate (fig. 3.3 and 4) carrying a motor unit (fig. 3.5), which can be fastened on to the cylinder. A ring (fig. 3.6) has been machined out in the basal plate in such a way that it remains connected with the plate by a narrow 'bridge' of metal (fig. 3.7) and fits around the double-threaded cylinder. The motor unit consists of two parts: the body and the house of the motor. The cylindrically shaped motor house contains a small 3 V, DC electromotor (fig. 3.8) connected to a shaft with a pin-vice (fig. 3.9). The pin-vice accepts exchangeable drill rods (fig. 3.10) with a diameter of 2·5 mm

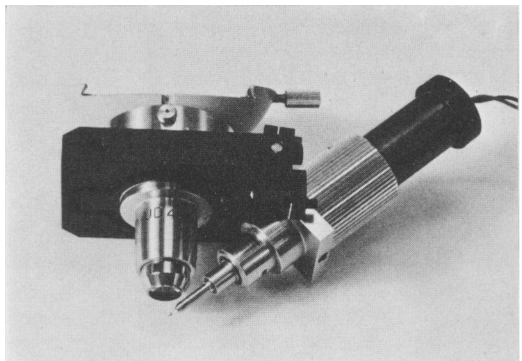

FIG. 1. Micro-drill mounted on a centrable holder for single objectives. A drill rod furnished with a polished monocrystalline diamond tip is in the pin-vice of the drill.

furnished with monocrystalline diamond tips,[2] polished to sharp faceted points with a top angle of about 40°. The complete drill rod is 20 mm in length. The axle of the motor (fig. 3.11) is connected to the drill shaft by a short length of rubber tubing (fig. 3.12) to ensure vibrationless operation of the drill. The shaft of the drill is locked in a ball-bearing (fig. 3.13). The house of the motor unit can be moved with regard to the body of the motor unit along the axis of rotation of the motor by means of a low-speed screw system (fig. 3.14). This is done by turning the milled cylinder at the outside of the motor house. The motor unit is connected with the basal plate by means of two pivots (fig. 3.15) in the body of the motor unit that fit into notches in the basal plate. This enables the motor unit to tilt in a plane containing the optical axis of the micro-

[1] For transmitted light or both transmitted and reflected light.

[2] To drill material with a Mohs hardness less than 7, a pointed all-metal drill rod made of hardened steel may be used instead.

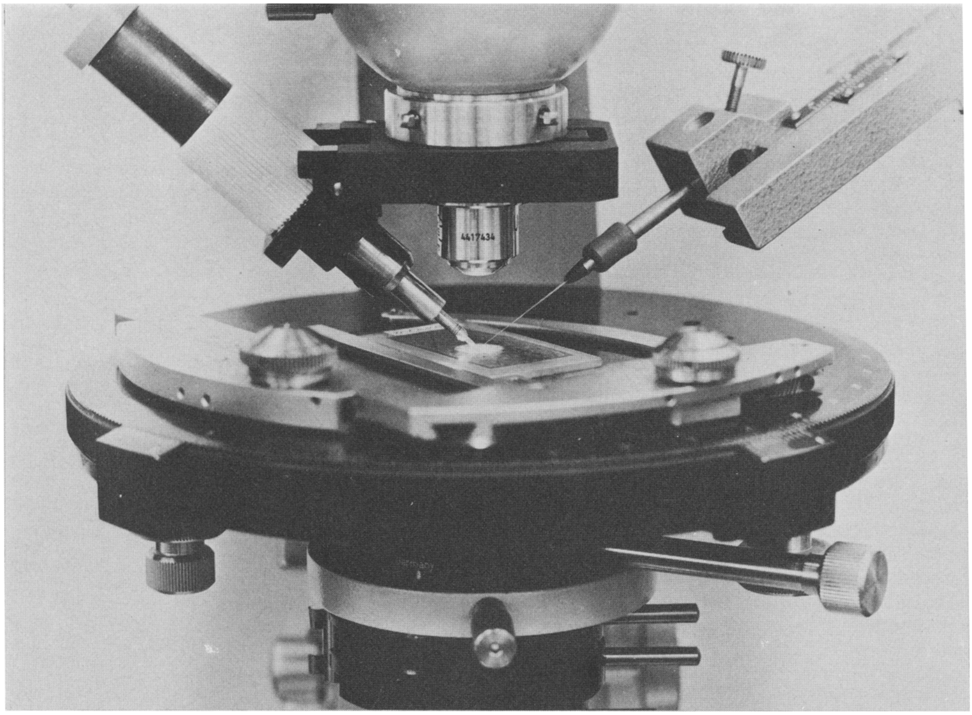

FIG. 2. Detail of the micro-drill during a drilling operation. Rotary stage with mechanical stage holding a polished thin section, objective holder with micro-drill, micromanipulator holding metal tube with rubber mouthpiece. In the mouthpiece the hollow cylindrical X-ray camera mounting plinth with microcapillary can be seen.

