'Nail-Head Spicule' Inclusions In Natural Gemstones

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Although 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 the features seen with magnification. For example, curved lines or bands (flame fusion), "hounds tooth" patterns (hydrothermal), and "wispy veil" inclusions (flux) are all classically associated with synthetics.

Nail-head spicules are 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.

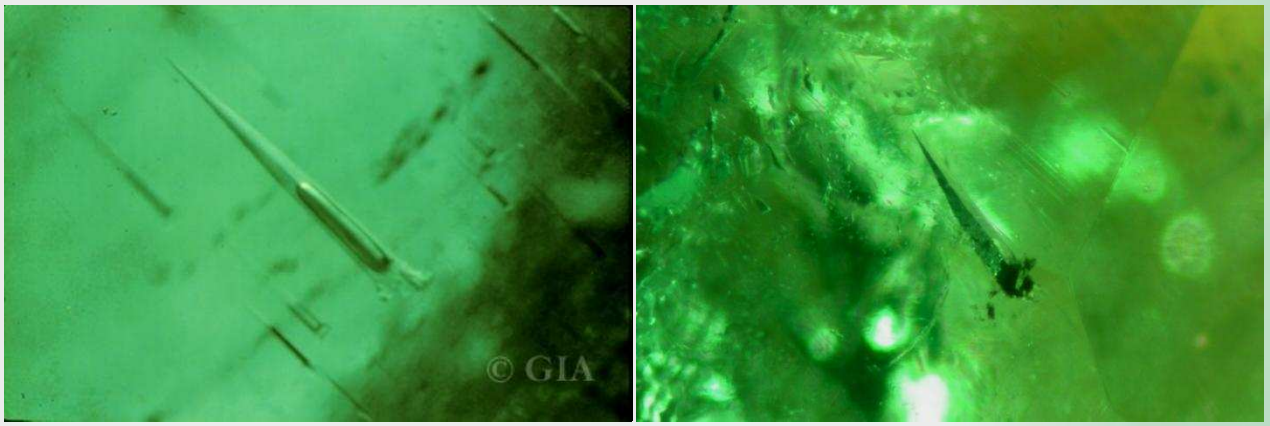


Figure 1: At left is a classic nail-head spicule in a hydrothermally grown synthetic emerald. On the right is a spicule breaking on the surface of a flux-grown synthetic emerald. Note the black material at the surface break. Photomicrographs by John I. Koivula (left, magnified 80x) ©GIA and G. Choudhary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

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 mainly they appear near the seed plate. According to Gübelin and Koivula (1986), 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 emeralds, the crystals cap is usually phenakite, beryl, or chrysoberyl and may even be gold from the crucible (again, see table 1). These flat platelets/ crystals are not always easily resolved with a standard gemmological 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 synthetic emeralds (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.



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 30x

TABLE 1: Selected reports of nail-head spicules and similar inclusions in natural and synthetic stones.

Gem Material	Inclusions	References
	Natural	
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
	Synthetic	
Hydrothermal emerald (Biron)	Short 'needle' emanating from a tiny cluster of euhedral phenakite crystal	Sechos, 1997
	Cone shaped void filled with a fluid and a gas bubble and a phenakite crystal at base; nail head spicules at the edge of gold inclusions	Kane and Liddicoat, 1985
Hydrothermal emerald (Chinese)	Needle like tubes with one or two phase 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-phase associated with double refractive crystals	Schmetzer, 1988
Hydrothermal emerald (Regency)	Reddish brown crystals with pointed growth tubes containing liquid and gas.	Gubelin and Koivula, 1997
Flux emerald (Chatham)	Elongated cone shaped spicules associated with tiny birefringent 'phenakite' crystals and filled with colourless or yellowish molybdenum.	Schmetzer et al.,1999
Flux emerald (Nacken)	Cone shaped cavities with tiny crystals (beryl) and partially filled with multicomponent inclusions: V-bearing polymerized molybdate, non-polymerised molybdate, isolated aluminous silicates	Schmetzer et al.,1999
	Wedge shaped nail-like inclusions with 'phenakite' crystal at wider end and 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) capped by a colourless or coloured solid inclusion of unknown nature	Shigley et al., 2001
Hydrothermal quartz (Citrine)	'Bread crumb' inclusions associated with two-phase spicule	Gubelin and Koivula, 1997

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 gemmologist 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 gemmological tests were conducted on all six stones; however, we could not determine the refractive index of spinel as it was a water-worn. We examined the internal features of the samples with both a binocular gemmological microscope, with fibre -optic and other forms of lighting, and a horizontal microscope. Infrared spectra (in the 6000-400 cm^{-1} range for all stones, with particular attention in the 3800-3000cm^{-1} for the quartz) were recorded using a Nicolet Avatar 360 FTIR spectrophotometer at room temperature with a transmission accessory. The stones were oriented in the direction of best transmission. In the case of the rock crystal quartz, spectra were taken according to the optic axis direction as well (both parallel and perpendicular to the c- axis).

