

Preparing oriented single-crystal sections for measurements of internal morphology

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Abstract. Random crystal sections are not suitable for measurements permitting to derive quantitative data on the kinematics and kinetics of crystallization processes from the sector zoning patterns exposing the internal morphology of crystals. The preparations needed should be perpendicular to well developed zones in the crystal, and their number should be sufficient for reaching statistically valid results. For the purpose, a procedure is described which uses a two-circle reflection goniometer for orienting the crystal to be studied (larger than about one millimeter), and several simple auxiliary tools for transferring and fixing it onto a planar support with the aid of which oriented one-sided or two-sided polished sections are made by conventional methods. Preliminary drawings of sections with the SHAPE computer program are very helpful for reaching the desired depth of sectioning. The procedure is illustrated with preparations of clinopyroxene phenocrysts 1 to 2 mm in size. About 40 thin sections perpendicular to two zones, 0.05 to 0.10 mm thick, have been made. The accuracy of the final section orientation is within 1°. The procedure has been also used for orienting laboratory grown crystals of centimeter size to be used for seeds in crystal growth experiments and for measuring their physical characteristics.

Key words: crystallography, internal morphology, sector zoning, section preparation
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Случайни прерези на кристалите не са подходящи за измерванията, които позволяват от секторната зоналност, разкриваща вътрешната морфология на кристалите, да се получат количествени данни за кинематиката и кинетиката на кристализационните процеси. Необходими са препарати, ориентирани перпендикулярно на добре развити зони в кристала, при това в достатъчен брой, за да има статистически значими резултати. Описаната за тази цел методика използва двукръжен отражателен гониометър за ориентиране на изучаваните кристали (по-големи от около един милиметър) и набор от прости допълнителни инструменти за прехвърляне и фиксиране на получената ориентация върху подложка, с помощта на която от ориентирания кристал чрез стандартни методи се изработват едностранно или двустранно полирани препарати. Предварителното чертане на сеченията с компютърната програма SHAPE е удобно помощно средство за достигане на подходяща дълбочина на пререзите. Методиката е илюстрирана с изработването на ориентирани препарати от порфирен клинопироксен с преобладаващи размери 1-2 mm. Направени са около 40 препарата, ориентирани по две зони, с дебелина 0,05—0,10 mm. Точността на ориентирането е в рамките на 1°. Методиката е използвана и за ориентиране на лабораторно получени кристали със сантиметрови размери за зародиши в опити по израстване на кристали и за препарати за измерване на физични характеристики.

Ключови думи: кристалометрия, вътрешна морфология, секторна зоналност, препарати
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Introduction

In the study of internal morphology, commonly used for qualitative reconstruction of growth histories of minerals (e.g. S u n a g a w a, 1987, 1994), a crystal is treated as consisting of the growth pyramids of its external surfaces coming out of the original crystal nucleus. The pyramid sides are formed by the crystal edges moving outwards parallel to themselves during growth. Unlike external shapes, internal morphologies are studied in sections in which the growth pyramids are visualized by a number of imaging techniques as sector zoned patterns that show the traces of pyramid side surfaces as linear boundaries dividing sectors of concentric zoning which outlines past shapes of the crystal. L e m m l e i n (1948) showed that internal morphologies can be measured to gain information about the evolution of growth rate ratios of crystal surfaces, i.e. to collect quantitative data on the kinematics of crystallization. G r a y (1971) went further on by attempting to model the kinetics of formation of an hourglass structure in titanite on the basis of its parabolic shape. Although the great potential of internal morphology as a source of quantitative information about crystallization phenomena in nature has been thus demonstrated, little advantage has been taken of the opportunity by researchers in the field so far. Variability of crystal shapes during growth, shown so well by V a v r a (1990, 1993) in his systematic studies on the kinematics of accessory zircon growth, and the many factors, which have to be assessed in interpreting a sector-zoned pattern (V e s s e l i n o v, K e r e s t e d j i a n, 1995), require measurement and comparison of many patterns in a crystal population to reach statistically significant results. Random cuts, such as those in thin and polished sections, are of little use in an internal morphological study because the true angles between a sector boundary and the adjacent zones are seen only in sections perpendicular to the edge which have produced that boundary (L e m m l e i n, 1948). Besides, other growth-related phenomena (compositional changes, distribution and regular orientation of inclusions, regular orientations of sector boundaries, etc.), clearly manifested in oriented sections (e.g. B o n e v, 1974 a,b; V e s s e l i n o v, 1994; V e s s e l i n o v, K e r e s t e d j i a n, 1995), may remain hidden in random ones. It is also seen that generally more than one orientation in a crystal is to be studied in order to observe and measure the ratios between all forms or faces. All that requires preparation of many oriented sections if one is to get the full benefit of internal morphological patterns. So far this preparative problem has not been solved satisfactorily and studies are based either on suitably oriented cuts in random sections (e.g. G r a y, 1971) or on crystals mounted and cut parallel to the best developed faces or zones (e.g. K a s t n e r, W a l d b a u m, 1968; V a v r a, 1990; V e s s e l i n o v, K e r e s t e d j i a n, 1995). This paper describes a procedure for preparing all needed oriented sections of crystals of millimeter size (larger crystals can be oriented by simpler methods which need not be described, and smaller ones become progressively difficult to orient and process). It is based on standard techniques and instruments supplemented by a few simple auxiliary tools.

