Natural Diamond Drawing Dies with <111> Crystal Orientation

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It became known at the very latest when grinding diamonds for jewellery in 15th century Renaissance workshops, in order to impart greater brilliance, that the hardness of a diamond depends on the orientation.

There is proof that the difference in the hardness of diamonds has been used for industrial applications, including wire drawing, by means of defined orientation since the end of the 19th century. This subject was revitalised by Messrs. Sumitomo Electric in 1987, when they introduced the first ever synthetically manufactured monocrystalline diamonds with <111> crystal orientation on the market for industrial purposes.

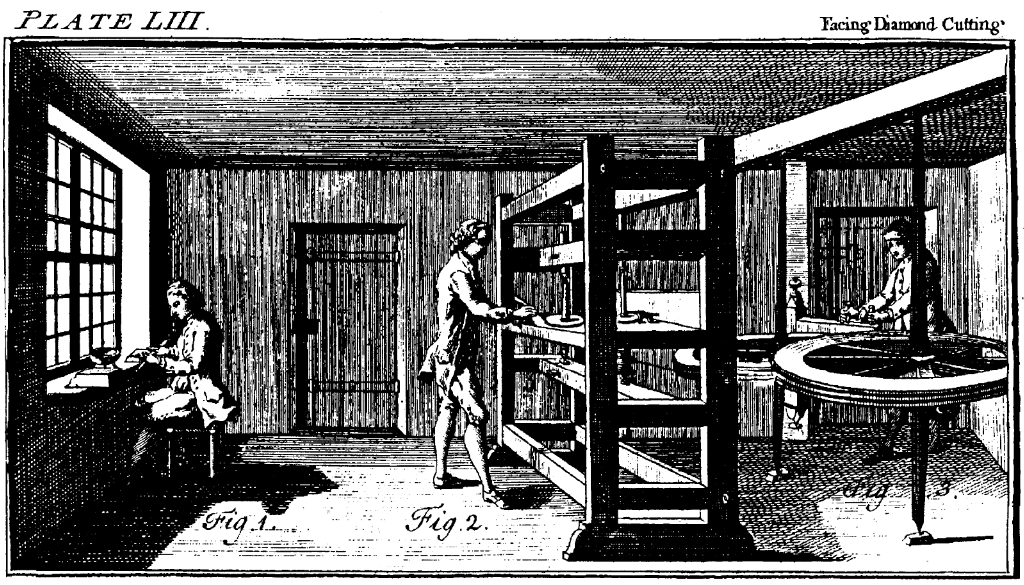
Since these new materials became known, UKD Ulrich Kiwus Diamanttechnik has performed extensive tests in its capacity as a manufacturer of drawing dies, particularly with steel wire drawers, as results became apparent relatively soon in the case of hard materials. At the same time, the methods of grinding natural diamond were systematised and natural diamond drawing dies with various orientations tested.

Since 1992, we at UKD have progressively changed to locating the bore in the drawing die geometry in natural diamonds as closely as possible in the crystal orientation corresponding to Miller's indices <111> (so-called three-point orientation). This yielded the product **UKD Natural <111>.** Experience with this product over the past three years shows the following:

- Better roundness of the drawing dies throughout their service life

- Consequent increase in the service life of the drawing die sets, particularly in the case of precisely graduated sets manufactured by the wire elongation method (see our "Drawing Die Sets" brochure)

- Resultant improved utilisation of raw materials as regards cost-effectiveness and ecological aspects.



1. Diamond materials for drawing dies

Polycrystalline materials for drawing dies have been known since the early 70s. These materials are produced from diamond powder by pressure sintering processes, yielding products which are known in the trade as "synthetic diamonds", "artificial stones" or COMPAX (tradename of Messrs. General Electric). Other tradenames are SYNDIE (De Beers) and SUMIDIA (Sumitomo Electric).

As already mentioned, synthetic monocrystals in the millimetre range have been available as industrial raw materials since 1987. These are synthesised from graphite at temperatures in excess of 1,300 °C and pressures of 50,000 bar. On account of the high thermal conductivity of diamond, these materials were initially developed as heat sinks (cooling components) for semiconductor technology, but manufacturers soon put them to use as base materials for drawing dies. Tradenames are SUMICRYSTAL (Sumitomo Electric) and MONODIE (De Beers).

Thus, the term "synthetic diamond" is no longer clear, and a more apt distinction is made between *polycrystalline* and *monocrystalline* diamond. There is a considerable difference between these materials as regards their structure, wear performance and processing.

In contrast, the difference between natural and synthetic monocrystals is less distinct, although not without significance.

This brochure explains the various crystal orientations of monocrystalline diamond, and provides a comparison of natural and synthetic monocrystals as raw materials for drawing dies.

