CAUSES OF IRIDESCENCE IN

NATURAL GEM MATERIALS

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ABSTRACT

Iridescence is so highly prized in gem materials that gemologists have fabricated techniques that artificially impart a play of colors to solids. Iridescence may be caused by one of two processes: the interference of light by thin films or through diffraction by periodic substructures. Therefore, manmade gems with rainbow effects can be created by coating non-iridescent crystals with thin metal films (as with “flame-aura” quartz), or by synthesizing solids with modulated microstructures. However, naturally iridescent gemstones are rare and therefore highly valued. For this study, we have explored the cause of iridescence in a gem quality hematite from João Monlevade, Minas Gerais, Brazil and natural quartz crystals from the Jalgaon District, India.

Iridescent hematite, also known as “rainbow hematite”, was investigated with field-emission scanning electron microscopy, X-ray energy-dispersive spectroscopy, atomic force microscopy, and synchrotron X-ray diffraction. This study reveals that rainbow hematite has a microstructure that consists of spindle-shaped hematite nanocrystals with minor aluminum and phosphorus. The nanorods are 200-300 nm in length and 50-60 nm in width, and they are arranged in three orientations rotated by 120° with respect to each other and stacked layer by layer to form the bulk crystal. The distances between adjacent parallel spindle-shape particles in the same layer are in the range of 280 – 400 nm, generating a diffraction grating for visible light. The sub-structure is apparent on all freshly fractured surfaces, indicating that it is not merely an exterior surface coating. Rather, we interpret the periodic sub-structure as the result of crystal growth by oriented aggregation of hematite nanorods.
The iridescent quartz specimens occur as euhedral quartz crystals within chalcedonic geodes that filled cavities in the Deccan Trap basalts. The quartz crystals exhibit strongly expressed terminal faces, and iridescence is visible only on the smaller \( \{011\} \) faces and not on the \( \{101\} \) faces. Our scanning electron microscopy ruled out the existence of a thin film on the iridescent faces and suggested a fine-scale substructure. AFM imaging revealed that the iridescent \( z \) faces exhibit periodic ridges, and the distance between the ridges varies from 400 nm to 700 nm, generating a diffraction grating for visible light. On the other hand, the non-iridescent \( r \) faces are quite flat with no apparent ridges observable by AFM. We interpret the modulated surface topography on the \( z \) faces as the result of preferential dissolution. Previous investigators have hypothesized that the iridescence in quartz is associated with Brazil twinning. Thus, we employed focused ion beam lift-out and transmission electron microscopy to determine whether Brazil twins were concentrated at the ridge boundaries. However, instead of Brazil twin boundaries, we observed periodic planar defects parallel to the \( c \) axis. The regularly spaced planar defects might have formed by the episodic injection of silica-rich fluids into the host rock cavities (leading to periods of crystal growth), followed by periods of quiescence and crystal stasis. The planar defects formed by the incorporation of fluid inclusions on crystal faces at the onset of a new growth cycle.
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INTRODUCTION

Iridescent solids exhibit a change in surface color in response to variations in the angle of illumination, and this behavior is so highly prized in gem materials that gemologists have fabricated techniques that artificially impart a play of colors to solids. Moreover, iridescence in natural materials has attracted attention in diverse research fields, including photonics, evolutionary and developmental biology, materials science, and computer graphics (Meadows et al. 2009). For example, studies of the physics of iridescence have informed the design of cosmetics, paints, and anti-counterfeiting devices (Parker & Townley 2007; Vigneron et al. 2007). In this thesis, we review the two physical mechanisms by which iridescence occurs in natural materials, and we report the results of our investigations into the causes of iridescence in naturally formed crystals of hematite and quartz.

BACKGROUND: PHYSICS OF IRIDESCENCE

Iridescence may be caused by the interference of light either by thin films or by repetitive substructures. I will discuss these mechanisms in turn.

Thin-Film Iridescence. Thin-film interference effects are seen when a liquid or solid is coated by a thin film that is nanometers to micrometers in thickness. When incident light strikes the surface of a thin film, some of the light is transmitted and some is reflected. After the transmitted light impinges the lower boundary of the film, some or all of that
light wave may be reflected. The two waves reflected from the thin film’s upper and lower boundaries interfere with each other to produce a new light wave (Fig. 1).

If the source is monochromatic, the interference pattern generated by the reflected light waves will appear as bright and dark bands. If the incident light is polychromatic or white, then the interference pattern will be iridescent. The colors associated with the iridescence will depend on the thickness of the thin film and of the relative refractive indices of the film and substrate. Refractive indices play a role because reflected light waves experience a phase shift of 180° when the light wave travels through a film with a refractive index lower than that of the substrate.