scope. The motor unit is fixed at an angle of about 45° towards the microscope stage. A small alidade (fig. 3.16 and 17) enables minor tilt adjustments. It is possible to centre the intersection of the axis of rotation of the drill in the centre of the focal plane of the microscope. Adjustment in one direction is performed by tilting the motor unit, and adjustment perpendicular to this direction by rotating the basal plate around the centre of the metal bridge (fig. 3.7) in the plate. This rotation can be effected by the operation of a system of two aligning bolts in the basal plate (fig. 3.18). Beforehand the microscope has to be centred optically. The drilling is started by lowering the motor unit along the direction of the rotation axis of the drill, until the point of the drill is in contact with the surface of the section. The actual drilling can be done either by further downward displacement of the motor unit, or by using the focal adjustment system of the microscope itself. When crystals smaller than 50 μm in diameter are drilled, the radius of the rotational eccentricity of the point of the diamond has to be less than 5 μm. For larger crystals, eccentricities up to 20 μm are desirable for a better chipping action. It must be stressed that the point of the diamond is vulnerable and is easily broken off when accidentally forced down into an object. If the drill is used with care, the diamond tips of drill rods with small eccentricities show little wear even after prolonged use, also when drilling hard minerals such as topaz and corundum. Drilling of glass, however, should be avoided; this material tends to slow down rotation due to its toughness and may tear off the point of the diamond.

Mode of operation. The cover glass of the thin section has to be removed before drilling. This is easily done by cooling the slides below 0 °C (deep freezing compartment of a refrigerator) and inserting a thin knife or razor blade under one of the edges of the cover glass. Removal of the cover glass cement is attained with a suitable solvent. The best results are obtained by drilling in a liquid medium, as then practically no material is lost by scattering. A low-viscosity silicon-oil is recommended. This oil has a low surface tension and tends to form a 'planparallel' layer on the surface of the section, minimizing distortion of the optical image. A sufficient quantity of liquid is deposited upon the area to be drilled. For the collection of the liquid-plus-borings a microcapillary tube of Lindemann

