

# A STUDY OF NAIL-HEAD SPICULE INCLUSIONS IN NATURAL GEMSTONES

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Nail-head spicules are inclusions that have traditionally been associated with hydrothermal synthetic quartz and emerald. They are caused primarily by rapid growth conditions and disturbances during crystallization of their host. However, these or similar-looking inclusions have also been found in natural gems. The authors examined a natural emerald and blue sapphire showing true nail-head spicules, and a yellow sapphire, spinel, diamond, and rock crystal quartz with inclusions that strongly resembled them. Nail-head spicules remain a notable feature of rapid and disturbed growth, but they do not confirm a stone's natural or synthetic origin without further examination.

**A**lthough the identification of any gem material requires testing with a variety of instruments, in most cases the stone's natural or synthetic origin can be determined conclusively by features seen with magnification. For example, curved lines/bands (flame fusion), "hounds tooth" patterns (hydrothermal), and "wispy veil" inclusions (flux) are all classically associated with synthetics.

Nail-head spicules are typically associated with synthetic hydrothermal (and occasionally flux-grown) emerald (figure 1) and synthetic quartz (figure 2). However, such inclusions have been reported in a number of natural and other synthetic gem materials (table 1), including natural emerald (DelRe, 1992; Rockwell, 2003). Similar-appearing features have been observed in photomicrographs of natural sapphires from Madagascar (Kiefert et al.,

1996; Milisenda and Henn, 1996), though they have not been described in detail.

This article documents nail-head spicules and similar-appearing features in a variety of natural gem materials, which could potentially lead to their misidentification as synthetics.

## NAIL-HEAD SPICULES AND THEIR FORMATION

Nail-head spicules are wedge-shaped two-phase (liquid and gas) inclusions capped by crystals that act as growth obstacles. In synthetic materials, they may occur in numerous places throughout the sample, but they mainly appear near the seed plate. According to Gübelin and Koivula (1986), since the fluid component is in direct contact with the crystal cap, an inclusion of this kind is technically a *three-phase* inclusion.

Relatively disturbed growth is the primary cause of nail-head spicules. During growth of the host crystal, a small crystal or platelet is deposited on its surface. As the crystal continues to grow past the inclusion, a tapered void is created, which traps the hydrothermal growth medium such that, upon cooling, it becomes two phases consisting of liquid and a gas bubble. In flux-grown synthetics, such voids may contain flux (Schmetzer et al., 1999). In the case of synthetic emeralds, the crystal cap is usually phenakite, beryl, or chrysoberyl, and may even be gold from the crucible (again, see table 1).

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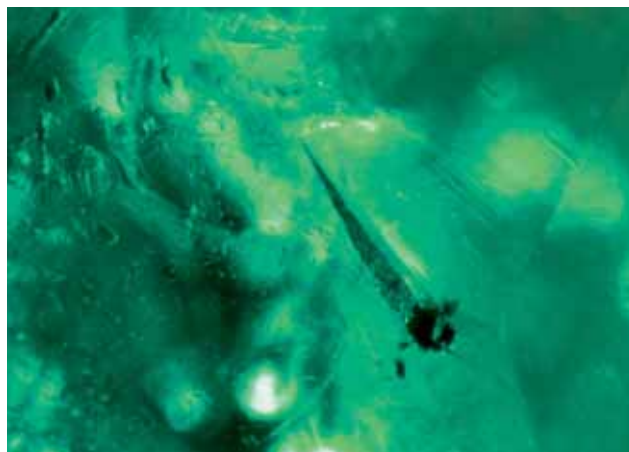


Figure 1. At left is a classic nail-head spicule in a hydrothermally grown synthetic emerald. On the right is a spicule breaking the surface of a flux-grown synthetic emerald. Note the black material at the surface break. Photomicrographs by John I. Koivula (left, magnified 80 $\times$ ) and G. Choudhary (right, magnified 60 $\times$ ).