2. Orientation and wear behaviour

To start with, a look at the essential characteristics of diamond monocrystals:

- Homogeneous material (single-phase, uniform material structure independent of direction)
- Crystallisation in the symmetry of the isometric system
- Isotropic (direction-independent) properties: density, colour
- Anisotropic properties: hardness, thermal conductivity, optical properties.

Hardness is taken to mean the resistance to wear. We shall give this property our attention to start with.

Figure 2 shows the most important forms of the regular diamond crystal system. These are: cubes, rhombododecahedrons and octahedrons. Diamond crystallises in the isometric system with 90° angles. The forms actually found in nature are shown on the right in Fig. 2 according to this classification.

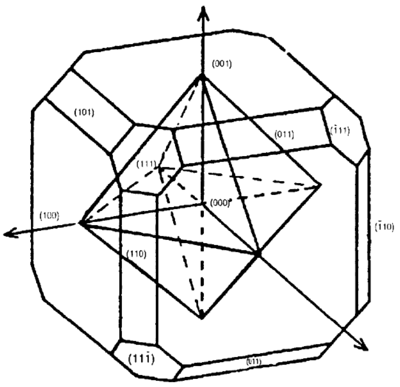
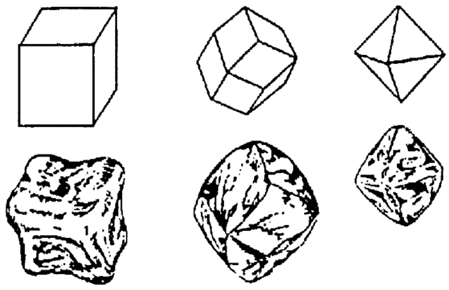


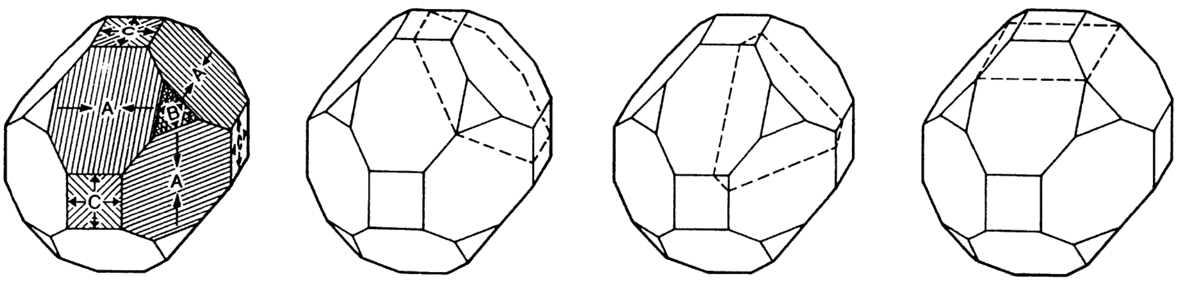
Fig. 2 Octahedron with its basic facets



(100) (110) (111)
Crystal surfaces of the planes

Figure 3 shows which basic facets can be ground on diamond crystals. Plane-parallel facets are ground on diamonds for drawing dies for the following reasons:

- Selection of the bore direction following thorough inspection of the direction of growth of the crystal
- Better fixation in the drawing die mount and avoidance of "tilting" when sintering the diamond into the mount
- Defined wall thickness in the inlet area and resultant lower risk of the drawing die fracturing
- Avoidance of ovalities, which are almost impossible to prevent when drilling the natural, uneven facets.



Basic facets (110) or 2-point (111) or 3-point (100) or 4-point

Fig. 3 Basic facets

After grinding sufficiently large facets perpendicular to the main directions of the diamond crystal, the drawing die is given its geometry (Figs. 4 to 6). Miller's indices are used in order to indicate the position of the facets, these being customary in crystallography. In order to simplify matters, only the three main types are taken into account: cubic, dodecahedral and octahedral facets. These possess the lowest crystallographic indices.

<100>

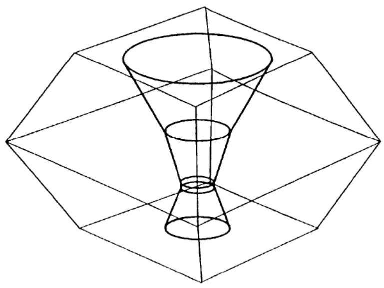


Fig. 4
Bore perpendicular to ground octahedral peak

<110>

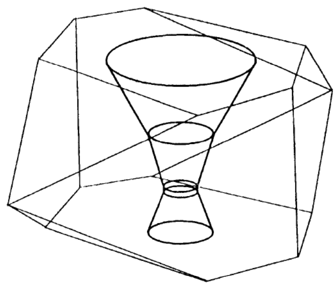


Fig. 5
Bore perpendicular to ground octahedral edge

<111>

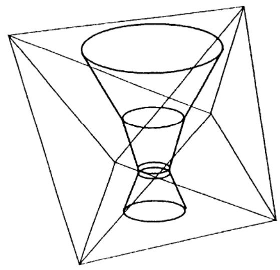


Fig. 6
Bore in octahedral crystal perpendicular to (111) plane

In the past, a host of wear investigations have been conducted on diamond crystals such as penetration and grinding tests, in particular, but also abrasion tests (see references). The following conclusions can be drawn from these tests for the manufacture of drawing dies:

- The wear factor between the hardest and the softest facet orientation of a diamond is roughly 1:9. This is a considerable difference, and thus an indication that attention must be paid to the number of hard orientations cut by the wall of the bore in the drawing die.

