GROWTH METHOD AND GROWTH-RELATED PROPERTIES OF A NEW TYPE OF RUSSIAN HYDROTHERMAL SYNTHETIC EMERALD

By Karl Schmetzer

A new type of Russian hydrothermal synthetic emerald is produced by seeded growth in steel autoclaves without noble-metal inserts; the seed slices have been cut parallel to a face of the second-order hexagonal dipyramid s *{1121}. This seed orientation avoids the easily recognizable growth pattern seen in earlier Russian production. However, characteristic growth planes of a different nature—that is, parallel to* s *and forming a 45° angle with the optic axis—are present in the new material.*

Hydrothermally grown synthetic emeralds from Russia have been discussed in the gemological literature since 1983. Gemological, chemical, and spectroscopic properties of these synthetic emeralds were comprehensively described by Schmetzer in 1988. Production methods were also detailed. The most noteworthy features of this older manufactured material are:

- Normal chromium, high iron, and (unlike other synthetic or natural emeralds) measurable amounts of nickel and copper.
- Absorption bands of Cr^{3+}, Fe^{3+}, Ni^{3+}, and Cu^{2+} in the visible and near-infrared, with chromium and nickel as dominant color-causing trace elements.
- Absorption bands of type I and type II water molecules in the infrared.
- Series of parallel growth lines with a step-like microstructure, which are occasionally connected to color zoning (figure 1), revealing an inclination of 30°–32° vis à vis the optic axis of the samples.

Details of the production technique explain why these properties were unique for commercially produced synthetic emerald. Specifically, seed slices oriented parallel to a second-order hexagonal dipyramid $(5\ 5\ \overline{10}\ 6)$ or its symmetric equivalent are placed in steel autoclaves without noble-metal inserts. With this seed orientation (for that of other commercial producers, see Kiefert and Schmetzer, 1991), extremely fast growth can be obtained (Klyakhin et al., 1981; Lebedev and Askhabov, 1984; Lebedev et al., 1986).

The crystal form $\{5\ 5\ \overline{10}\ 6\}$ has not been observed in natural beryl (see Goldschmidt, 1897), because crystal faces generally correspond to the directions of slow growth. As a consequence of the rapid growth of the early Russian hydrothermal synthetics, however, a distinct step-like microstructure is produced parallel to the seed surface, and subindividuals of synthetic emerald are found

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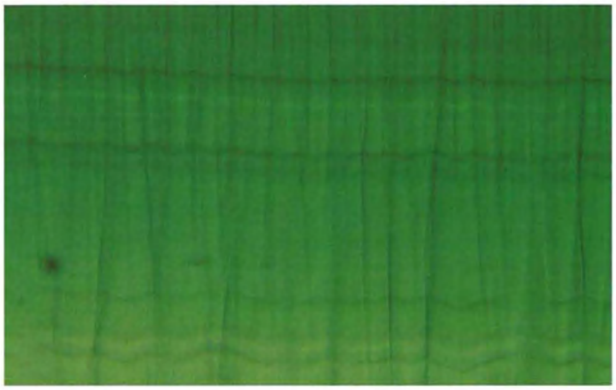

Figure 1. The earlier production of Russian hydrothermal synthetic emerald shows step-like growth lines and color zoning, as well as irregularly changing subgrain boundaries between subindividuals that are almost perpendicular to the color zoning. Crossed polarizers, immersion, magnified 45×.

Figure 2. Also in the earlier production of Russian hydrothermal emerald, boundaries between subindividuals appear as an angular growth pattern. Immersion, magnified 45×.

with a preferred orientation oblique to the seed plate (figure 1). The boundaries between these subindividuals are characterized by angular growth patterns (figure 2), which are also easily recognizable with a microscope.

Because of these characteristic growth features, such Russian hydrothermally grown synthetic emeralds can be distinguished easily from their natural counterparts by microscopic examination. Additional techniques, such as spectroscopy or X-ray fluorescence, are rarely necessary.

An apparently new type of Russian hydrothermally grown synthetic emerald was first mentioned by Scarratt (1994) and is comprehensively described by Koivula et al. in this issue (1996). This new material does not show the distinct growth pattern of the previous material, although the gemological, spectroscopic, and chemical properties were similar to that of the older type. This article describes the unusual growth pattern of this newer material and suggests the changes in growth technique that have caused it.