These flat platelets/crystals are not always easily resolved with a standard gemological microscope.

In synthetic emeralds, nail-head spicules develop most readily when growth occurs on a seed plate inclined at an angle to the crystallographic axes, as in the case of Biron material (Kane and Liddicoat, 1985). To the best of the authors' knowledge, no detailed research has been performed on the formation of nail-head spicules in synthetic gem materials, and further correlation with the growth conditions is beyond the scope of this article.

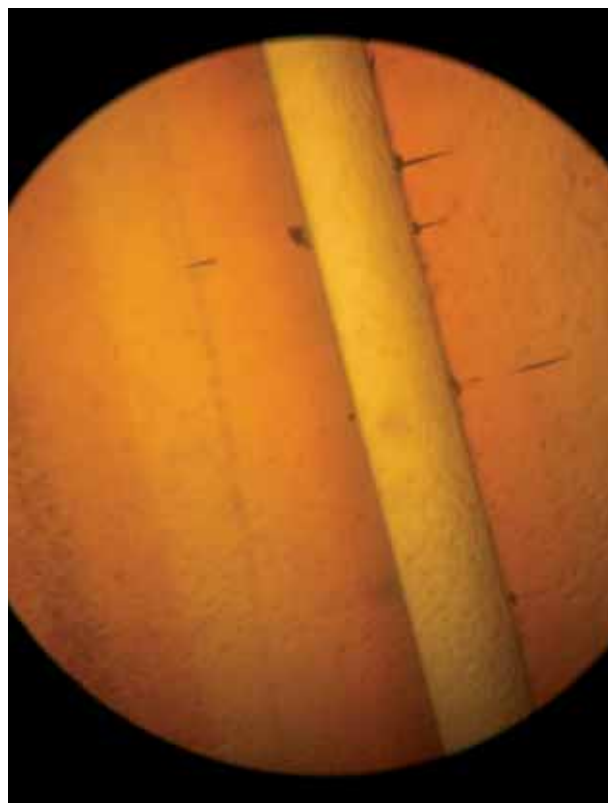
## MATERIALS AND METHODS

Six stones are documented in this report: two sapphires, an emerald, a spinel, a diamond, and a sample of rock crystal quartz. All were faceted except the spinel (a pebble), and all were submitted for testing at the Gem Testing Laboratory, Jaipur, India. The emerald was brought in by a gemologist who had purchased it as a natural specimen of Sandawana (Zimbabwe) origin but was concerned about its identity due to the presence of nail-head spicules along some planes. The remaining stones were submitted for routine identification reports.

Standard gemological tests were conducted on all six stones; however, we could not determine the refractive index of the spinel as it was water-worn. We examined the internal features of the samples with both a binocular gemological microscope, with fiber-optic and other forms of lighting, and a horizontal microscope. Infrared spectra (in the 6000–400  $\text{cm}^{-1}$  range for all stones, with particular attention in the 3800–3000  $\text{cm}^{-1}$  range for the quartz) were recorded using a Nicolet Avatar 360

Fourier-transform infrared (FTIR) spectrometer at room temperature with a transmission accessory. Multiple infrared spectra were collected to find the

Figure 2. Nail-head spicules are also seen in synthetic quartz, where they are commonly situated along the seed plate, as in this synthetic citrine. Photomicrograph by C. Golecha; magnified 30 $\times$ .