- The most favourable orientation for drawing dies is a bore in the <111> direction, followed by <110>.

- A bore direction of <100> is the least favourable.

Figures 7 and 8 show the wear for bore directions of <110> and <111>, both schematically and on the basis of real drawing dies displaying signs of wear (photographs of the interference rings generated by laser light). If you explore the peripheral line of the bore in Fig. 7, you find 4 hard zones, as opposed to 6 in Fig. 8. The hexagonal arrangement of the "supporting points" in the case of drawing dies with <111> orientation results in more uniform wear, thus leading to a lesser tendency towards ovality and, consequently, a longer service life.

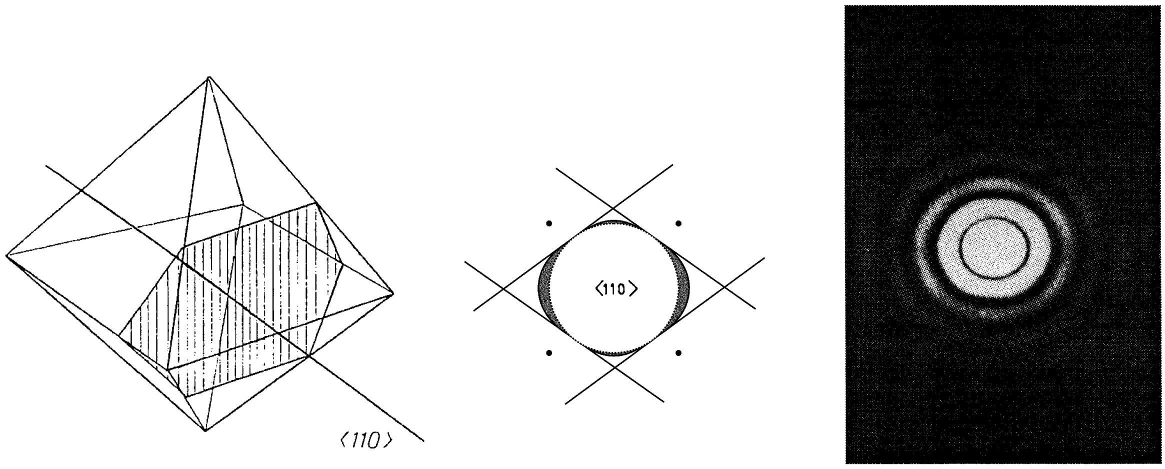


Fig. 7 Direction of bore in octahedral crystal <110>

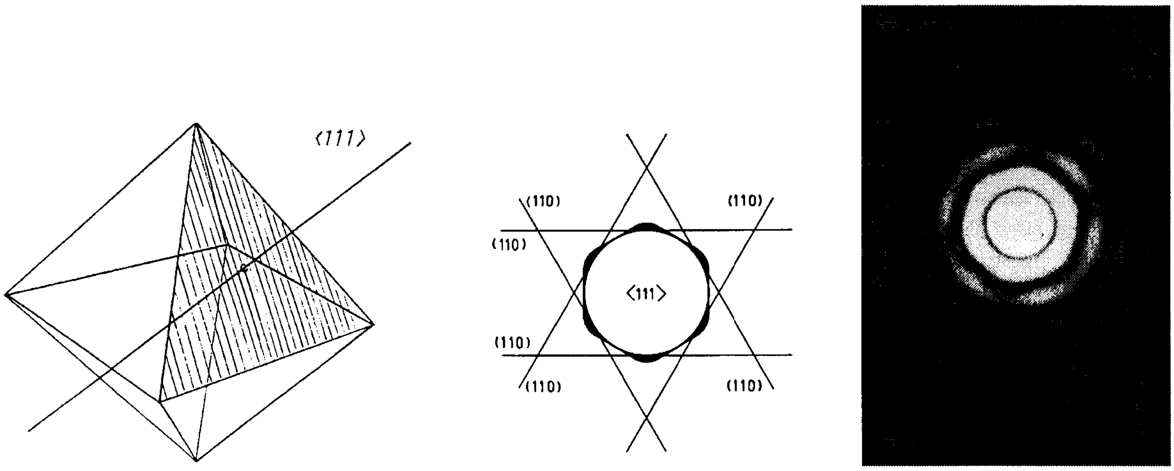


Fig. 8 Direction of bore in octahedral crystal <111>

A bore direction of <111> is preferable for the manufacture of drawing dies.