Procedure for preparing oriented crystal sections

Briefly, a crystal is oriented on a goniometer along a chosen zone axis, then it is transferred and fixed onto a planar support with this axis in perpendicular position, and ground and polished parallel to the support to a depth exposing the sector-zoned pattern perpendicular to the zone. The procedure includes the following steps.

1. *Selecting crystals from a population.* The available crystals are divided as usual into groups of similar form combinations, habits, sizes, or other relevant features, and enough specimens of well-defined edges are selected for orientation. Crystals smaller than one millimeter are very difficult to section. Those larger than about two millimeters can be cut in parts to make sections of different orientations of one and the same crystal (as in the example given further on) and to measure all elements of its internal morphology. The wire saw proposed recently (S e r g e e v et al., 1997) seems a useful device for the purpose.

2. *Selecting zone axes.* It is only in rare cases of simple morphologies that sections in a single orientation pass through all growth pyramids of a crystal. To minimize preparative work in crystals of richer form combinations the zone axes for orientation should be carefully chosen. By examining external shapes, and thin and polished sections, if available, one decides on the number of zones both necessary and sufficient for exposing all growth pyramids in perpendicular cross-sections (see example). The use of the crystal-drawing computer program SHAPE 5.0 for Windows (D o w t y, 1995) is highly recommended at this stage. After drawing a crystal, the program can section it at any depth in any desired orientation providing a preliminary picture of what might be expected during further work.

3. *Orienting a crystal.* It is made by the classical methods of reflection goniometry which need not to be described here. It should be noted only that a two-circle rather than one-circle instrument should be preferred because it permits full examination of the external morphology parallel with orienting the crystal thus providing additional useful information. The usual margin of error in orienting crystals along a zone is a few minutes of arc.

4. *Transferring an oriented crystal to a support.* This is the most delicate operation in the whole procedure and the accuracy of the final orientation of a section depends practically entirely on it. It needs a few auxiliary tools described in the next section along with some directions for use. After finishing this operation, the crystal is fixed, ideally, with the chosen zone axis exactly perpendicular to the plane of the support.

5. *Grinding and polishing a section.* Standard techniques for preparing wafers are used. Excessive grinding may ruin the oriented specimen by overshooting the desired depth of section. This is avoided by frequent checking the section outlines under a stereomicroscope comparing them with the SHAPE drawings made at the beginning. Both one-sided and double-sided (see example) polished sections can be made.

Auxiliary tools for making preparations

The simple tools shown in Fig. 1 can be made in any mechanical workshop with sufficient precision. Their dimensions depend mainly on the goniometer head used. Those given here are made for a head about 70 mm long and 45 mm in diameter.