RESULTS AND DISCUSSION

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

TABLE 2: Gemmological properties of six natural stones with nail-head spicule or similar inclusions.

Property	Sapphire		Emerald	Spinel	Diamond	Rock crystal
	Specimen 1	Specimen 2				
Colour	Blue	Yellow	Green	Blue	Light yellow	Colourless
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
Abs. Spectrum	nd	nd	Typical chromium spectrum	Iron band at 460 nm	nd	nd
UV Fluorescence	Apricot (LW); weaker in SW	Apricot (LW); weaker in SW	Inert in LW& SW	Inert in LW& SW	Blue (LW); weaker in SW	Inert in LW & SW

Sapphire (Specimen 1)

Viewed with magnification, the 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 inclusion pattern illustrated by Kiefert et al. (1996) and Milisenda and Henn (1996). Most of the spicules were situated among a group of birefringent transparent colourless 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 colour.

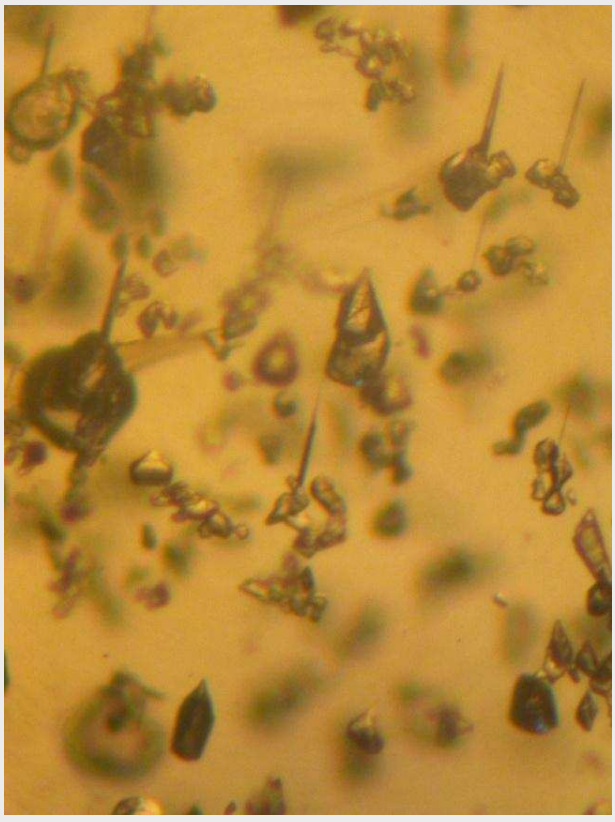


Figure 3: Nail-head spicules are oriented parallel to optic axis in this natural blue sapphire, seen here immersed in methylene iodide. Each consists of a cluster of crystal terminations connected to a cone. Some of the cones are two-phase inclusions. Photomicrograph by C. Golecha, magnified 40x

A weak undulating chevron pattern, which indicated rapid growth necessary for formation of nail-head spicules, was seen mainly where the spicules were concentrated. (Unfortunately, these features could not be resolved clearly for photography.) This undulating pattern somewhat followed the hexagonal colour 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 lead to misidentification had the stone been cut with that inclusion alone.

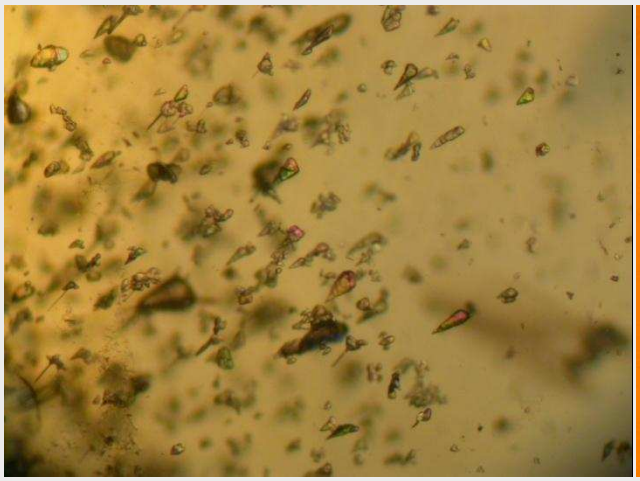


Figure 4: Viewed with crossed polarizers, the crystal clusters proved to be birefringent. Also note the conical elongated apatite crystals, which are commonly observed in Sri Lankan sapphires. Photomicrograph by C. Golecha, magnified 25x