MATERIALS AND METHODS

In November 1995, the author purchased eight "rough" samples of this new type of Russian synthetic emerald in Bangkok, where they were offered as a new type of internally "clean" synthetic emerald. All samples were fragments or slices of what were originally larger synthetic emerald crystals. Two contained residual portions of colorless (figure 3) or slightly greenish seeds. One of the samples had small external crystal faces, which were identified as prisms $\{10\bar{1}0\}$ and $\{11\bar{2}0\}$ in combination with a face of the hexagonal dipyramid s $\{11\bar{2}1\}$.

Four additional faceted samples were made available by colleagues from GIA, part of the sample described in Koivula et al. (1996). Because the gemological properties of the eight rough samples were consistent with the material described in the Koivula et al. article, the reader is referred to that comprehensive description for additional information.

The internal growth structures of these samples were characterized by means of a Schneider horizontal (immersion) microscope with a specially designed sample holder and with specially designed

Figure 3. In this sample of the new type of Russian hydrothermal emerald, we can see a colorless seed plate and growth zoning parallel to the seed/synthetic emerald boundary at a 45° angle to the c-axis. Crossed polarizers, immersion, magnified 16×.

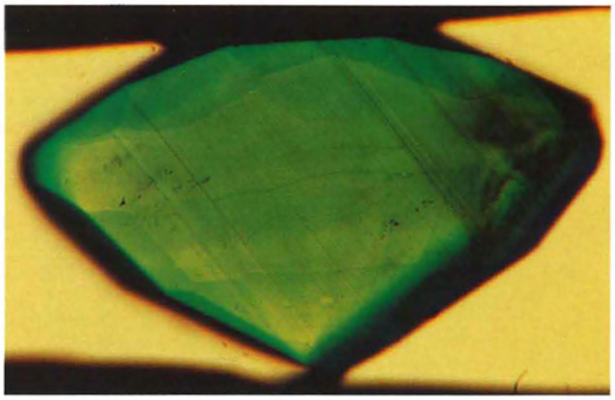

Figure 4. A pattern of growth planes parallel to the hexagonal dipyramid s *can be seen intersecting the c-axis at an angle of 45° in this new type of Russian hydrothermal emerald. Immersion, magnified 25×.*

(to measure angles) eyepieces. For more on the techniques used to determine growth structures, refer to Box A of Peretti et al. (1995, p. 8).

RESULTS

Energy-dispersive X-ray fluorescence (EDXRF) analysis and absorption spectroscopy revealed the presence of chromium, iron, nickel, and copper as trace elements, which is consistent with known data on the earlier material; the infrared spectra were also similar to those seen for the earlier product (see, e.g., Schmetzer, 1988). All of these properties indicate that the new material is still produced by seeded growth in steel autoclaves without noble-metal liners. That is, the copper and nickel (and high iron) originate at least partly from the walls of the autoclave, and would not be evident if a noble-metal liner (a more expensive technique) were used.

The two samples that retained seed residue revealed distinct growth zoning consisting of one series of planar growth faces parallel to the seed/synthetic emerald boundary (figure 3). All other rough and faceted samples showed a similar series of parallel growth planes (figure 4). In all 12 samples examined, these dominant growth patterns formed an approximately 45° angle with the optic axes of the emerald crystals. These measurements indicate an orientation of the seeds parallel to a face of the second-order hexagonal dipyramid *s* $\{11\bar{2}1\}$. In addition to the distinctive growth pattern parallel to *s*, the rough sample with prism faces showed small areas with subordinate growth zoning parallel to both prisms $\{10\bar{1}0\}$ and $\{11\bar{2}0\}$. One faceted sample had growth zoning parallel to one prism face in a small growth area, too. These two samples probably came from the growth area of a synthetic crystal that was confined to the upper or lower end of the respective seed. No growth pattern similar to that of the older material was observed.

Hydrothermally grown synthetic emeralds of other producers, in general, also reveal only one dominant orientation of growth planes relative to the respective seed, the angles of which are summarized in table 1. Note the significantly greater angle for the new Russian material. By comparison, natural emeralds typically show more than one orientation of growth planes, and they are different from those seen in these hydrothermal synthetics. In particular, *s* faces in natural emeralds will normally occur in combination with prism faces, with a basal pinacoid, and with other hexagonal dipyramids, but not as a single and dominant growth plane (see Kiefert and Schmetzer, 1991).