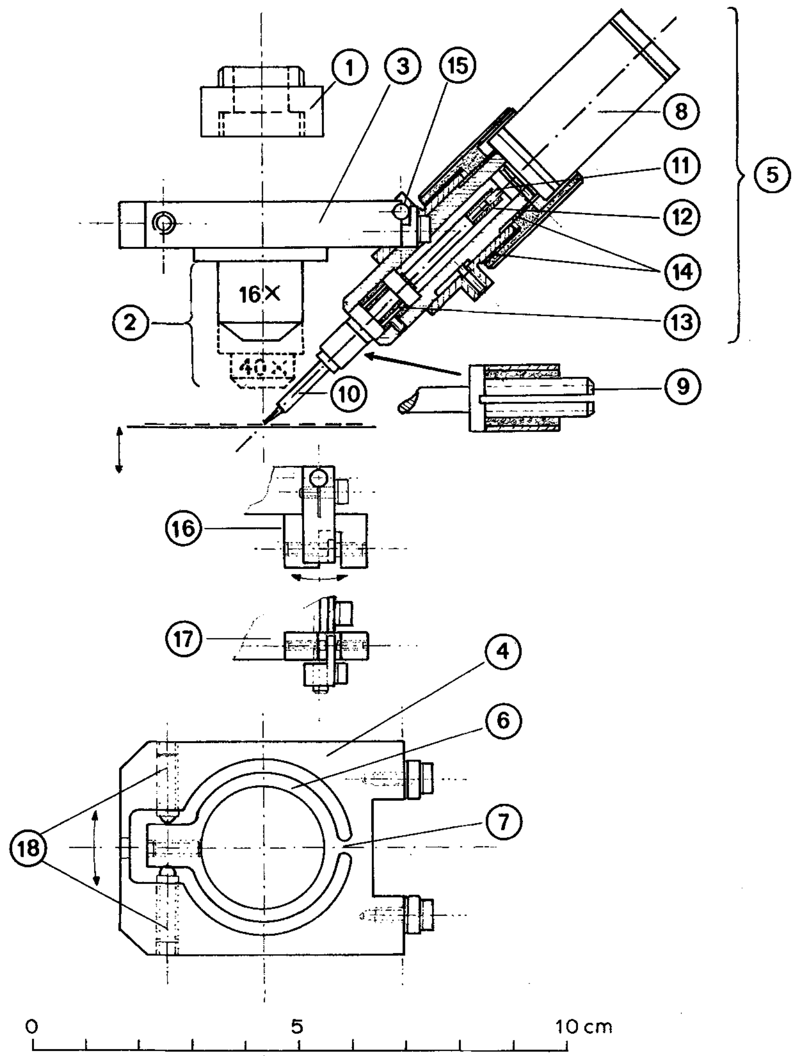

FIG. 3. Dimension sketch of the micro-drill. 1, Cylinder; 2, Objective (either 16 ×, or 40 ×); 3, Basal plate (from side); 4, Basal plate (from above); 5, Motor unit; 6, Inner ring; 7, 'Metal bridge', connection between inner ring and basal plate; 8, Electrical motor; 9, Pin-vice (detail); 10, Drill rod with diamond tip in pin-vice; 11, Motor axle; 12, Rubber tube; 13, Ball-bearing; 14, Low-speed screw system; 15, Pivot; 16, Alidade (from side); 17, Alidade (from above); 18, Aligning bolts.

glass is used. The preparation and operation of such microcapillaries will be described in a separate section of this paper.

Before drilling, the place of impact of the tip of the drill is determined by gently lowering the motor unit (with turning motor) along the axis of rotation of the drill. The motor unit is then raised again and the rotary table of the microscope arrested. The selected area of the section is covered with an ample amount of liquid. This will be described later. Subsequently the motor unit with turning motor is lowered again cautiously. First it can be observed that the point of the diamond passes through the meniscus of the liquid layer, after which a faint scratching noise signals the contact of the diamond with the surface of the section. A small groove then begins to form. For further drilling the fine focal adjustment system of the microscope is used. It is profitable to ascertain in advance the correspondence of the dimensions of the conical drill hole that is formed, with the depth of drilling indicated by the number of scale divisions of the focal fine adjustment knob of the microscope. This helps to avoid 'overdrilling', i.e. the contamination of the borings with borings of another mineral in the thin section. In the case of drilling of irregularly shaped areas, the mechanical stage can be used in combination with a drill rod having a rotational eccentricity up to 20 μm of the diamond tip. After completing the drilling operation the motor unit is raised and the drill rod removed from the pin-vice. Finally, the liquid with the borings is collected by carefully dipping the tip of an empty microcapillary into the 'cloud' of borings in the liquid. Capillary action draws the liquid together with the borings into the microcapillary tube. The collection of the borings in a microcapillary can be done also when the drill is still turning. In that case the microcapillary is brought into the eddy around the tip of the drill. In the eddy the borings are 'kept together' and can be drawn into the microcapillary in a more concentrated form.

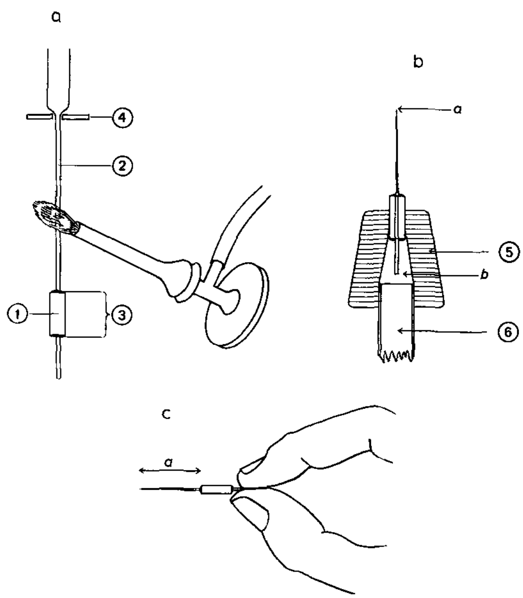

FIG. 4. Details of the preparation and operation of microcapillary tubes. *a*. Glass capillary (2) placed in the slot of a metal plate (4). The capillary is furnished with a hollow, cylindrical mounting plinth (1) secured at both ends (3) with modelling clay. The capillary is heated with a small gasburner until the glass weakens and the capillary draws out to a gradually narrowing microcapillary by the weight of the plinth. The plinth has to be caught on a soft 'bed' consisting of a piece of cotton wool to prevent the breaking of the capillary. *b*. Section of the microcapillary in the plinth cut at '*a*' and '*b*'. The plinth is held in the rubber mouth (5) at the end of the metal tube (6). *c*. Retraction of the microcapillary in the specimen mounting plinth to the exact length '*a*' enabling the focusing of the liquid-plus-borings in the centre of the X-ray beam. The modelling clay at both ends of the plinth has then to be moulded again around the glass. Finally, the wider end of the capillary has to be broken off to enable the insertion of the plinth in the central axial holder of the camera.