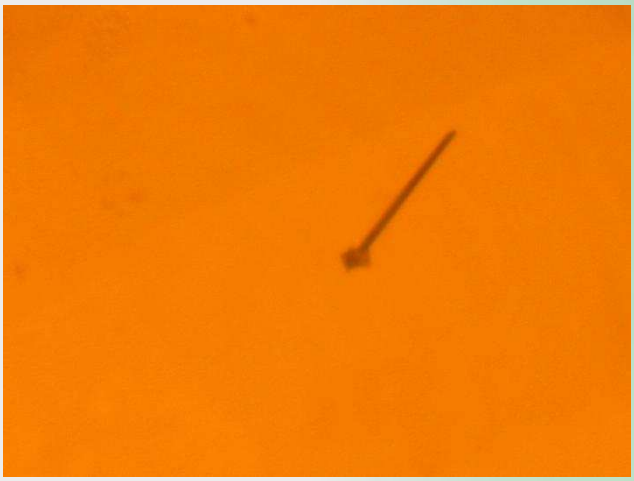


Figure 5: A single spicule was present near the pavilion of the sapphire. Identification by standard techniques could be difficult if the stone was cut with this inclusion alone. Photomicrograph by C. Golecha, magnified 30x.

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

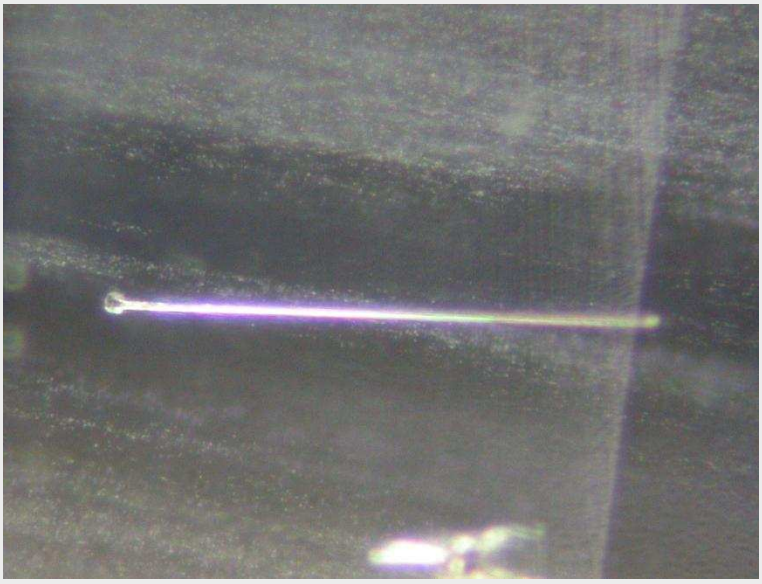


Figure 6: This etch channel in a yellow sapphire is in contact with an included crystal, resulting in a nail-head spicule –like appearance. Photomicrograph by G. Choudhary, magnified 30x

Natural Emerald

The 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 synthetic emeralds. In this case, the crystals at the base of the spicules appeared to originate from the plane itself.