If, after its extended travel path and possible phase shift, the ray that reflects off the thin film-substrate boundary (Ray 2 in Fig. 1) is exactly in phase with the ray that reflects only from the thin film surface (Ray 1 in Fig. 1), the two reflected waves will constructively interfere and combine into a light ray with high intensity; otherwise, the emergent waves interfere destructively and are cancelled. When the incident light is polychromatic, only one wavelength will be reinforced by constructive interference for a given reflection angle. As a result, thin film interference is selective for specific wavelengths depending on the angle. Steeper angles select for longer wavelengths, and thus a full rainbow spectrum is evident when the film is viewed from a perspective that samples a range of reflection angles.

Among minerals, thin-film iridescence effects are well-documented in bornite and in fire obsidian. Bornite (Cu₅FeS₄) is an important ore mineral for copper, and it exhibits a bronze color on freshly fractured surfaces. However, a characteristic peacock tarnish
quickly forms on fresh surfaces after exposure to the atmosphere (Fig. 2). The coating on the bornite surface comprises a layer of hydrated iron oxide and a layer of copper sulfide beneath it (Vaughan et al. 1987, Buckley et al. 1983). Silver, existing as a minor element in the bornite structure, also tends to congregate within the copper sulfide layer during oxidation (Buckley et al. 1983). Iron hydroxide populates the top layer because Fe$^{3+}$ is more stable in octahedral coordination, leaving the copper in the underlying sulfide layer in its preferred trigonal coordination (Vaughan et al. 1987). The non-uniform thickness of the oxidized layer and the variation in chemical composition give rise to the peacock hue (Buckley et al. 1983).

Fire obsidian, a rare variety of clear volcanic glass, also reveals thin-film iridescence (Fig. 3). Field-emission scanning electron microscopy (FESEM) and electron backscatter diffraction analyses have revealed the existence of thin layers formed by concentrated nanoscale magnetite crystals within the host glass. The 300- to 700-nm layers have a higher refractive index than does obsidian, and they generate reflective films that give rise to the iridescence (Ma and Rossman 2007).

**Iridescence from Diffraction Gratings.** The dispersion of light also can be induced by interference effects that arise from natural diffraction gratings within minerals. The interaction with the diffraction grating can be purely reflective from surface grooves (Fig. 4), or it can be transmissive through modular substructures with periodic variations in refractive indices. The equations that govern the conditions for transmissive diffraction through parallel slits differ from those that describe reflective iridescence, but they
similarly define the conditions for constructive interference of emergent light rays
(Kinoshita 2008).

Mineralogical iridescence that is produced by intrinsic diffraction gratings is of
particular interest, because the substructures that create the gratings often yield insights
into crystal growth processes. These in turn may allow geologists to quantify aspects of a
mineral’s crystallization history, and they can suggest to materials scientists new
pathways towards the self-assembly of hierarchically ordered structures. Precious opal is
the most well-known mineralogical example of iridescence produced by a natural
diffraction grating (Fig. 5). Precious opal is composed of silica nanospheres, which are
remarkably uniform in size and consistently closest-packed (Sanders 1964). This
substructure forms by the gravitational settling of silica nanospheres in quiescent aqueous
solutions that are supersaturated with respect to silica (Darragh et al. 1966). The
refractive index of the transparent silica spheres differs from that of the hydrous
interstices between them, giving rise to a three-dimensional optical diffraction grating
whose iridescence is determined by sphere size and refraction at the surface (Sanders,

Labradorite, a variety of plagioclase whose composition lies between those of Na-
rich albite and Ca-rich anorthite, also exhibits a strong iridescence (Fig. 6). Transmission
electron microscopy reveals that labradorite consists of exsolution lamellae that are
alternately Na- and Ca-rich, giving rise to oscillations in refractive index with a
periodicity of ~100 nm (Nissen 1971; Cliff et al. 1976; McConnell 1974; Miura and
Tomisaka 1978). These exsolution lamellae emerge as Na\(^+\) and Ca\(^{2+}\) cations segregate
during the cooling of plagioclase crystals, and their compositions and sizes can be used to infer cooling rates (Laves et al. 1965). Moonstone (a pearly white gemstone consisting of orthoclase and albite, also characterized by a play of light) and peristerite plagioclase (An$_2$ - An$_{17}$) are similarly characterized by diffractive iridescence from regularly spaced exsolution layers (Fleet 1963, 1965).

Iris agate is a gem quality chalcedony with dense concentric bands that disperse white light into rainbow colors (Fig. 7). As with labradorite, iris agate contains oscillating layers with variations in grain size, defect concentration, and water content (Taijing and Sunagawa 1994; Heaney and Davis 1995). The wavelength of the oscillation is on the order of 0.1 to 5 um, and the change in refractive index results from modulations in concentrations of quartz and moganite (SiO$_2$ polymorphs). Heaney and Davis (1995) observed a hierarchical superimposition of these modulations at four discrete length scales (from the unit-cell to the centimeter scale), and the mechanisms that generate this fractal symmetry remain unknown.