3. Synthetic monocrystals and their crystallographic orientation

Without doubt, the manufacture of synthetic diamonds is an impressive scientific and technical achievement in the field of applied thermodynamics. The dream of alchemists became reality, albeit not with gold, but with diamond.
Following laboratory-scale developments since the 60s, monocrystals in a size range of 0.5 to 1.5 mm became available for industrial use at the end of the 80s.

Initially, synthetic monocrystals with <100> crystal orientation were also offered as drawing die blanks. It was already known from theory that drawing dies with <100> orientation have relatively unfavourable wear properties. In the case of natural diamond, the grinding-off of the octahedral peaks (four-point grind) is uneconomical, because the grinding wheels are more likely to be damaged than the diamond actually cut.

As early as when processing synthetic diamonds with a bore direction of <100>, the impression arose that the material was "softer" than natural diamond. When used, the drawing dies were soon subject to excessive wear and became unround relatively quickly.

More success was experienced with synthetic diamond crystals with <111> orientation (Fig. 9). However, in the opinion of a number of our customers, particularly steel wire drawers, they still fail to achieve the merits of natural diamond, particularly when taking the overall lifetime of the drawing dies into consideration (after repolishing).

This could be due to the following causes:
The yellow-brownish colour of synthetic monocrystals is attributable to nitrogen inclusions and possibly to catalyst residues. Synthesis can only be performed with an economically viable production time by adding metallic catalysts. As far as we are aware, no data are available concerning the extent to which catalyst residues lead to disruption of the crystal lattice and thus to a possible impairment of the hardness of the diamond. It is, however, known that increasing concentrations of nitrogen are associated with a reduction in hardness.

The MONODIES shown in Fig. 9 are 0.8 mm high. MONODIES are offered in a product range with crystal heights of up to 1.5 mm, SUMICRYSTALS up to 2.0 mm. Thus, the largest drawing dies which can be made from synthetic monocrystals cannot have a diameter greater than roughly 1.0 mm in new condition. In contrast, UKD offers drawing dies made of natural diamond up to a diameter of 3.0 mm.

4. Natural monocrystals and their orientation

Figure 10 shows a batch of unprocessed natural diamonds in the size range of 15 to 20 stones per carat (1 carat = 0.2 gram), such as are customarily used for drawing dies with a diameter of 0.2 mm. At UKD, these goods are "filtered out" of the lots available on the market by means of a multi-stage selection process. Purchasing only takes place after this selection process, with unsuitable goods being rejected. The selection criteria are as follows (using 40 x magnification microscopy):

- Freedom from inclusions and microscopic cracks
- Suitable shape for grinding the parallel facets with <111> orientation
- Surface extensively free from chipping and cracks
- Absence of sharp stress profiles (polarisation microscopy).