**TABLE 1.** Selected reports of nail-head spicules and similar inclusions in natural and synthetic gem materials.

Gem material	Inclusions	References
<b>Natural</b>		
Emerald	Tapered voids with flat platelets, filled with two-phase inclusions	Rockwell (2003)
	Spicule-like inclusion capped by a yellowish crystal (calcite)	DelRe (1992)
Diamond	Hexagonal columnar indented natural	Smith (1991)
	Etched out needle-like crystals	Chapman (1992)
Sapphire	Rounded apatite crystals accompanied by growth tubes	Kiefert et al. (1996)
	Apatite crystals at the ends of hollow tubes	Milisenda and Henn (1996)
Pezzottaite	Fine growth tubes emanating from crystals of tourmaline	Laurs et al. (2003)
<b>Synthetic</b>		
Hydrothermal emerald (Biron)	Short "needle" emanating from a tiny cluster of euhedral phenakite crystals	Sechos (1997)
	Cone-shaped void filled with a fluid and a gas bubble, with a phenakite crystal at its base; nail-head spicules at the edge of gold inclusions	Kane and Liddicoat (1985)
Hydrothermal emerald (Chinese)	Needle-like tubes with one or two phases associated with beryl or chrysoberyl crystals at broader ends.	Schmetzer et al. (1997)
Hydrothermal emerald (Linde)	Phenakite crystals with wedge-shaped voids extending from them	Liddicoat (1993)
Hydrothermal emerald (Russian)	Growth tubes filled with liquid or two phases, associated with doubly refractive crystals	Schmetzer (1988)
Hydrothermal emerald (Regency)	Reddish brown crystals with pointed growth tubes containing liquid and gas	Gübelin and Koivula (1997)
Flux emerald (Chatham)	Elongated cone-shaped spicules associated with tiny birefringent "phenakite" crystals, and filled with colorless or yellowish molybdenum	Schmetzer et al. (1999)
Flux emerald (Nacken)	Cone-shaped cavities with tiny crystals (beryl), partially filled with multicomponent inclusions: V-bearing polymerized molybdate, nonpolymerized molybdate, isolated aluminous silicates	Schmetzer et al. (1999)
	Wedge-shaped nail-like inclusions with a "phenakite" crystal at the wider end, filled with flux; rarely, large dark brown tapered inclusions with a polycrystalline appearance	Nassau (1980)
Hydrothermal red beryl	Hollow or two-phase (liquid and gas) inclusions, capped by a colorless or colored solid inclusion of unknown nature	Shigley et al. (2001)
Hydrothermal quartz (citrine)	"Breadcrumb" inclusions associated with a two-phase spicule	Gübelin and Koivula (1974)

orientation of best transmission. In the case of the quartz, spectra were taken according to the optic-axis direction as well (both parallel and perpendicular to the c-axis).

## RESULTS AND DISCUSSION

The gemological properties of the six stones are summarized in table 2. In all cases, these were consistent with those reported in the gemological literature for natural samples of each material.

**Sapphire (Specimen 1).** Viewed with magnification, this stone exhibited many crystalline inclusions and some elongate inclusions, mainly concentrated along the wider girdle end. Also observed were elongated, somewhat conical or rectangular inclusions terminated by crystals; these appeared to be nail-head spicules.

When the specimen was immersed in methylene

iodide, the nature of the inclusions became clearer. All the nail-head spicules were oriented in a single direction parallel to the optic axis (figure 3). Each typically consisted of a cluster of crystal-like terminations connected to an elongated conical cavity; several also exhibited two-phase inclusions within the cones. Those spicules with a rectangular tube-like projection were somewhat similar to the inclusion patterns illustrated by Kiefert et al. (1996) and Milisenda and Henn (1996). Most of the spicules were situated among a group of birefringent transparent colorless crystals, many of which had highly reflective faces (figure 4). Also observed were conical apatite crystals, which are commonly associated with Sri Lankan origin (Hughes, 1997). Viewed with diffused illumination (while still in immersion), the sapphire showed strong hexagonal growth zoning with uneven patches of color.