IRIDESCENT HEMATITE

Hematite specimens that display iridescence are frequently described by the names “rainbow hematite” and “turgite”. The latter term originated with the German mineralogist Rudolph Hermann, who coined “turgite’ in 1844 to describe iron hydroxide specimens found near the Turginsk River in the Ural Mountains (Hermann 1844). However, “turgite” was discredited as a distinct mineral name in the 1920s based on thermogravimetric (Posnjak and Merwin 1919) and X-ray diffraction (Böhm 1928;
Palache et al. (1944) studies that purportedly identified such specimens as mixtures of microcrystalline hematite ($\text{Fe}_2\text{O}_3$) and goethite ($\text{FeOOH}$) or amorphous Fe hydroxide. Nevertheless, “turgite” has been retained by the mineral collecting and gem community, and an internet image search using the term reveals stunning examples of naturally iridescent iron (hydr)oxide minerals.

Rossman and Ma (2003) analyzed rainbow hematite specimens from Brazil, Mexico, Italy, and several sites in the United States, and they have presented their results online and in Nadin (2007). Using field-emission SEM, they report that the hematite specimens were coated with a “thin film” of rod-shaped nanocrystals, each measuring 5 to 35 nm in thickness and hundreds of nm in length. These nano-crystals were oriented in three directions, rotated by $120^\circ$ with respect to each other, and they formed a grid-like network. The nano-crystals were too small for Rossman and Ma to determine precise chemical compositions, but their energy dispersive spectroscopy (EDS) revealed high concentrations of Al and P in a ratio that varied from 2.2 to 3.8. They note that “the minute crystals have failed to produce either an X-ray powder diffraction pattern, an electron back-scatter diffraction pattern, or a Raman spectrum.” They inferred that the rod-like nanocrystals represented a new mineral, but that a full characterization fell below the resolution of their instruments. Nevertheless, Rossman and Ma were the first to document a connection between the surface nanorods and the iridescence displayed by some hematite specimens.

Using more sophisticated analytical techniques than were available to Rossman and Ma when they performed their study, we sought to determine whether iridescence in
rainbow hematite can be attributed to thin-film effects involving Al phosphate phases, as they hypothesized. For our investigation, we employed a combination of light optical microscopy, X-ray diffraction (XRD) and Rietveld analysis, FESEM, and atomic force microscopy (AFM).

**EXPERIMENTAL METHODS**

**SPECIMEN DESCRIPTION**

**Sample characteristics.** The iridescent hematite samples we purchased for this research originated from the Andrade Mine in João Monlevade, Minas Gerais, Brazil. Rock Currier (2012) has posted images of the iridescent hematite outcrop at the Andrade Mine (Fig. 9). The Andrade Mine serves as a major source for hematite (Rosière et al. 2011), and the iridescent hematite occurs as a distinct vein in this ore (Mindat.org). Fifteen tons of this iridescent material has been imported to the United States for sale in the gem/mineral market (Currier 2012).

**Geological setting.** The Andrade iron ore deposit is located in the eastern high strain domain of the Quadrilátero Ferrífero district (Fig. 10, Fig. 11) in the southern part of the São Francisco Craton in Brazil (Almeida 1977). The Quadrilátero Ferrífero (the “Iron Quadrangle”) is so-called because the Paleoproterozoic metasediments in the Minas Supergroup exhibit a quadrangular arrangement of synclines (Rosière et al. 2001, 2008). The Caraça, Itabira, Piracicaba and Sabará groups are four sequences of the Minas Supergroup rocks (Dorr 1969) (Fig. 12). The iron ore deposits are located in
metamorphosed banded-iron formations in the Cauê Formation, Itabira group. The Andrade Mine is located in a contact-metamorphic zone. Post-tectonic recrystallization gave rise to the mosaic granoblastic texture that is typical of iridescent hematite (Rosière et al. 2001).

**Scanning Electron Microscopy (SEM) / Energy Dispersive Spectroscopy (EDS).** SEM and EDS were used to characterize surface topography and composition of the specimen from the Andrade mine in Brazil. As iridescence was evident even from freshly fractured surfaces of the Andrade specimen, we removed a flake from the specimen and fixed the flake to an SEM mount using carbon tape such that the flat iridescent surface was parallel to the surface of the SEM mount. We used a FEI Nova NanoSEM 600 field emission scanning electron microscope (Czech Republic) outfitted with an Oxford instruments X-Max (Model 51-XMX1105) EDS detector in the Materials Characterization Laboratory (MCL) at Penn State University to examine the iridescent hematite. Nano-scale secondary electron images were obtained at 5 kV. EDS data were processed using the Oxford Instruments NanoAnalysis Aztec software (version 2.4).