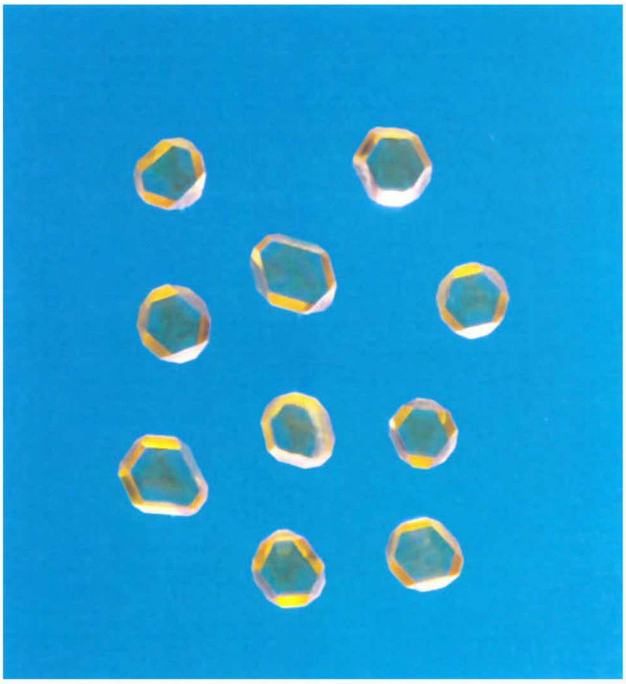


Fig.9: Monodies 111

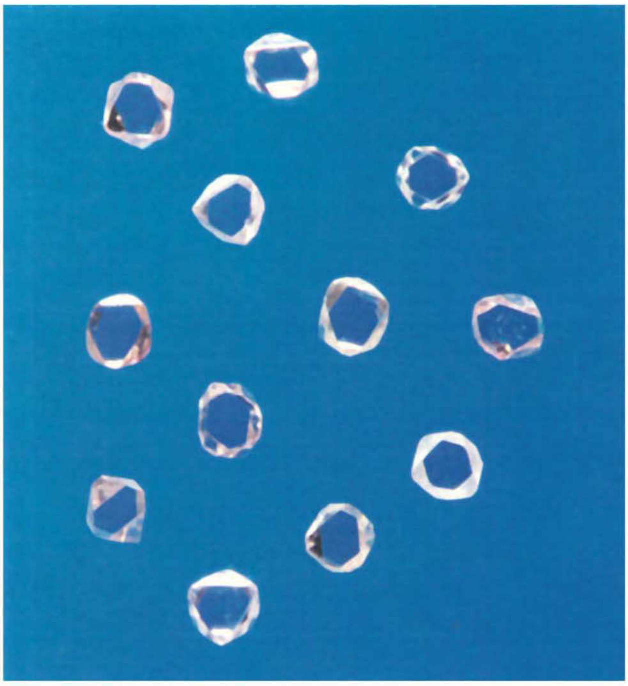


Fig. 11: Natur <111>

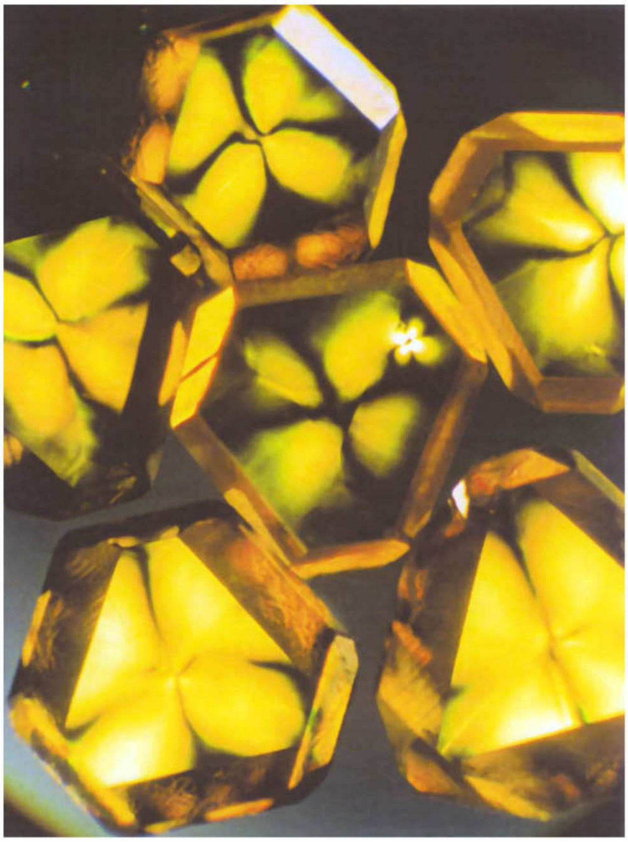


Fig.13: Monodies 111 (polarizing microscope)

Fig. 14: Natur <111> (polarizing microscope)

The process of diamond selection is given the highest possible attention by us at UKD. In this context, we safeguard the interests of our customers by adhering to the adage: rule out a problem by not buying it in the first place!

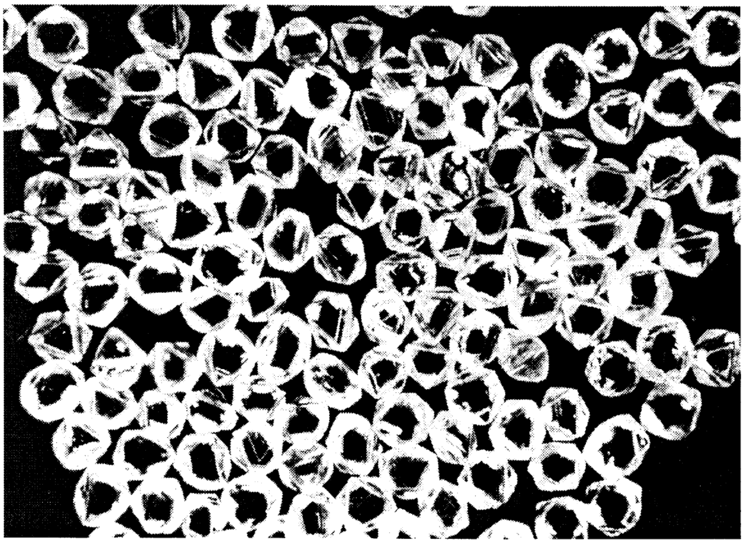


Fig. 10

It is immediately noticeable that the diamonds in Fig. 10 are considerably whiter than synthetic monocrystals, meaning that there are fewer inclusions of foreign atoms.

Yellow, or slightly brown natural diamonds are used for larger drawing dies, because uncut stones which are also suitable for use in jewellery cannot be purchased for price reasons.

In terms of internal purity, the natural diamond raw material selected by UKD has advantages over synthetic monocrystals.

