Lightnovo

APPLICATION NOTE

RGm Raman Microscope

for Non-Destructive Gem Analysis: Rutile Needles in Star Sapphire



ApN-011

Key words

Star sapphire; corundum; asterism; rutile inclusions; silk; confocal Raman microscopy; Raman mapping; MCR-ALS; needle orientation; gemmology

Introduction

Corundum (α-Al_2O_3) is one of the most valued gem minerals: red corundum is ruby, all other colours are sapphire. Some stones show **asterism**, a six-rayed star of light that moves across the surface of a cabochon (title photo). The star is caused by fine needle-shaped inclusions of **rutile** (TiO_2), the so-called silk [1, 2].

Why a star with six rays? Each needle is a thin cylinder that reflects and scatters light into a narrow fan perpendicular to its length. Many parallel needles therefore produce one bright line of light across the domed surface, at 90° to the needles (the cat's-eye effect). In corundum the rutile needles grow along three directions at 60°/120° in the basal plane, following the hexagonal symmetry of the host [1]. Three sets of needles give three lines of light crossing at 60°, i.e. a star with six rays. The star is sharpest when the dome of the cabochon is centred on the crystal c-axis.

Inclusions are key identification markers in gemmology: silk proves that a corundum is natural [1]. An optical microscope shows the needles, but cannot identify their phase or resolve grains below a few micrometres. **Confocal Raman microscopy** identifies each inclusion by its spectrum, non-destructively and with a depth resolution of a few micrometres.

This Application Note shows how the Lightnovo **RGm** Raman microscope identifies, counts, sizes and orients the rutile inclusions of a star sapphire in three dimensions.

Materials and methods

Lightnovo RGm Raman microscope

The measurements were made with the Lightnovo **RGm** Raman microscope (Fig. 1), equipped with a 532 nm laser and a 100× objective (NA 0.9). The measurement chamber is temperature stabilized to 0.1 °C, which allows unattended high-resolution maps over several days.

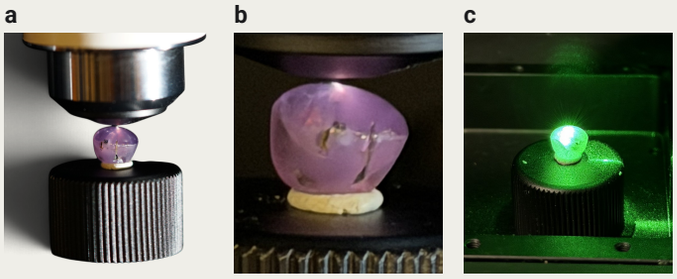


Figure 1. (a) RGm objective and sample. (b) Star-sapphire crystal. (c) Crystal under 532 nm laser light in the RGm.

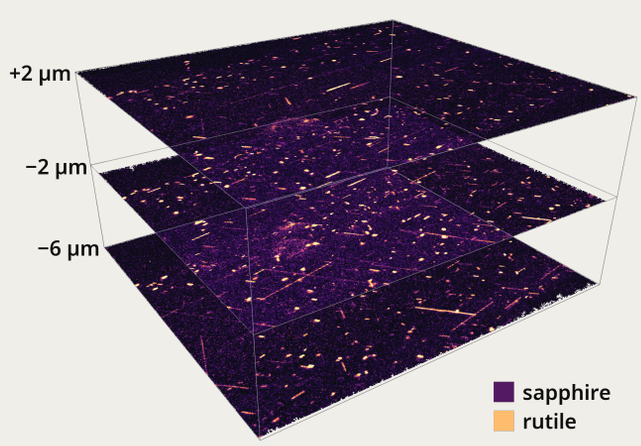


Figure 2. 3D view of the MCR maps at the three focus positions, 200 × 180 µm each (brightness enhanced). Purple: sapphire host; yellow: rutile needles and grains.

Sample and measurements

A star-sapphire cabochon was mapped through its polished surface at one spot. Each map covers **200 × 180 µm** with a step of 0.5 µm (401 × 361 = 144 761 spectra), 0.5 s exposure per spectrum and a range of 50–1000 cm^{-1}. Maps were recorded at three focus positions of the stage, **+2, −2 and −6 µm**, i.e. at three depths below the surface (about 32 h per plane, Fig. 2).

Data processing

The three planes were aligned to each other by cross-correlation of the rutile band maps (595–645 cm^{-1}). Fitted positions of more than 300 inclusions confirm the alignment to ≈ 0.5 µm. The spectra of all three planes were then decomposed jointly by multivariate curve resolution (**MCR-ALS**, non-negative, 3 components, 100–900 cm^{-1}, 4 cm^{-1} bins).

Inclusions were detected on the rutile maps: grains by fitting 2D Gaussians, needles by an oriented line filter (36 directions) followed by a fit of a line-segment model that returns the centre, length, width and angle of each needle. Orientation families were found by length-weighted circular k-means.