**Atomic Force Microscopy (AFM).** To obtain high-resolution surface topography, we used a Bruker Icon AFM (MCL, Penn State University) in PeakForce Tapping (PFT) mode with ScanAsyst® image optimization technique. In PFT mode, the cantilever is brought in and out of contact with the surface. Because PFT algorithms can precisely control the instantaneous force interaction, it allows the use of reduced forces in the
imaging process. In this way, both fragile probes and samples can be protected without decreasing image resolution. The ScanAsyst automatically adjusts the appropriate parameters (such as set points, feedback grains, and scan rates) during the scan and continuously monitors image quality. The AFM probe used in this analysis was a Bruker ScanAsyst-Air probe, which has a silicon tip on a nitride lever. The front angle of the tip is 15° and the back angle is 25°. For AFM imaging, the peak force set point ranged from 1 to 4 nN and the scan rate was 1 to 0.5 Hz, with 512 data points per line in each scan. The NanoScope Analysis software (version 1.50) was used to process the AFM data.

X-ray diffraction. Hematite was powdered in an agate mortar and pestle under acetone. When dry, the powder was loaded into a 0.7 ID polyimide capillary for X-ray diffraction. Synchrotron X-ray diffraction data were collected at the GeoSoilEnviroCARS (GSECARS) 13-BM-C beamline at the Advanced Photon Source (APS), Argonne National Laboratory (ANL). The X-ray wavelength was 0.8315(4) Å, and the detector - sample distance was 100.469(1) mm. A MAR165 CCD camera was used to collect XRD data. The diffraction pattern was integrated into intensity versus 2θ plots using the program Fit2D with a polarization factor of 0.99 (Hammersley 1996).

Structure Refinement. Rietveld refinements (Rietveld 1969) were performed for the synchrotron XRD data collected from both a lanthanum hexaboride (LaB₆) standard and from the iridescent hematite from Andrade, Brazil. We used the EXPGUI interface (Toby 2001) of the general structure analysis system (GSAS) (Larson and Von Dreele
To obtain instrumental broadening parameters, we refined the structure of LaB₆ using the structure reported in Ning and Flemming (2005) as the starting parameters. In the first stage of the refinement, we allowed only unit-cell parameters, background, the zero position, and the scale factor to refine using a range in 2θ from 6.5° to 36° (d_{hkl} = 7.3 Å to 1.3 Å). Temperature factors (U_{iso}) for both La and B atoms were set to 0.002. A cosine Fourier series polynomial with 12 profile terms fitted the synchrotron XRD background intensities. The peak profile was best fitted with a pseudo-Voigt function described by Thompson et al. (1987) and corrected for asymmetry by Finger et al. (1994). The peak cut-off value was 0.0001 and we let both Gaussian (GU, GV, GW) and Lorentzian (LX, ptec) profile parameters refine. The final goodness-of-fit parameters for this refinement indicated an excellent match between our calculated and observed patterns for the LaB₆ standard, with $\chi^2 = 0.8290$, $R_{wp} = 0.0236$, and $R_{Bragg} = 0.00507$.

The values of the Cagliotti functions from the LaB₆ refinement provided instrumental broadening parameters for the subsequent refinement of hematite. For the hematite refinement, the initial structure parameters came from Blake (1966). Using a 2θ range of 11.5° to 37.5° (d_{hkl} = 4.1 Å to 1.3 Å), we refined the background using a cosine Fourier series polynomial with 8 profile terms. For the peak profile, the Cagliotti values derived from the LaB₆ refinement (GU= 0.199000E+03, GV= -0.611000E+02, and GW=0.257000E+02) were held constant, and we allowed only the Lorentzian (LX) term to refine. After the scale factor, background, unit-cell parameter, zero position, and profile terms had converged, the atom positions and the Fe occupancy were refined. With only Fe in the octahedral site, the occupancy converged to 93.9(4)%. Since our EDS
results suggested that Al also was present in the Andrade hematite, we included Al on the same site as Fe with the constraint that the occupancies for Al and Fe would sum to 1. The inclusion of Al improved the goodness-of-fit, with $R_{\text{Bragg}}$ decreasing from 0.1164 to 0.1055.