The ground octahedral triangular facets are readily identifiable on the natural diamonds in Fig. 11. The skilled diamond grinder, who can cut a full 57 facets on a crystal with a size of 50 stones per carat, can identify the "growth" - in other words, the crystal orientation - of a diamond crystal with a high degree of accuracy.

In the case of diamond crystals of the size shown in Fig. 11, we are able to achieve mean deviations of approximately 1 degree on the ground facets, and maximum deviations of approximately 3 degrees from perpendicular in the <111> orientation. These values were determined by investigations conducted using the Laue method in crystallographic laboratories (Figs. 12.1 and 12.2).

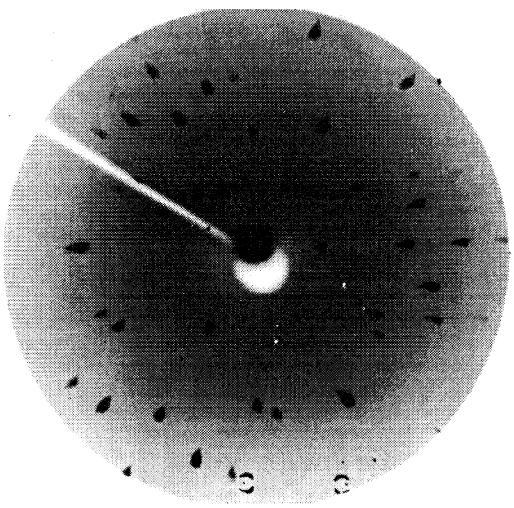


Fig. 12.1
Laue photo of the ground (111) facet

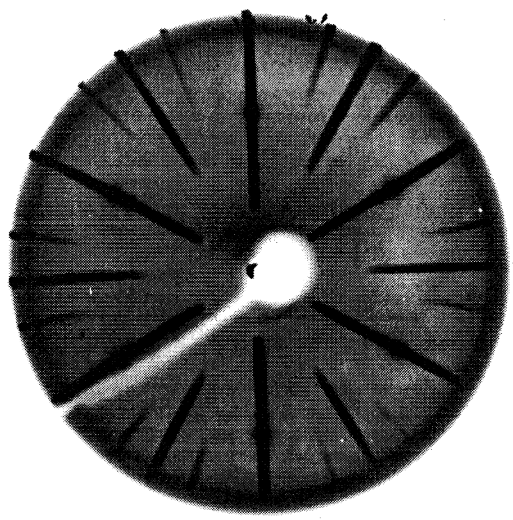


Fig. 12.2
Precession photo of same
(moving Laue photo)

However, creating facets perpendicular to the <111> orientation (three-point grind; the diamond grinder sees three edges or "points", see Fig. 6, on the ground facet) entails a greater amount of work in comparison with the customary method of grinding perpendicular to the <110> orientation (two-point grind, see Fig. 5). Because a natural edge against which the diamond can be ground (the lateral edge of the octahedron) is already available in the case of two-point grinding, this facet can be ground with a single tool setting.

In contrast, the triangular facet cannot be ground directly using three-point grinding. The stone "has no purchase", as a diamond-grinder would say. An auxiliary facet has to be ground first, at a certain angle to the triangular facet. Subsequently, the grinding tool can be aligned parallel to the triangular facet and grinding can be performed against the previously created edge. This procedure requires the full attention of a well trained grinder, and is naturally more time-consuming.

Automatic grinding machines, which provide a host of diamonds with parallel facets on both sides at the same time, cannot be used for three-point grinding. It is also questionable whether stones coming back from an external diamond-grinder working on a contract basis (payment by number of stones) - as is customary with many drawing die manufacturers (if parallel facets are even ground at all!) - would have a three-point grind applied with adequate care.

At UKD, this grinding work is performed exclusively on our premises, under the supervision of an experienced diamond-grinder.
Young grinders are carefully introduced to the skills of this trade, which has almost died out in central Europe. Digital measuring techniques are employed to maintain constant angles during the grinding process.

When using natural diamonds as the raw material for drawing dies, a defined <111> orientation is achieved at UKD by means of a systematic grinding process.

5. Internal stresses in diamonds

Internal stresses in crystals can lead to cracks in the material. Stresses can be rendered visible by means of polarisation microscopy. The light passing unimpeded through the optical path of the microscope can be extinguished by means of two polarisation filters offset by 90°. If stresses are present in the crystal, these become visible as coloured areas as a result of the two-dimensional rotation of the three-dimensional light wave at the site of the stress.

Figure 13 shows the internal stresses in MONODIES 111. Relatively sharp stress gradients can be seen around the crystal nucleus. The centre crystal also has an inclusion at the top right.

The stress gradients in the natural diamonds in Fig. 14 appear to be smoother.

The relatively pure natural diamond displays a more favourable stress profile. This means a lower tendency to crack.

6. Economic comparison of natural diamond versus synthetic monocrystal

Our in-house specification at UKD (Fig. 15) assigns each drawing die diameter (column 1) a diamond height (column 2) and a minimum diameter for the ground facets (column 3). The facet diameters of the synthetic monocrystals of a certain manufacturer are listed in column 4 for corresponding diamond heights. These are clearly smaller.