DISCUSSION

Experiments with hydrothermal emerald synthesis have shown that growth rates perpendicular to *s* $\{11\bar{2}1\}$ are somewhat slower than growth rates perpendicular to $\{5\ 5\ \overline{10}\ 6\}$. By using seed slices cut parallel to *s*, however, good growth rates can still be obtained (Klyakhin et al., 1981; Lebedev and Askhabov, 1984; see also Flanigen, 1971; Flanigen and Mumbach, 1971), and the resulting material lacks the easily recognizable growth pattern of the older material.

TABLE 1. Orientation of seeds and dominant growth planes in hydrothermally grown synthetic emeralds.[a]

Producer or trade name	Inclination of seed and/or growth planes versus the optic axis
Linde	36°–38°
Regency	38°
Lechleitner	32°–40°
Biron	22°–23°
Pool	22°–24°
AGEE	19°–21°
Swarovski	0°
Russian (old)	30°–32°
Russian (new)	43°–47°

[a]From Kiefert and Schmetzer, 1991, and author's files (based on examination of at least 10 samples for each product). Note that each producer normally used only one specific orientation. Even hydrothermal synthetic emeralds distributed under different names (e.g., Linde and Regency) can be shown from the orientation of their seeds, vis à vis the c-axis of the beryl crystals, to be products of the same manufacturing technique.

The recognition of one dominant growth pattern parallel to *s* in an emerald of doubtful origin is of diagnostic value as an indication that it may be synthetic. Further diagnostic techniques (e.g., absorption spectroscopy and/or EDXRF) should be used to confirm or disprove such a preliminary result. This is due to the possible presence of *s* faces in natural emerald.

REFERENCES

Flanigen E.M. (1971) Hydrothermal process for growing crystals having the structure of beryl in an alkaline halide medium. United States Patent No. 3,567,642, issued March 2, 1971.

Flanigen E.M., Mumbach N.R. (1971) Hydrothermal process for growing crystals having the structure of beryl in an acid halide medium. United States Patent No. 3,567,643; issued March 2, 1971.

Goldschmidt V. (1897) *Krystallographische Winkeltabellen.* Verlag von Julius Springer, Berlin, pp. 65–66.

Kiefert L., Schmetzer K. (1991) The microscopic determination of structural properties for the characterization of optical uniaxial natural and synthetic gemstones. Part 2: Examples for the applicability of structural features for the distinction of natural emerald from flux-grown and hydrothermally-grown synthetic emerald. *Journal of Gemmology*, Vol. 22, No. 7, pp. 427–438.

Koivula J.I., Kammerling R.C., DeGhionno D., Reinitz I., Fritsch E., Johnson M.L. (1996) Gemological investigation of a new type of Russian hydrothermal synthetic emerald. *Gems & Gemology*, Vol. 32, No. 1, pp. 32–39.

Klyakhin V.A., Lebedev A.S., Il'in A.G., Solntsev V.P. (1981) Growing of hydrothermal beryl. *Sintez i Vyrashchivanie Optich. Kristallov i Yuvelir, Kamnei*, Novosibirsk, pp. 45–66 [in Russian].

Lebedev A.S., Askhabov A.M. (1984) Regeneration of beryl crystals. *Zapiski Vsesoyuznogo Mineralogicheskogo Obshchestva*, Vol. 113, No. 5, pp. 618–628 [in Russian].

Lebedev A.S., Il'in A.G., Klyakhin V.A. (1986). Hydrothermally-grown beryls of gem quality. In *Morphology and Phase Equilibria of Mineral,* Proceedings of the 13th General Meeting of the International Mineralogical Association, Varna 1982, Vol. 2, publ. Sofia, pp. 403–411 [in Russian].

Peretti A., Schmetzer K., Heinz-Jürgen B., Mouawad F. (1995) Rubies from Mong Hsu. *Gems & Gemology*, Vol. 31, No. 1, pp. 2–26.

Scarratt K. (1994) Lab report from the Asian Institute of Gemological Sciences (AIGS). *JewelSiam*, Vol. 5, No. 1, pp. 62–69.

Schmetzer K. (1988) Characterization of Russian hydrothermally-grown synthetic emeralds. *Journal of Gemmology*, Vol. 21, No. 3, pp. 145–164.

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