Results

Rutile is the only inclusion phase

Rutile has broad bands at 610 (A_{1g}), 445 (E_g) and ≈ 240 cm^{-1}, clearly different from anatase [3]; sapphire has narrow bands at 378, 418, 430, 448, 577, 645 and 750 cm^{-1} [4] (Fig. 3).

MCR resolves three components (Fig. 4). **C1** (610, 446, 230 cm^{-1}) matches rutile and shows needles and grains. **C3** (418, 578, 750 cm^{-1}) is corundum; inclusions appear in it as dark spots where the host is displaced. **C2** is a broad background. No anatase, hematite or ilmenite was found: **rutile is the only crystalline inclusion phase**.

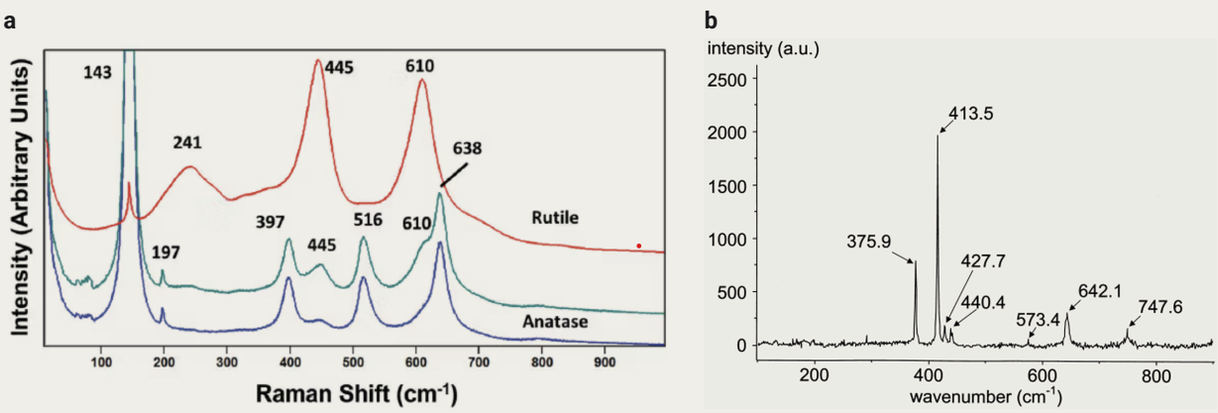


Figure 3. Reference Raman spectra. (a) Rutile and anatase, adapted from Cao and Yi [3]. (b) Sapphire, adapted from Kadlečíková et al. [4].

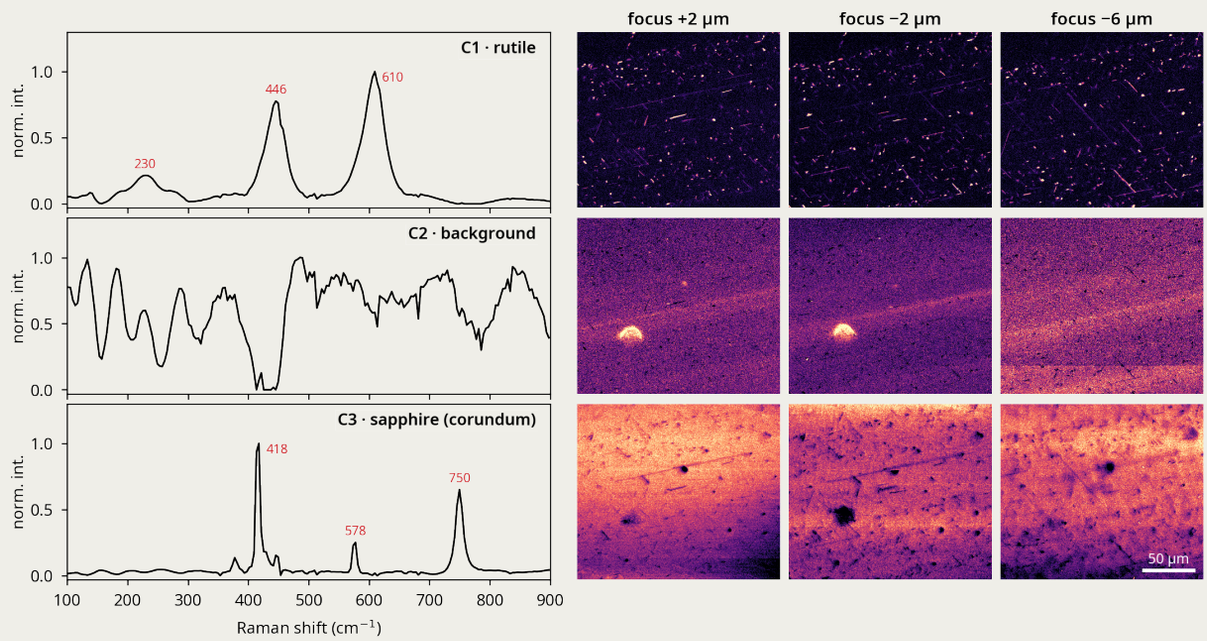


Figure 4. MCR-ALS decomposition of the three focus planes. Left: pure component spectra; right: concentration maps (200 × 180 µm) at +2, −2 and −6 µm. C1 = rutile, C2 = background, C3 = sapphire.