The metal plate (75 mm in diameter, Fig. 1*a*) is made with a central perpendicular female thread fitting the male one by which the goniometer head is fastened to the goniometer axis. The tool in Fig. 1*b* consists of a bolt piece (30 mm high, 30 mm in diameter) with a slot for a screwdriver, and a nut piece (20 mm high), with three perpendicular bores (5 mm), drilled at radii of 25 mm, into which three removable rods 95 mm long (Fig. 1*c*) can be fixed. It is convenient to thread the nut-and-bolt screw with a step of 1 mm. Fig. 1*d* shows a plastic disk 1 mm thick and 20 mm in diameter with two bored receptacles for millimeter-size crystals. Such disks are made by casting cold-setting resins (e.g. acrylic, polyester) in suitable molds.

PLATE I

1. A SEM photograph of a typical clinopyroxene crystal, oriented and sectioned as shown in Fig. 2*b*. Bar 1 mm
2. Thin section of 1. perpendicular to [101] (cf. Fig. 2*c*), transmitted light. Bar 1 mm
3. Thin section of 1. perpendicular to [001] (cf. Fig. 2*c*), transmitted light. Bar 1 mm

ТАБЛИЦА I

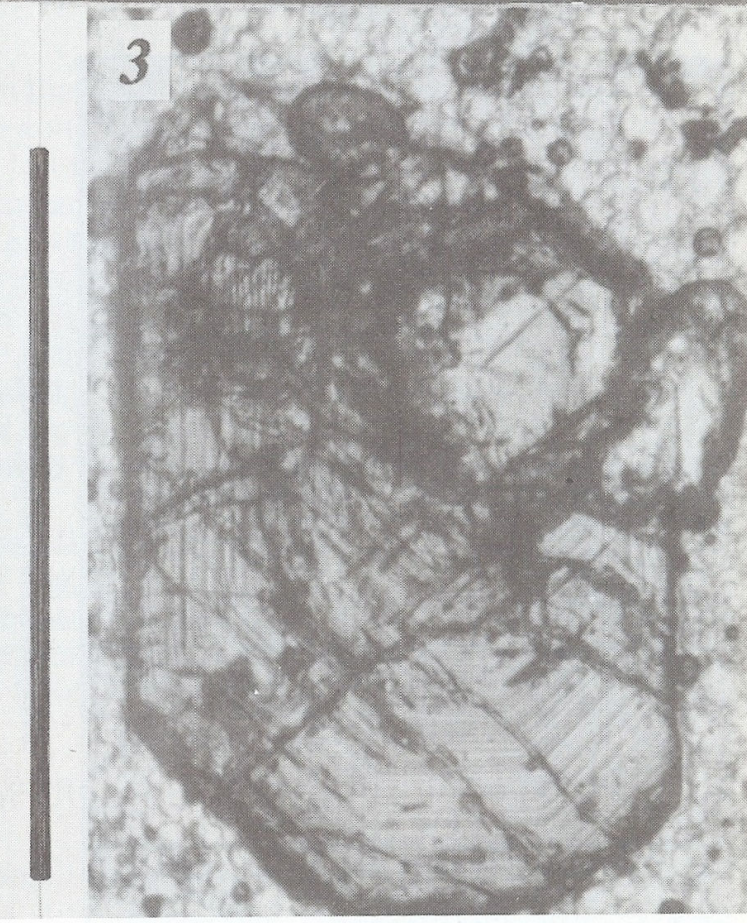
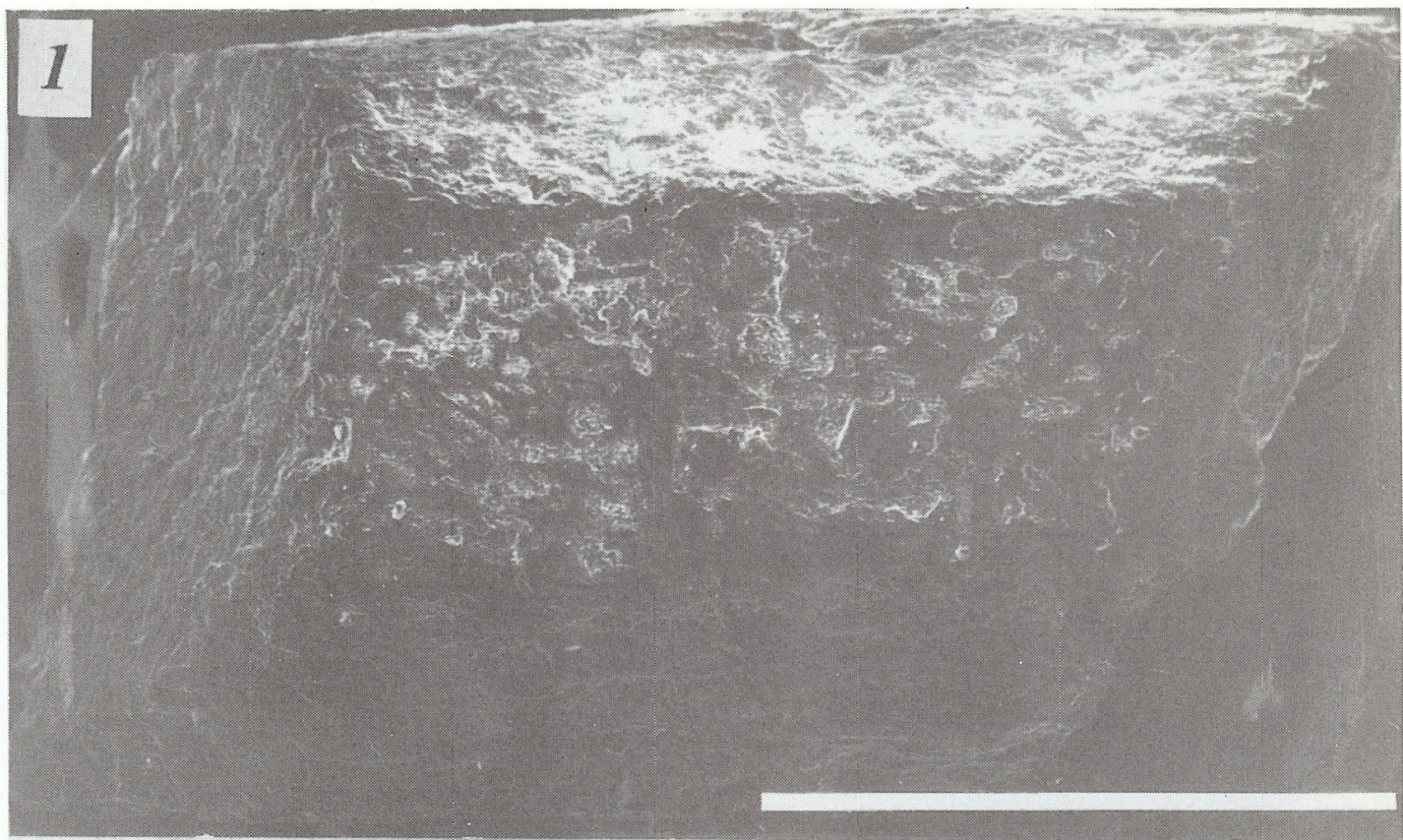
1. СЕМ снимка на типичен клинопироксенов кристал, ориентиран и срязан, както е показано на фиг. 2 *b*. Мерна отсечка 1 mm
2. Дюншлиф от 1., перпендикулярен на [101] (срв. фиг. 2*c*), проходяща светлина. Мерна отсечка 1 mm
3. Дюншлиф от 1., перпендикулярен на [001] (срв. фиг. 2*c*), проходяща светлина. Мерна отсечка 1 mm