A weak undulating chevron pattern, which indi-

**TABLE 2.** Gemological properties of six natural stones with nail-head spicule or similar inclusions.<sup>a</sup>

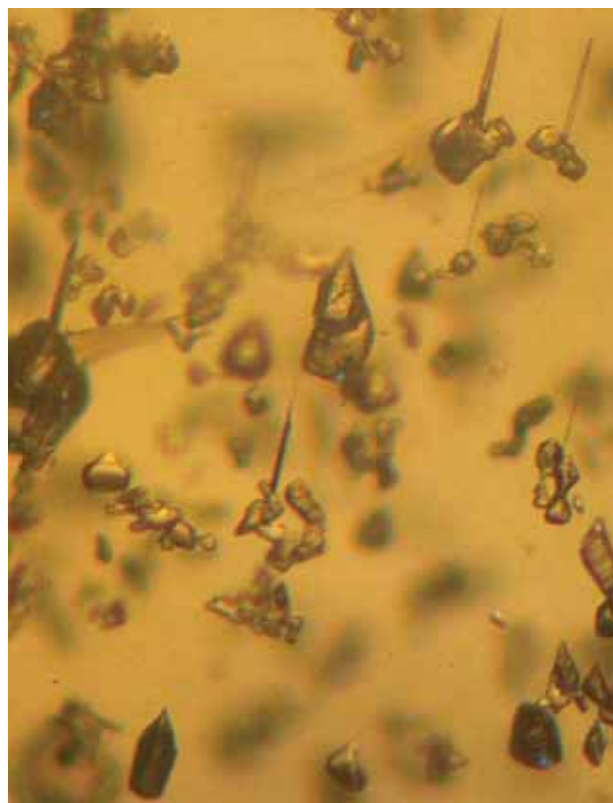
Property	Sapphire		Emerald	Spinel	Diamond	Rock crystal quartz
	Specimen 1	Specimen 2				
Color	Blue	Yellow	Yellowish green	Blue	Light yellow	Colorless
Weight (ct)	3.09	5.00	12.56	13.84	0.61	5.00
Cut style	Cushion mixed	Oval mixed	Octagonal step	Rough	Round brilliant	Oval mixed
RI	1.762–1.770	1.762–1.770	1.584–1.591	nd	OTL	1.542–1.551
SG	3.99	3.99	2.73	3.61	3.52	2.64
Absorption spectrum	nd	nd	Typical chromium spectrum <sup>b</sup>	Iron band at 460 nm	nd	nd
UV fluorescence						
Long-wave	Strong “apricot”	Strong “apricot”	Inert	Inert	Medium blue	Inert
Short-wave	Similar, but weaker	Similar, but weaker	Inert	Inert	Weak blue	Inert

<sup>a</sup>Abbreviations: nd=not determined, OTL=over the limits of the standard refractometer.

<sup>b</sup>Band at 580–625 nm, line at 640 nm, and doublet at 680 nm.

cated the rapid growth necessary for formation of nail-head spicules, was seen mainly where the spicules were concentrated. (Unfortunately, these

*Figure 3. Nail-head spicules are oriented parallel to the optic axis in this natural blue sapphire, seen here immersed in methylene iodide. Each consists of a cluster of crystal terminations connected to a cone. Some of the cones are two-phase inclusions. Photomicrograph by C. Golecha; magnified 40×.*



features could not be resolved clearly for photography.) This undulating pattern somewhat followed the hexagonal color zoning. A literature search turned up no reports of nail-head spicules in synthetic sapphires, so this stone was particularly unusual, as this chevron pattern is often seen in hydrothermal synthetics.

The natural origin of this sapphire was easily confirmed with standard instruments and the presence of other features such as zoning and fluorescence. However, another similar spicule (figure 5) was present near the pavilion, which could have led

*Figure 4. Viewed with crossed polarizers, the crystal clusters in the blue sapphire proved to be birefringent. Also note the conical elongated apatite crystals, which are commonly observed in Sri Lankan sapphires. Photomicrograph by C. Golecha; immersion, magnified 25×.*