RESULTS

**Reflected Light Microscopy.** When iridescent hematite from the Andrade Mine was viewed using reflected light optical microscopy at low magnification, the sample appeared as a composite of gray hematite platelets with strong silvery luster, and the platelets varied in diameter from 150 to 250 μm (Fig. 13). The lenticular to equidimensional texture is consistent with the mosaic granoblastic fabric described by Rosière et al. (2001) in their study of the Andrade Mine hematite (Fig. 14). Even when the iridescent Andrade hematite was freshly fractured, all surfaces exhibited intense rainbow colors, leading us to interpret the iridescence as a bulk behavior rather than as the result of a surface coating.

**XRD and Rietveld refinement.** Conventional X-ray diffraction of ~15 samples that were labeled “turgite” in the Smithsonian Mineral Research Collection revealed that most were iridescent goethite rather than hematite. Nevertheless, some iridescent hematite specimens were nearly phase-pure, as was the specimen from the Andrade mine in Brazil that we purchased. Because of the high crystallinity of the Andrade specimen, we focused our characterization on it. The final Rietveld refinement results based on our
synchrotron XRD are presented in Figure 15 and the parameters are presented in Table 1. Atom positions and occupancies are included in Table 2. Rietveld refinement suggests the occupancy of Fe in the Andrade hematite was 0.879(7) and that of Al was 0.121(8).

**Surface Chemical Composition.** The elemental composition of the iridescent surface of the Andrade hematite as obtained by EDS is presented in Table 3. As was reported by Ma and Rossman (2003), the iridescent surfaces contain minor Al and P in addition to the major Fe and O. Whereas Ma and Rossman (2003) interpreted the Al and P as constituents of a separate nanophase, however, we argue that Al and P are minor impurities within the hematite structure. Al commonly substitutes for Fe in natural hematite, and synthetic hematite with minor Al has been extensively studied (De Grave et al. 1985; Gialanella et al. 2010; Schwertmann et al. 1979; Liu et al. 2011; Stanjek et al. 1992). Moreover, our refinement of the Andrade hematite with only Fe in the octahedral site yielded an occupancy of 0.94 rather than 1.00, indicating the presence either of vacancies or of a light element substituting for Fe. Since the refinement of the Andrade hematite structure with Al and Fe in the octahedral site yielded an improved goodness-of-fit, and since our synchrotron XRD of the Andrade hematite offered no evidence for any phases other than hematite, it seems most reasonable to conclude that Al is substitutional.

Similarly, we suggest that P also occurs as a trace impurity, either as an interstitial or perhaps within the octahedral Fe site. Like Al, P has been reported as an impurity within natural hematite, and P-containing hematite can be grown synthetically (Colombo et al. 1994; Galvez et al. 1999). Perhaps significantly, previous studies have revealed that
P preferentially attaches to the prismatic (100) and (110) faces of hematite rather than the (001) and (104) basal faces (Colombo et al. 1994; Torrent et al. 1994). This favored mode of attachment may control the particle morphology of hematite.

**Surface Morphology of Iridescent Hematite.** When the iridescent surfaces of the Andrade hematite platelets from a freshly fractured surface were analyzed at low magnification (3,000x) using the scanning electron microscope, the surface appeared flat and smooth (Fig. 16). As was first described by Ma and Rossman (2003), however, at sub-micron resolution, spindle- and rod-shaped nanocrystals arranged with 3-fold symmetry became apparent. The spindle-shape particles were 200-300 nm in length and 50-60 nm in width. These oriented nanoparticles were clearly apparent on freshly fractured surfaces, and they occur as stacked sheets. Thus, they are representative of the bulk crystal and are not merely coatings or etch features (Fig. 16).

This three-fold grid structure was revealed even more clearly by AFM imaging (Fig. 17). Although individual particle widths were less than 100 nm, the average distances between two parallel nanorods within the same layer were in the range of 280 – 400 nm, which is the same order of magnitude as the wavelength of visible light.
DISCUSSION

A Natural Diffraction Grating Generated by Oriented Aggregation. Based on our observations, we argue that the iridescence in hematite results from the interference of visible light from stacked nanorods that constitute the bulk crystal structure. Consequently, the iridescence is not a thin film phenomenon, as previously suggested by Ma and Rossman (2003). Instead, the rainbow effect is an analog to the modulated substructure observed in opal. Whereas opal consists of amorphous spheres, the nanorods in iridescent hematite are crystalline, but with a composition close to (Fe$_{1.76}$Al$_{0.24}$)O$_3$, based on our EDS and Rietveld analyses.

The ordered substructure, in which nanorods are rotated by 120° with respect to each other and are stacked layer by layer, strongly suggests that the bulk crystals assembled by oriented aggregation (Banfield 2000). In this crystal growth mechanism, individual primary crystallites nucleate and develop to a specific dimension, and then the crystallites assemble in a common crystallographic orientation through spontaneous self-organization (Penn and Banfield 1998; Niederberger et al. 2006). Hematite has a particular tendency to form through an aggregation-based process. Oriented aggregation of pseudocubic, ellipsoidal, and disc-shaped (sometimes interpenetrating) crystals have been described from hematite synthesis in previous research (Sugimoto et al. 1993; Ocana et al. 1995; Niederberger et al. 2002).