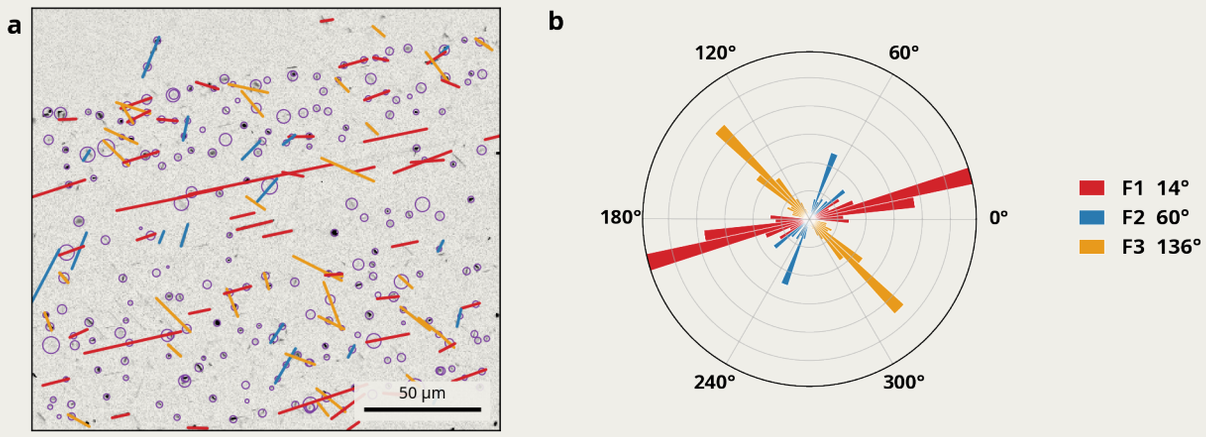


Figure 5. (a) Rutile map (C1, +2 µm): fitted needles coloured by orientation family, grains as circles (2σ). (b) Length-weighted rose of all 158 needle orientations, measured counter-clockwise from the horizontal axis of the map.

Rutile needles and grains

The rutile maps show straight **needles** and small round **grains** (Fig. 5a, Table 1). Grains and needle cross-sections are at the optical resolution limit (FWHM ≈ 1.6 µm), so their true diameters are below ≈ 1.5 µm. The needles are typically 12 µm long, the longest reach 63 µm.

Table 1. Rutile inclusions per focus plane (densities per mapped area).

	+2 µm	−2 µm	−6 µm
Rutile needles	99	102	89
Needles per mm²	2 740	3 140	2 480
Average needle length	12.9 µm	12.2 µm	13.7 µm
Rutile grains	228	226	205
Grains per mm²	6 300	6 950	5 710

Needle orientations

The needles form **three orientation families** at 14°, 60° and 136° (Fig. 5b). The two dominant families (80 % of the needle length) are 57–59° apart, and the angles between all pairs of needles peak at 58–61°: the 60° geometry of the rutile directions in the basal plane of corundum that produces the star. The third family is smaller and less well defined.

Distribution in depth

The three planes show largely different inclusions (Fig. 2): of the 158 needles, 92 appear in at least two planes and 40 in all three. The rutile is thus distributed through the depth of the stone. Needles seen in several planes shift by only 1–2 µm, i.e. they lie at a shallow angle to the surface.

References

1. Jeršek et al., 2021. doi.org/10.1007/s40828-021-00143-0
2. Gem Rock Auctions, Star sapphire. gemrockauctions.com
3. Cao and Yi, 2020. doi.org/10.1002/slct.202002931
4. Kadlečíková et al., 2001. doi.org/10.1016/S0026-2692(01)00087-8

Conclusion

The Lightnovo **RGm** Raman microscope maps the inclusions of a star sapphire non-destructively, through the polished cabochon, at 0.5 µm steps and in several planes of depth. Multivariate analysis identifies **rutile as the only crystalline inclusion phase**. Individual needles and grains close to the optical resolution limit are fitted, which gives their density, size and orientation.

The needles form **three families at ≈ 60°**. Each family reflects light into one bright line perpendicular to its needles; the three lines cross at 60° and form the **six-rayed star**. The Raman maps thus show directly the inclusion geometry behind asterism. Such phase-resolved 3D inclusion maps can support the gemmological identification of natural corundum and its origin.

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