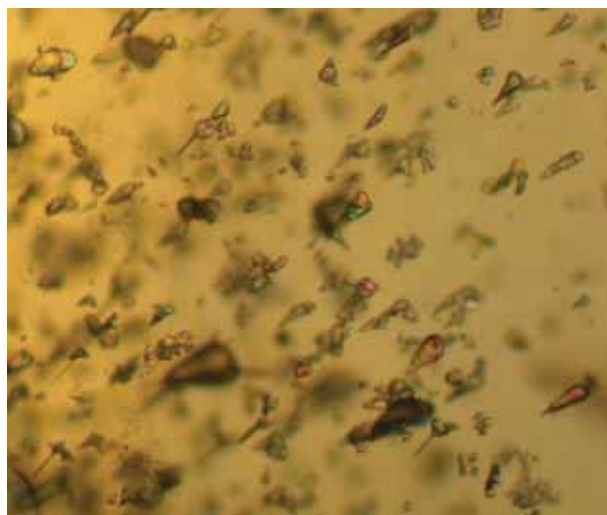






Figure 5. A single spicule (upper right) was present near the pavilion of the blue sapphire. Identification by standard techniques could be difficult if the stone was cut with this inclusion alone. Photomicrograph by C. Golecha; immersion, magnified 30 $\times$ .

to misidentification had the stone been cut with that inclusion alone.

**Sapphire (Specimen 2).** This yellow sapphire contained numerous etch channels, and one of them reached a crystalline inclusion, giving the general appearance of a nail-head spicule (figure 6). The iden-



Figure 6. This etch channel in a yellow sapphire is in contact with an included crystal, resulting in a nail-head spicule-like appearance. Photomicrograph by G. Choudhary; magnified 30 $\times$ .

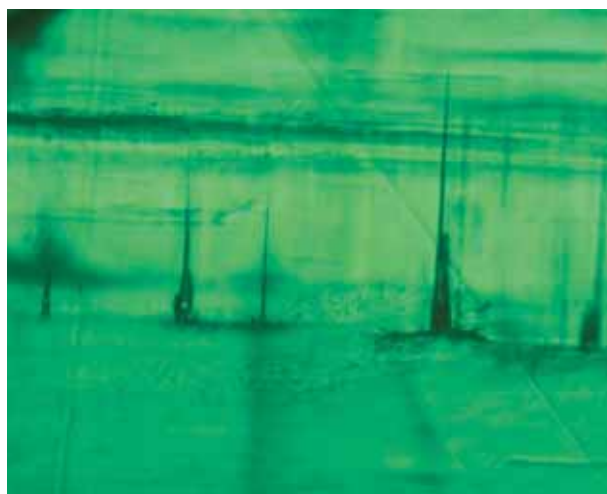
tification of the stone and its natural origin was easily established; however, the inclusion could create confusion for a novice gemologist.

**Emerald.** This stone contained abundant curved, tremolite-like fibrous inclusions (figure 7), which proved its natural origin and indicated its source as Sandawana (Gübelin and Koivula, 1986). When it was viewed from various angles, however, numerous nail-head spicules were observed in a single direction originating from parallel planes (figures 7 and 8) that were oriented perpendicular to the optic axis. The effect was very similar to that seen in

Figure 7. In this emerald, small, dark conical inclusions can be seen projecting from the parallel planes in the background, similar to nail-head spicules projecting from the seed plate in a synthetic emerald. However, the presence of abundant curved, fibrous tremolite-like inclusions indicates a natural origin: Sandawana, Zimbabwe. Photomicrograph by G. Choudhary; magnified 20 $\times$ .



Figure 8. At higher magnification, the parallel growth planes in figure 7 showed abundant nail-head spicules; also note the two-phase inclusions at the broader end. Photomicrograph by G. Choudhary; magnified 35 $\times$ .