Here we propose that iridescent hematite forms through two stages of growth. During the first stage ultrafine crystallites grow as spindle-shaped nanoparticles, perhaps as the result of the incorporation of Al and/or P in preferred orientations within the
crystal structure. The shape of hematite particles can be affected by both Al and P (Barron et al. 1988; Colombo et al. 1994; Galvez et al. 1999; Ocana et al. 1995; Reeves and Mann 1991; Schwertmann et al. 1979; Stanjek and Schwertmann 1992; Sugimoto et al. 1993, 1998). In particular, it has been reported that hematite will exhibit an ellipsoidal shape if it is precipitated from a solution with phosphate (Barron et al. 1988; Colombo et al. 1994; Galvez et al. 1999; Ocana et al. 1995; Reeves and Mann 1991; Sugimoto et al. 1993, 1998).

In the second stage of growth, the nanorods assemble such that their crystallographic c-axes are aligned parallel to each other. Because hematite has trigonal symmetry with S.G. R-3c, any two crystals can align in one of three orientations with equivalent energetics of attachment, giving rise to the three-fold rotational orientations seen in Figs. 16 and 17. This interpretation suggests that the particles are elongated perpendicular to c, in contrast to reports that ellipsoidal hematite is lengthened along c (Colombo et al. 1994). Whereas most crystals that form by oriented aggregation eventually fill all spaces, the iridescent hematite from the Andrade mine exhibits an arrested growth, with significant retention of void space. The resulting porous and grid-like substructure constitutes a diffraction grating for visible light. Since iridescent hematite specimens from other localities reveal similar substructures (Ma and Rossman 2003), we suggest that iridescent hematite specimens from other localities also result from arrested oriented aggregation.
IRIDESCENT QUARTZ

Naturally iridescent quartz has been recognized for over a century, and it is known by many names: iris quartz, rainbow quartz, schiller quartz, anandalite and adularaescent quartz. Petrov and Tanaka (2011) have published online the only detailed review of the history of iridescent quartz, and our introduction draws on their survey. Although there is some ambiguity regarding the source locality for this material, most of the iridescent quartz on the market derives from the Deccan Traps in an area west of Burhanpur, Madhya Pradesh State, near the border with the Jalgaon district, Maharashtra State, India. Indeed, the first known reference to iridescent quartz originates with the gift of an iris quartz cluster to the British Museum of Natural History in 1860 from an engineer who worked on the construction of the Indian railroad. In addition, iridescent quartz has been reported from the state of Washington (USA), Uruguay, Brazil, Germany, Turkey, and Morocco.

The iris quartz in the British Museum (#55795) is described by vom Rath (1873) and Scharff (1875), and their descriptions precisely match the characteristics of the specimens examined in the present study (Fig. 18). They occur as clusters of euhedral quartz crystals that evidently filled vugs within the Deccan Trap basalts. At the base of these clusters is a 0.2 - 0.4cm thick rind of chalcedony, which abruptly transitions to euhedral quartz, as is characteristic of geodes in Brazil, Germany, and many other localities. The euhedral quartz crystals exhibit strongly expressed terminal faces, and iridescence is visible only on the smaller z \{011\} faces and in zones closely surrounding them (Fig. 19), but not on the dominant r \{101\} faces. The colors vary from red to blue depending on the angle of incident light. Etch pits are frequently located on both the z
and $r$ faces, and iridescence is particularly strong in the etch pits on the $z$ faces. However, rainbow colors are not visible in broken portions of otherwise iridescent $z$ faces. The prismatic $m\{100\}$ faces are poorly developed in these crystals. A single quartz crystal is about 0.5-1mm in length.

Until 2009 or so, one importer in Japan controlled the market for iridescent quartz, importing several tons of material, but since that time production has exceeded that importer’s capacity and the market has opened internationally (Petrov and Tanaka 2011). Much of the quartz produced is of relatively low quality, but a brilliant small cabinet piece exhibiting strong colors can easily cost up to $2,000. In light of the growing market for iridescent quartz specimens produced artificially through the deposition of thin films of various metals (as with “aurora quartz”), gem buyers were initially skeptical of the authenticity of the iridescent quartz from India. Nevertheless, Petrov and Tanaka (2011) report that the Japanese gemologist Koichi Iida (Japanese Saitama Jewelry Institute) examined iris quartz from India and confirmed that the iridescence is of natural origin. In light of the historical evidence, the validity of iridescent quartz from the Deccan Traps of India as a naturally occurring material seems unimpeachable.