In Fig. 16, 10 "UKD-<111>" natural diamonds and 10 synthetic monocrystals with an identical height of between 1.0 and 1.05 mm were weighed. The average weight of the "UKD-<111>" natural diamonds is 48% greater than that of the synthetic monocrystals, thus naturally offering greater repolishing possibilities.

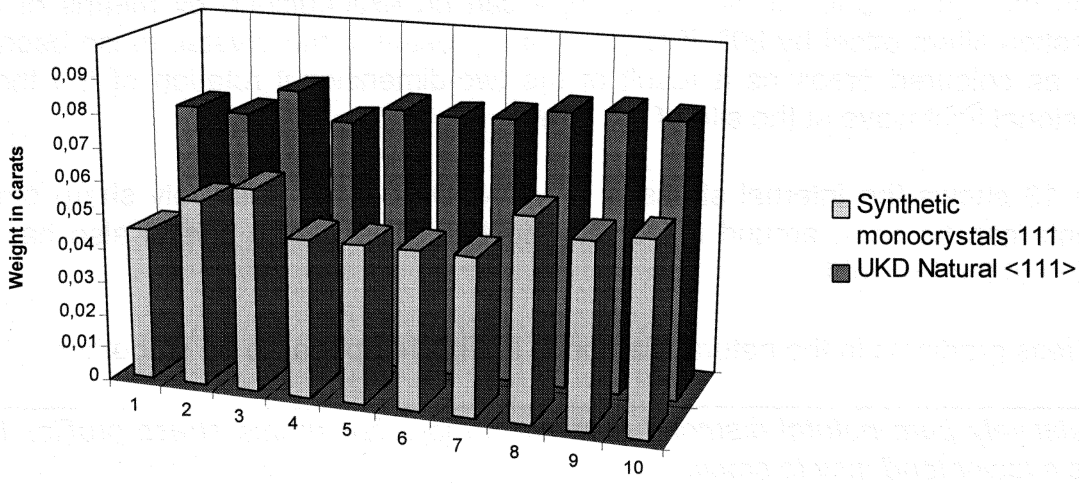
Fig. 15: UKD specification (excerpt) for drawing cone angles of 12 and 14 degrees (hard materials)

Drawing die diameter in mm	Diamond height in mm	Minimum diameter of the ground facets in mm	
		Natural diamond	Synthetic monocrystal
0.10	0.80	0.90	0.82
0.20	0.90	1.00	0.90
0.25	1.00	1.10	0.98
0.30	1.10	1.20	1.05
0.40	1.30	1.40	1.17
0.50	1.40	1.60	
0.60	1.50	1.80	
0.70	1.70	2.10	
0.80	1.80	2.30	
0.90	1.90	2.40	
1.00	2.00	2.50	
1.10	2.10	2.60	
1.20	2.20	2.70	
......	......	......	

The height tolerance is +/- 0.05 mm.

Fig. 16: Comparison of the carat weights at a diamond height of 1.0 to 1.05 mm

Material	Average weight	Index
Synthetic monocrystals 111	0.0534 carat/pc.	100
UKD Natural <111>	0.079 carat/pc.	148



When using synthetic monocrystals corresponding to the conventional weights (sizes) of natural diamonds, the cost of the raw materials is very expensive, although this may change in the long term.

Today, "UKD Natural <111>" drawing dies are the more economical solution in comparison with those made of synthetic monocrystals.

7. Quality assurance for "UKD Natural <111>"

The fact that synthetic monocrystals are an industrially reproducible product is countered by the systematic selection and grinding process at UKD.
The goods offered by the dealers are subjected to a multi-stage selection process. After the first visual inspection by a skilled person (Fig. 17), assessment of the good material is repeated by the head of the Pre-Production Department. Stone for stone. Only then are the suitable diamonds purchased, and those diamonds classed as unsuitable are returned to the dealer.