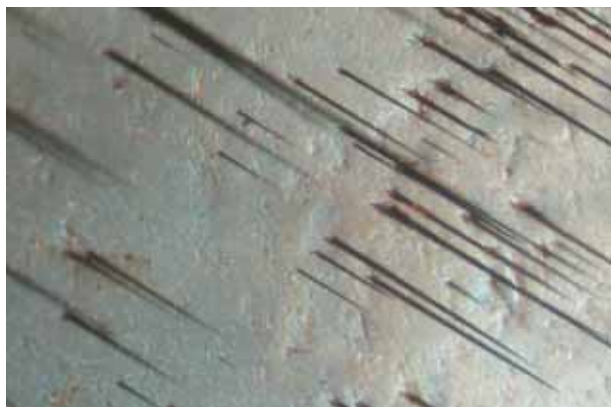


Figure 9. Near the surface of this spinel pebble, a number of long, conical etch features resemble oriented spicule inclusions. Photomicrograph by C. Golecha; magnified 30 $\times$ .



Figure 10. Spicule-like inclusions, some displaying a sharp bend, were observed in various directions pointing toward the interior of the spinel pebble. Photomicrograph by C. Golecha; magnified 20 $\times$ .

synthetic emeralds. In this case, the crystals at the base of the spicules could not be resolved, and all the spicules appeared to originate from the plane itself.

Also present were liquid “fingerprints,” flat reflective films oriented along the basal plane, and angular zoning. Confirmation of natural origin came from FTIR spectra taken in various directions for best transmission, which showed the strong peak at 5270  $\text{cm}^{-1}$  that is characteristic of natural emerald (e.g., Choudhary, 2005). Nail-head spicules have been previously encountered in natural emeralds (DelRe, 1992; Rockwell, 2003), so this sample served as a further reminder of the need for careful and complete examination.

**Spinel.** Magnification revealed numerous surface-reaching conical inclusions (figure 9) pointing toward the interior of the pebble from various directions. Some also exhibited a sharp angular bend (figure 10). Careful examination revealed that the inclusions broke the surface with rhomboid or sub-hexagonal/rounded cross sections (figure 11) that varied with the direction of entrance into the stone, which suggested that the shapes were determined by the growth orientation. Some of these inclusions were filled with a brownish epigenetic material (figure 11, right).

At certain angles, these inclusions were highly reflective, with flat surfaces intersecting each other in a pyramidal arrangement. Although their overall characteristics were indicative of etch channels, the

Figure 11. These spicule-like inclusions in spinel appear to be etch channels. Their cross sections varied from rhomboid (left) to sub-hexagonal or rounded (right). Also note the brownish epigenetic filling material (right). Photomicrographs by G. Choudhary; magnified 30 $\times$  (left) and 45 $\times$  (right).

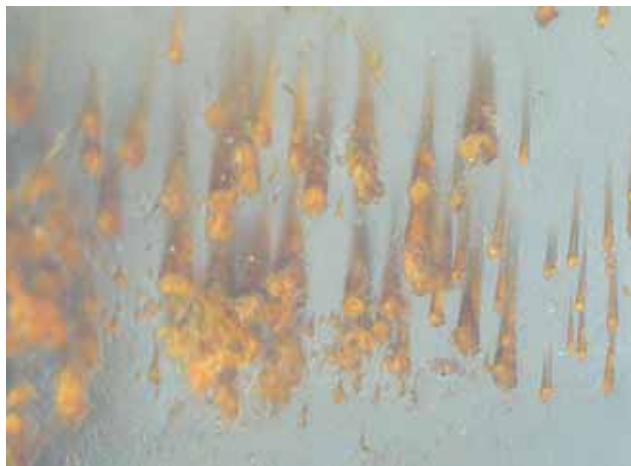




Figure 12. This diamond contains some nail-head spicule-like inclusions that are actually crystals with stress cracks at one end. Given the formation conditions of diamond, true nail-head spicules are not possible. Photomicrograph by C. Golecha; magnified 30 $\times$ .

possibility of these being elongated needles cannot be ruled out (Chapman, 1992). The authors have observed similar inclusions in a few flux-grown synthetic emeralds, where the spicules broke the surface and appeared to be filled with a black material.

Although etch channels are common in a number of natural gems, in this specimen they closely resembled nail-head spicules. Due to the absence of any other visible inclusions, this specimen could have easily confused a novice gemologist.