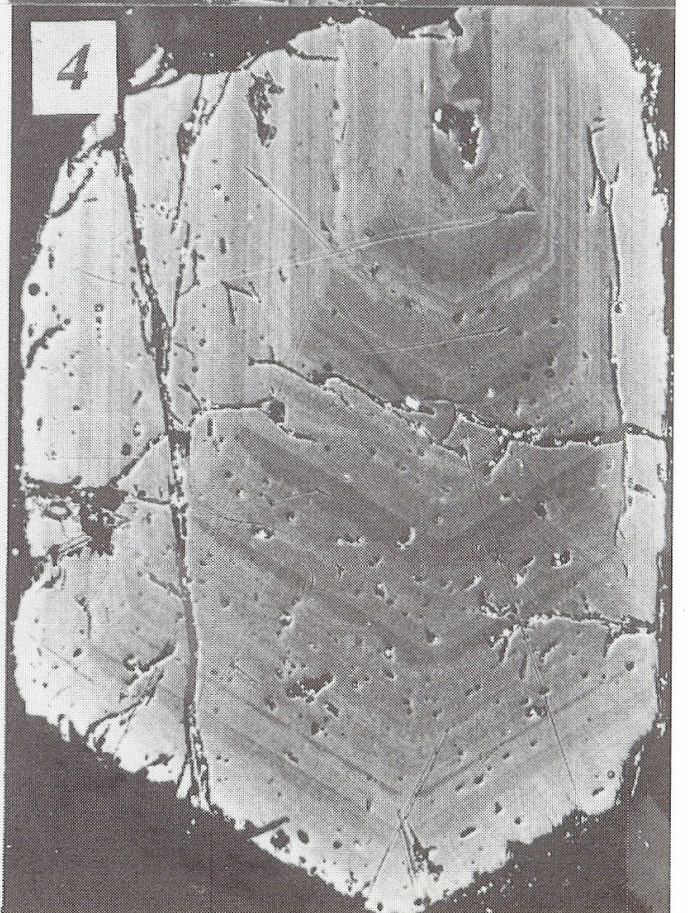
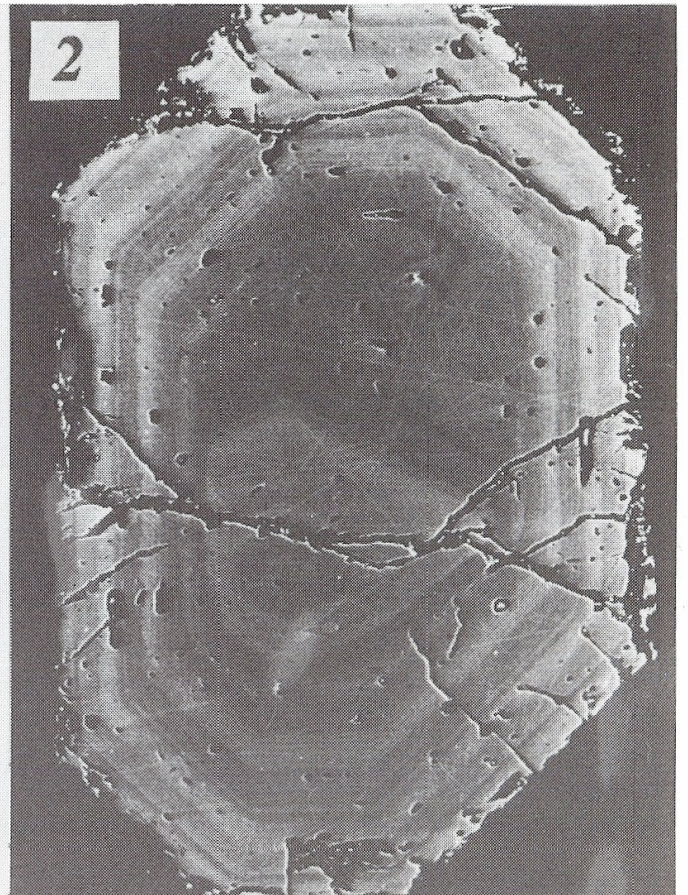