If dry-drilling is preferred, several techniques have been described to collect the borings (e.g. Hiemstra, 1956; Azàroff and Buerger, 1958; Sorem, 1960; van der Veen, 1963). Instead of a solution of natural rubber (latex) in benzene to collect material a rubber solution in a mixture of gasoline and kerosene may be used. This solution remains sticky for a longer period so that one can collect powder from several drilling sites.

Applications and results. Besides obtaining monocrystalline powder from minerals in thin or polished sections for identification by X-ray diffraction, the drill can be used to obtain pure mineral material for other purposes, e.g. chemical analysis or determination of refractive indices. In this case discs of mineral material are drilled out by using a decentred drill and turning the rotary stage of the microscope. The refractive indices may also be determined *in situ* with the usual calibrated refractive index liquids, after drilling a hole with a wedge-like edge through the mineral.

For mineral identification by X-ray diffraction the microcapillary containing the fluid-plus-borings is inserted directly into the camera. It was found that for many minerals quantities as small as 0·3 μg of material produce sufficiently observable diffraction lines on the X-ray film to enable identification (applied exposure times: 40 hours using the 114·6 mm diameter camera and 20 hours using the 57·3 mm diameter camera). Background darkening of the films caused by air-scattering was reduced by inserting in the collimator a pin-hole diaphragm with a diameter of 0·2 mm.

Preparation and operation of microcapillaries. The hollow, cylindrical plinth (fig. 4.1) for specimen mounting of the Debye-Scherrer X-ray powder diffraction camera is fastened around a conventional capillary (fig. 4.2) for X-ray powder diffraction work (e.g. Philips PW 1951, 0·3 mm diameter). The plinth at both ends of the capillary has to be secured carefully with modelling clay (fig. 4.3), so that it cannot slip off. The wider neck of the capillary is placed in the slot of a small metal plate held in a laboratory support (fig. 4.4). A small gasburner is used for gentle heating of the capillary. When the glass of the capillary softens the tube is drawn out to a gradually narrowing microcapillary by the weight of the plinth. The plinth has to be caught on a soft surface (e.g. cotton wool) so that the closed part of the capillary is not damaged. The thin, drawn-out part of the capillary is cut free with a pair of scissors. The closed end of the capillary protruding from the plinth is also cut off in such a way that the remainder of it can still be held between the fingertips (fig. 4.4). The plinth with the microcapillary is connected to the rubber mouthpiece (fig. 4.5) at one end of a metal tube held in a micromanipulator (fig. 4.6). This is a delicate operation because the end of the plinth from which the wider side of the capillary protrudes has to be inserted in the rubber mouth without breaking. The other end of the capillary, i.e. the gradually narrowing microcapillary part, is brought into the field of view of the microscope with the micromanipulator and cut at the desired diameter (about 30 μm) with a pair of scissors. The other end of the metal tube held in the micromanipulator is furnished with a long rubber tube. By gently blowing into the tube the microcapillary, filled with liquid, can be emptied at the desired spot on the section. Another way to cover the spot with liquid is to touch it with a needle with a blunt tip wetted with liquid. After drilling and collecting the liquid-plus-borings in the microcapillary by capillary action, the plinth with the microcapillary has to be removed carefully from the rubber mouthpiece. The thin part of the microcapillary is so far retracted into the plinth that the cylinder of liquid-plus-borings is located at the focal point of the X-ray beam, when the plinth is mounted in the central axial holder of the camera. This is effected by pulling the wider end of the capillary protruding from the plinth carefully with the fingertips (fig. 4.4). Care should be taken that the distance between the centre of the liquid in the microcapillary and the end of the plinth is adjusted to the exact length (*a*). The capillary should be secured again with the modelling clay before the wider end of the capillary (the handling end) is broken off and adjustments are no longer possible. The plinth with the filled microcapillary can then be mounted in the camera. Some focusing in horizontal direction is still possible by the final positioning of the plinth in the central axial holder of the camera with the help of a pair of tweezers. In vertical direction the focusing of the capillary is performed in the usual way by the centring device of the camera.

An alternative method to collect the borings is to prepare in advance a specimen mounting plinth with an empty microcapillary of the right diameter and length without handling end.

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