**Diamond.** Because diamonds form at conditions of very high temperature and pressure, they do not contain fluid inclusions that are resolvable with a gemological microscope. However, this diamond possessed several crystal inclusions with stress cracks (figure 12) that at certain angles appeared to be short needles associated with a crystal. The combination gave a strong resemblance to nail-head spicules. These features were mainly concentrated near the crown in random directions. The diamond's natural origin was ascertained by the presence of naturals on the girdle.

**Rock Crystal Quartz.** Scattered inclusions consisting of whitish aggregates were concentrated along two parallel planes in this stone (figure 13). The scene resembled the "breadcrumb" inclusions often present along the seed plate in synthetic quartz. In certain orientations, some of these whitish clusters closely resembled nail-head spicules (figure 14) and seemed to be formed by the orientation of crystal inclusions almost perpendicular to each other. At

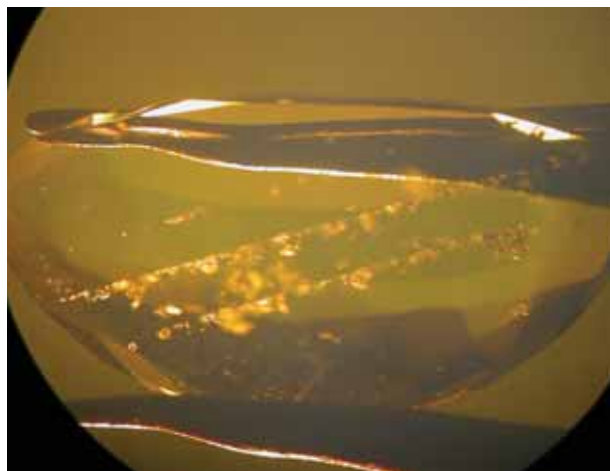


Figure 13. Inclusions are concentrated along two parallel planes in this faceted quartz. The pattern resembles a common inclusion scene in synthetic quartz: "breadcrumbs" along a seed plate. Photomicrograph by C. Golecha; magnified 15 $\times$ .

higher magnification, the clusters proved to be aggregates of a whitish mineral (figure 15). The morphology of the clusters resembled muscovite flakes (Gübelin and Koivula, 2005), but we could not conclusively identify them due to lack of access to Raman spectroscopy.

Further analysis with FTIR spectroscopy showed absorptions in the 3600–3000  $\text{cm}^{-1}$  region, along with a strong peak at 3483  $\text{cm}^{-1}$  that is characteristic for natural crystalline quartz. Similar spectra were record-

Figure 14. The aggregation patterns of these inclusions in quartz strongly resemble nail-head spicules at certain orientations. Photomicrograph by C. Golecha; magnified 35 $\times$ .







Figure 15. At higher magnification, the inclusions in figure 14 proved to be aggregates of a natural mineral, possibly muscovite. Photomicrograph by C. Golecha; magnified 45 $\times$ .

ed parallel and perpendicular to the optic axis, with only minor differences in the intensity of the peaks.

Although the pattern of the whitish crystal aggregates indicated a natural specimen, the presence of spiculate-like inclusions and the concentration of the inclusions along a defined plane could have led to the stone's misidentification as synthetic without careful examination.

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## CONCLUSIONS

Nail-head spiculate inclusions have long been associated with hydrothermally grown synthetic gem materials. Such inclusions indicate rapid, disturbed growth, which is the case for most synthetics but also for some natural gems. Similar-appearing inclusions may be produced by a combination of other features. Much like the spiral “fingerprint” inclusions that were once considered characteristic of Biron synthetic emerald (Gübelin and Koivula, 1986), the mere appearance of nail-head spicules should not be considered conclusive proof of synthetic origin. When such inclusions or similar structures are present, a more detailed examination is necessary to determine the nature of the sample. Such instances serve as reminders of the importance of not relying on any one feature for the identification of a gem material.

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