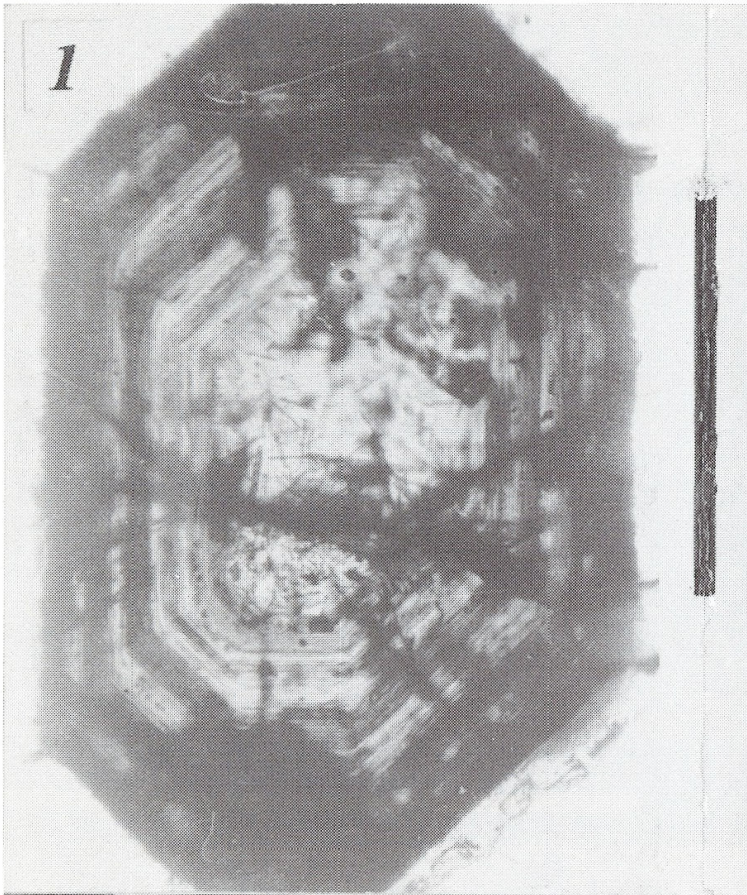
PLATE II