C.V. Raman, the 1930 Nobel Laureate in Physics, is the only person of whom we are aware who has published a scientific analysis of iridescent quartz. Raman (1950) examined specimens that he obtained from a jeweler in Bombay using reflections of polarized light and also by visible light spectroscopy. He recognized that the iridescence is not the result of a superficial film coating the quartz surface, as he observed iridescence occurring outside an iridescent face due to reflection within the crystal. Rather, he attributed the iridescence to “a layer of material which lies below the surface of the
Based on his spectroscopic study, Raman concluded that the iridescence is caused by “a stratified medium consisting of a great many parallel layers of extreme thinness”, and he estimated the thickness of each layer to be 0.34 µm. He further hypothesized that the striations are regularly spaced polysynthetic Brazil twins oriented parallel to the minor rhombohedral $z$ faces. Polysynthetic Brazil twins consist of alternating layers of right- and left-handed quartz, and they are particularly common in amethyst (Baran et al. 1987; McClaren 1967; Pitkethly and McClaren 1982; Schmetzer 1987; Schlössin 1965; Taijing and Sunagawa 1990). Raman puzzled over the absence of iridescence in Brazil-twin Amethyst amethyst and recognized that the mere change in chirality at the twin boundary was insufficient to generate the kind of interference effects that would lead to a Schiller effect. Consequently, he speculated that the twin boundaries in the Indian iris quartz were associated with “extremely thin layers of impurity material.”

Raman was unable to confirm his deductions regarding the role that Brazil twins play in the generation of iris effects in quartz. Moreover, several studies of quartz chirality have demonstrated that Brazil twin boundaries occur parallel to the rhombohedral planes ($\{101\}$ and $\{011\}$), but a specific Brazil twin boundary is never oriented parallel to a rhombohedral face with the same indices (so that the (011) face of quartz is not underlain by Brazil twin boundaries parallel to (011), as Raman posited) (Balakirev et al. 1975). Using more sophisticated analytical techniques than were available to Raman when he performed his study, we sought to determine the cause of iridescence in this quartz. For our investigation, we employed a combination of scanning
electron microscopy (SEM), atomic force microscopy (AFM), focused ion beam milling (FIB) and transmission electron microscopy (TEM).

**EXPERIMENTAL METHODS**

**Specimen.** We purchased two specimens of iris quartz clusters from Georges Claeys (Geonic Mineralen Collectie) at the Tucson Gem and Mineral Show (Fig 18). Mr. Claeys reported that he obtained the specimens from a dealer who collected them from the Jalgaon District, India. The physical attributes of the two specimens match precisely the descriptions of other iris quartz clusters from this locality, and we feel confident of its provenance.

**Scanning Electron Microscopy (SEM)) / Energy Dispersive Spectroscopy (EDS).** For the first stage of our study, we used an FEI Quanta 200 environmental scanning electron microscope (Materials Characterization Laboratory, Penn State) to map the surface topography of both iridescent and non-iridescent pyramidal faces of iris quartz. Selected quartz crystals were pried from the cluster, cleaned in methanol, and attached to a standard SEM aluminum mounting stub with double-sided carbon tape. Because quartz is not conductive and was not coated by carbon, SEM images were taken at low vacuum with an accelerating voltage of 20 kV. An Oxford instruments INCAx-act (Model 51-ADD0001) EDS detector on the SEM was used for surface chemical analysis. EDS data were analyzed using Oxford Instruments NanoAnalysis Aztec software (version 2.4). We used three different accelerating voltages (20kv, 10kv and 5kv) to acquire spectra for the same sites.
**Atomic Force Microscopy (AFM).** We next employed atomic force microscopy (AFM) to construct three-dimensional topographic maps of the pyramidal faces with high resolution. We removed two quartz single crystals from the cluster and placed one iridescent face and one non-iridescent face oriented parallel to the flat stage in the AFM. A PeakForce Tapping model with ScanAsyst® (MCL, Penn State University) was used for these surface measurements. The peak force set point ranged from 2.5 to 7.5 nN for AFM imaging, and the scan rate was 1 to 0.5 Hz. The AFM probe used in these analyses was a Bruker ScanAsyst-Air probe, which has a silicon tip on a nitride lever. The front angle of the tip was 15° and back angle 25°. The data were collected as line scans with 512 points per line, and 256 lines were collected in total. The NanoScope Analysis software (version 1.50) was used to process the AFM data, and the average distance between two adjacent ridges was calculated through a Fast Fourier Transform (FFT) algorithm using MATLAB (MathWorks, Inc).