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 20x

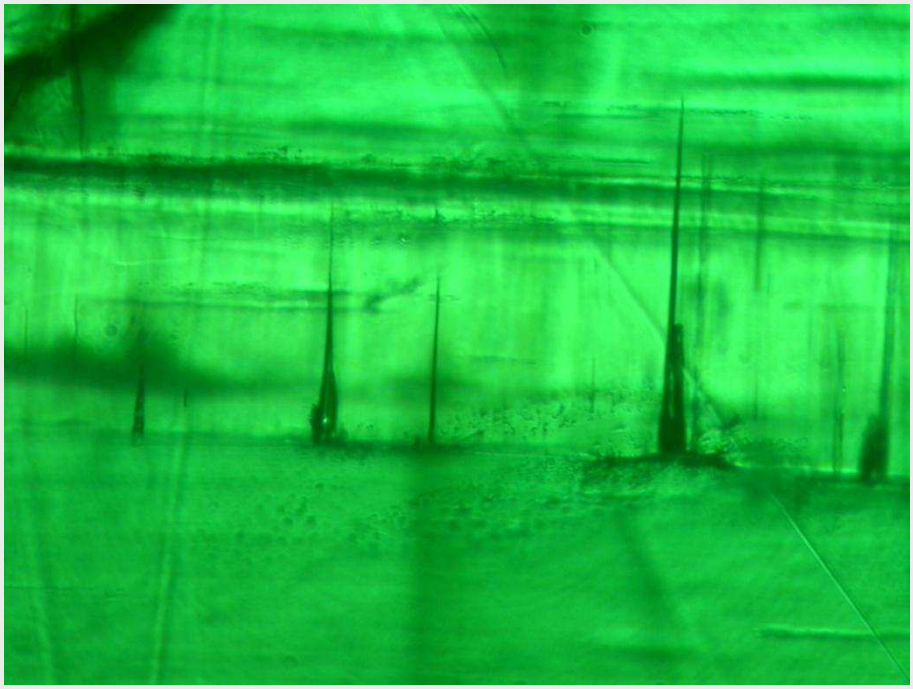


Figure 8: At higher magnification, the parallel growth planes in figure 7 showed abundant nail-head spicules; also note the two-phase inclusions at the broader end. Photomicrograph by G. Choudhary, magnified 35x.

Also present were liquid "fingerprints," flat reflective films oriented along the basal plane, and angular zoning. Further confirmation came from FTIR spectra taken in various directions, which showed the strong peak at 5270 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.

Natural 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 impurity (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 possibility of these being elongated needles cannot be ruled out (Chapman, 1992). The authors have observed similar inclusions in a few flux-grown emeralds, where the spicules broke the surface and appeared to be filled with a black material (again figure 1, right).

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 gemmologist.

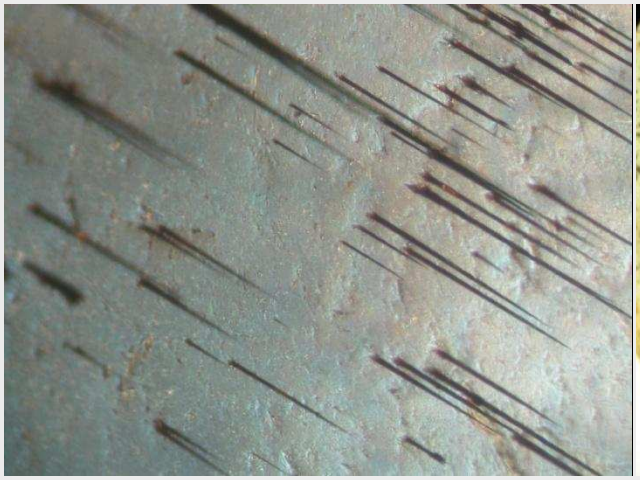


Figure 9: Near the surface of this spinel pebble, a number of long, conical etch features resemble oriented spicule inclusions. Photomicrograph by C. Golecha, magnified 30x



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 30x

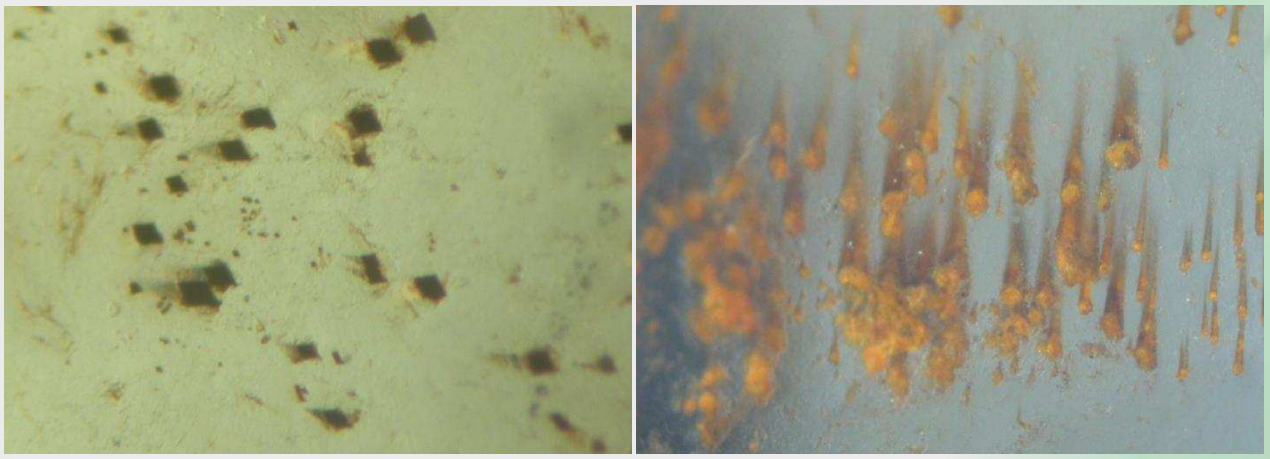


Figure 11: These spicule-like inclusions in spinel appear to be etch channels. Their cross-sections varied from rhomboid (left) to hexagonal (right). Also note the brownish epigenetic filling material (right). Photomicrographs by G. Choudhary; magnified 30x (left) and 45x (right).