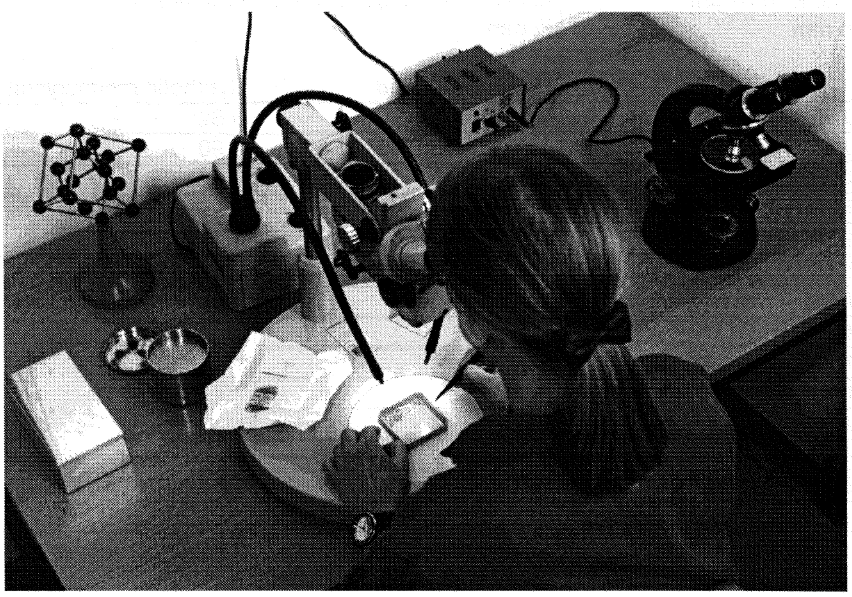
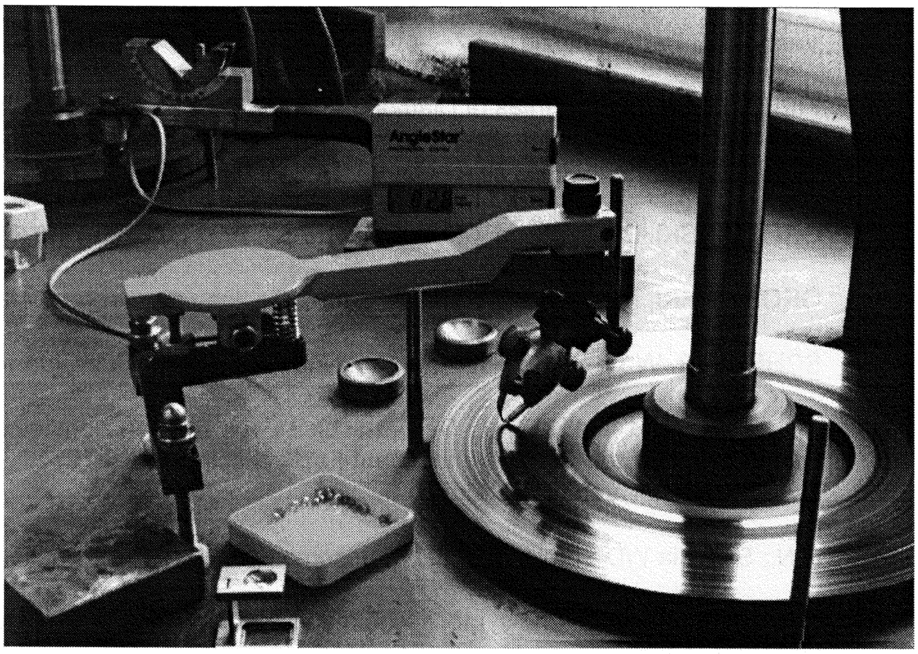


Fig. 17

A systematic grinding process allows the crystalline orientation of the drilling direction <111> to be maintained (Fig. 18).

Fig. 18



Drawing dies with <111> orientation are marked with a triangle (three-point grind) on the front side of the mount (Fig. 19).

This process is subject to our quality assurance system, which is certified pursuant to DIN/ISO 9001.

Fig. 19

References:

1. KLEBER, W.: Einführung in die Kristallographie. Verlag Technik GmbH, Berlin 1990

2. HALLA, F. and MARK, H.: Röntgenographische Untersuchung von Kristallen. Verlag J.A. Barth, Leipzig 1937

3. FIELD, J.E.: The Properties of Diamond. Academic Press Ltd., London 1990

4. VLEESCHDRAGGER, E.: Hardness 10: Diamond. Gaston Lachurié, Paris 1986

5. GRODZINSKI, P.: Diamond Technology. N.A.G. Press, London 1953

6. GRODZINSKI, P.: Diamond Tools. A. Smit & Co. Inc., New York 1944

7. SCHEDLER, W.: Hartmetall für den Praktiker. VDI Verlag, Düsseldorf 1988

8. SCHOB, O. and VRIEZE, W.: Der Verschleiss von Diamantziehsteinen in Abhängigkeit von der kristallographischen Orientierung. Draht-Fachzeitschrift No. 6/1974, Probst & Meiner-Verlag, Coburg

9. N.N.: Sumidia WD Sumicrystal CD. Sumitomo Eletric Ind. Ltd., Düsseldorf 1990

10. N.N.: De Beers PKD- und synthetische monokristalline Diamantziehsteinrohlinge. De Beers Industrial Diamond Division, Düsseldorf 1993

11. LEYER, R.: Bohren feinster Löcher kleiner 80 µm in Diamant. In: Abtragen und Bohren mit Festkörperlasern. VDI Verlag, Düsseldorf 1993

12. KIWUS, U.: Transforming Diamond into Graphite. Wire Industry, November 1991 issue, Publex Int. Ltd., Oxted, Surrey