1. Concentric zoning in a [001] thin section, transmitted light. Bar 0.5 mm
2. Back-scattered electron image of 1
3. Concentric zoning and sector pattern in a [101] thin section, transmitted light, crossed polarizers. Bar 0.5 mm
4. Back-scattered electron image of 3

ТАБЛИЦА II

1. Концентрична зоналност в дюншлиф, перпендикулярен на [001]; проходяща светлина. Мерна отсечка 0,5 mm
2. Снимка на 1. в обратно отразени електрони
3. Концентрична зоналност и сектори в дюншлиф, перпендикулярен на [101]; проходяща светлина, кръстосани николи. Мерна отсечка 0,5 mm
4. Снимка на 3. в обратно отразени електрони





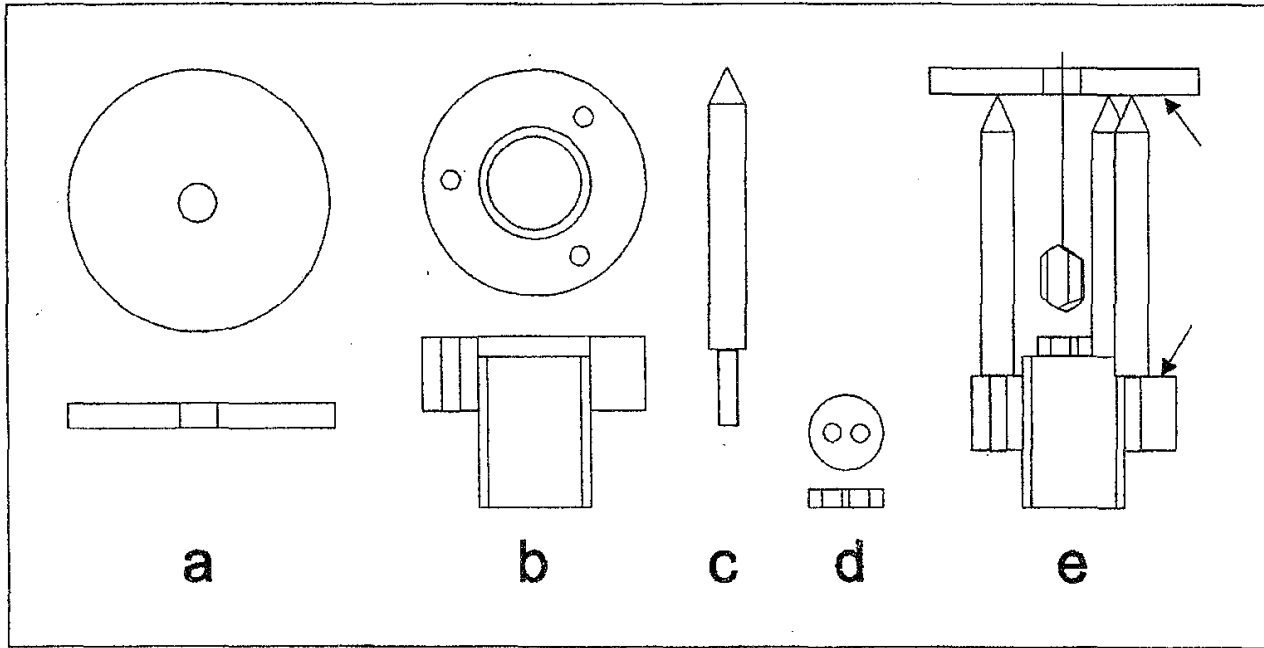


Fig. 1. Auxiliary tools for making preparations: *a* — plate (plane and side views); *b* — brass nut-and-bolt tool (plane and side views); *c* — rod; *d* — mounting disk; *e* — the tools assembled for use; the oriented zone axis of a crystal (exaggerated) is perpendicular to the planes marked by arrows

Фиг. 1. Помощни инструменти за изготвяне на ориентирани препарати: *a* — плочка; *b* — месингов инструмент тип болт и гайка; *c* — щифт; *d* — кристалодържател; *e* — цялото устройство за прехвърляне на зададената ориентация; зоналната ос на кристала (увеличен) е перпендикулярна на означените със стрелки равнини

The oriented crystal is removed from the instrument together with the goniometer head by carefully unscrewing it from the goniometer axis and then fastening it on the plate. Now the latter's plane is parallel to the plane along which the crystal is to be sectioned. Then the auxiliary tools are assembled as shown in Fig. 1*e* after adjusting the distance between the bolt and the plate to accommodate the goniometer head with the crystal. A drop of cold-setting resin is put into the hole of the mounting disk and the crystal is inserted into it by carefully turning the bolt piece by a screwdriver. After allowing the resin to set, the plate with the goniometer head and the three rods are removed, the bolt with the disk and crystal is adjusted in the nut piece at the necessary depth and the preparation is ground parallel to the nut plane to fix the section surface. After that the preparation can be safely transferred to a wafering instrument for further processing.

The three rods which secure the parallelism between the plate and nut planes make the handling of the assembly in Fig. 1*e* somewhat unwieldy. A convenient alternative is a vertical stand with a support for the plate mounted parallel to the stand base on a rack-and-pinion mechanism by the screw of which the oriented crystal can be gently and conveniently lowered into the mounting disk receptacle.

Example: oriented sections of clinopyroxene

Clinopyroxene phenocrysts from a small basalt body exhibit sector zoning patterns suitable for an internal morphological study. The crystals are of uniform augite morphology with $\{100\}$, $\{010\}$, $\{110\}$ and $\{\bar{1}11\}$ (Fig. 2*a*, Pl. I.1), and range in habit from isometric to short prismatic along $[001]$, the predominant size range being 1 to