Natural Diamond

Because diamonds form at conditions of very high temperature and pressure, they do not contain fluid inclusions that are resolvable with a gemmological 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.

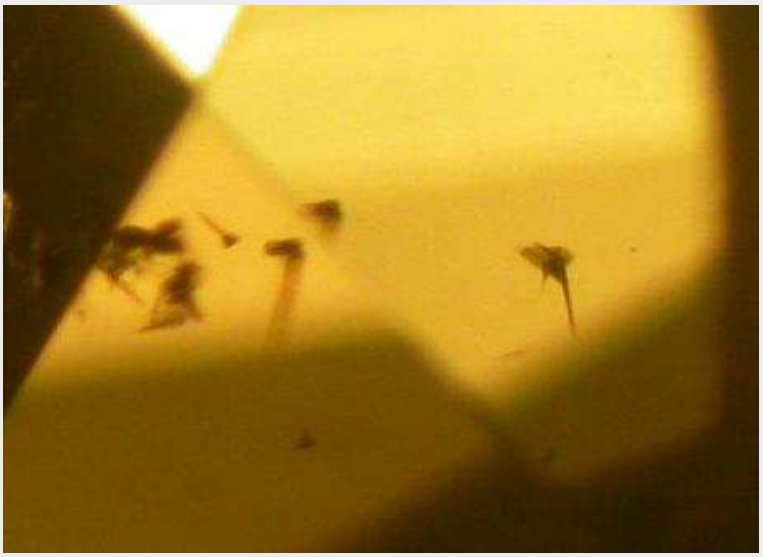


Figure 12: This diamond contains some nail-head spicule-like inclusions that are actually crystals with stress cracks at one end. Given the formation conditions of diamond, true nail-head inclusions are not possible. Photomicrograph by C. Golecha; magnified 30x

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 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.

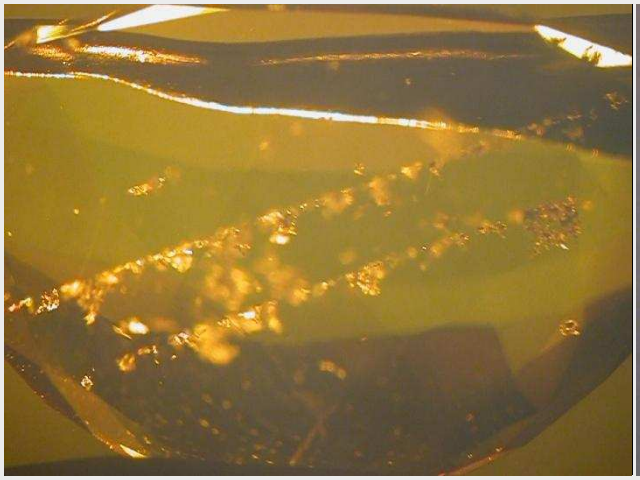


Figure 13: Inclusions are concentrated along two parallel planes in this faceted quartz. The pattern resembles a common inclusion scene in synthetic quartz: "breadcrumbs" along a seed plate. Photomicrograph by C. Golecha , magnified 15x



Figure 14: The aggregation patterns of these inclusions in quartz strongly resemble nail-head spicules at certain orientations. Photomicrograph by C. Golecha, magnified 35x



Figure 15: At higher magnification, the inclusions in figure 14 proved to be aggregates of natural mineral, possibly muscovite. Photomicrograph by C. Golecha, magnified 45x

Further analysis with FTIR spectroscopy showed absorptions in the 3600-3000 cm^{-1} region, along with a strong peak at 3483 cm^{-1} that is characteristic for natural crystalline quartz. Similar spectra were recorded 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 spicule-like inclusions and the concentration of the inclusions

along a defined plane could have lead to the stone's misidentification as synthetic without careful examination.

CONCLUSIONS

Nail-head spicule inclusions have long been associated with hydrothermally grown synthetic gem materials. Such inclusions indicate a 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 us reminders of the importance of not relying on any one feature for the identification of a gem material.

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