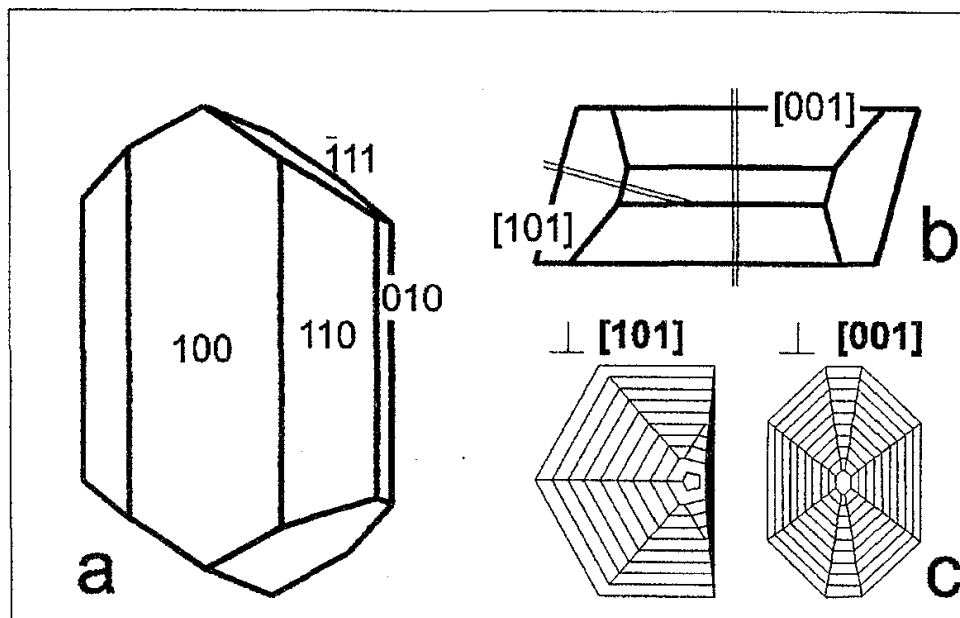


Fig. 2. SHAPE drawings of an augite crystal: *a* — perspective view; *b* — view along the two-fold axis with the two chosen sections; *c* — views of the sections perpendicular to the zone axes
 Фиг. 2. Авгитов кристал, представен с програмата SHAPE: *a* — коса проекция; *b* — проекция по двойната ос с двата избрани пререза; *c* — проекции на пререзите по зоналните направления

2 mm. Sections perpendicular to two zones are necessary and sufficient to expose all growth pyramids in this morphology — [001] for {100}, {010} and {110}, and [101] for {010} and {111} (Fig. 2*b,c*). Both sections pass through {010} so that all growth rate ratios to be derived by measurements can be conveniently expressed with the rate of {010} as a common denominator.

Rock specimens were crushed, screened and about 40 crystals with well defined edges were selected for preparation. Equal number of each type of section was made from different crystals mostly (Pl. II.1,2,3,4). Four longer crystals, 1 by 2 mm, were cut in two and sections perpendicular to [001] and [101] were made (Fig. 2*b,c*; Pl. I.1,2,3). Crystals were fixed by three or four in a single disk, sections were ground to thicknesses between 0.05 and 0.10 mm to permit observation of bulk phenomena, and were polished on both sides.

The interfacial angles measured on the sections made (such as those shown in Pl. I and II) deviate not more than one degree of arc from their values in the ideal drawings (Fig. 2*c*). Only little experience is needed to achieve this accuracy of oriented sectioning which is comparable to angle measurements on the microscopic stage.

Conclusion

The proposed procedure broadens the area of the crystallography of external shapes over the internal morphology of crystals, i.e. any goniometric study can be extended to cover the growth pyramids of external forms as well. Measurements of the exposed sector zoning patterns can be made directly on the microscopic stage or, even better, on suitably enlarged photographs. Although the high accuracy of initial orientation by the goniometer cannot be saved during the subsequent operations of transferring and polishing the crystals, the deviations are within the margin of error of the microscopic stage. With some instruction and experience, the preparation of dozens of sections can be made a routine procedure providing material for systematic

studies both of the kinematics and kinetics of growth of mineral crystals by combining the modern imaging techniques of internal inhomogeneities with accurate measurements of growth rate ratios and computer modelling of mineral formation phenomena. The same procedure and instruments have been also used by the author in laboratory growth for orienting seeds for crystal-pulling experiments (lead molybdate) and sections for testing physical characteristics of as-grown crystals (yttrium iron garnet, barium borate, strontium chloride).

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