**Focused Ion Beam Milling (FIB) and Transmission Electron Microscopy (TEM).**

In order to ascertain any relationship between iridescent behavior and Brazil twinning, we prepared a foil for TEM using focused ion beam lift-out. Focused ion beam milling was performed with an FEI Helios NanoLab 660 FIB (MCL, Penn State University). A single quartz crystal was removed from a cluster and coated with conductive carbon paint to avoid charging. After the sample was mounted within the FIB, an amorphous carbon strip was deposited over the area of extraction to protect the foil and preserve the surface structure during the milling process (Fig. 20). A Ga⁺ ion beam was used to excavate the material on both sides of the foil. The voltage of the Ga beam initially was 30 kV and
then reduced to 5 kV and lastly 2kV for the final thinning. Beam currents operated at 0.23 nA for the amorphous carbon deposition, 21 nA and 9.3 nA for intermediate milling stages, and finally 2.5 nA for milling prior to foil lift-out.

Next, the foil was soldered to a glass probe tip and deposited onto a V-shaped TEM half-grid post. Milling the sample on the grid began with a 30 kV ion beam with the stage tilted to 53.5°. As an angle of 52° is normal to the ion beam, that yielded an over-tilt of 1.5°. This over-tilt was increased to 3.5° for 5 kV milling, and finally to 5° for 2 kV milling. The thickness of the final foil was less than 100 nm to allow electron transparency, and the areal dimensions of the quartz foil were ~7 μm × 4.5 μm. The entire milling and extraction process was monitored by secondary electron imaging. The milled sample was loaded in a Philips double-tilt holder with a Be stage and examined using a Philips 420 TEM at 120 kV (MCL, Penn State University).

RESULTS

Surface Morphology and Composition. Topographic differences between the iridescent z and the non-iridescent r faces were apparent in SEM images of the crystals (Fig. 21). In particular, a high magnification BSE image of an apparent etch pit across both faces reveals that the iridescent z face featured parallel ridge and valley structures (Fig. 21 Bottom), whereas the non-iridescent face was relatively flat and smooth. The SEM images suggested that the average distances between adjacent ridges fell below a micron, but AFM analysis (see below) allowed for more rigorous quantification. Although the striations were more pronounced in the apparent etch pits, an examination of the unetched surfaces also revealed a substructure that consisted of alternating lamellae (Fig. 22).
Compositional analyses obtained by EDS on the iridescent faces indicated no trace metal concentrations (such as Au or Ti), and, therefore, offered no evidence of a thin film coating (Fig. 23). Minor amounts of Al were detected, but Al commonly substitutes for Si in natural quartz (Heaney 1994) and was present in equal amounts on both the iridescent and non-iridescent faces.

Atomic force microscopy confirmed the existence of extremely periodic ridge-and-valley structures on the iridescent $z$ faces and the absence of such surface modulations on the non-iridescent $r$ faces (Fig. 24). The ridges are oriented parallel to the edge between the $m$ and $z$ faces. We calculated an average distance between adjacent ridges by processing the AFM images using a Fast Fourier Transform (FFT) algorithm using MATLAB. As the ridge shapes were not identical from top to bottom, we sliced the images into 256 cross-sections from the top down (Fig. 25b), performing FFT on each section (Fig. 25c), and stacked the 256 FFT results to extract the most dominant frequency of ridge oscillation. The result of our FFT calculations is shown in Figure 26. The first few high amplitude peaks were caused by signal leakage in the FFT and should be ignored. The most dominant frequencies that reflect the wavelengths of the surface modulations ranged from 1.59 to 2.29 $\mu$m$^{-1}$. Therefore, the real-space wavelength of the oscillation was on the order of 437 nm to 629 nm. These distances fall within the range of visible light wavelengths, explaining the effectiveness of the substructure as a diffraction grating for visible light. These values are impressively close to Raman’s (1950) estimate of 0.34 $\mu$m (340 nm) for the periodicity of the iris quartz striations.
TEM Characterization. Brazil twins in quartz are readily identified through phase-contrast imaging in the transmission electron microscope (McLaren 1965, 1966). Because of constructive and destructive interference effects when electrons are scattered at the Brazil twin boundaries, bright- and dark-field images of the boundaries generate alternating bright and dark lines that diverge as a “V” with increasing foil thickness. Our TEM observation of an amethyst from Brazil (USNM #R1453) revealed these contrast fringes clearly (Fig. 27). When we examined the FIB extracted foil from an iridescent face of iris quartz, we clearly observed parallel striations that ran parallel to the c-axis of the quartz crystal (Fig. 28), but we were unable to discern the telltale modulations in electron intensity that would indicate Brazil twinning.

Instead, we infer that the striations observed in Fig. 28 represent one-dimensional traces of planar defects, and the defects appeared to be associated with nanoscale fluid inclusions (marked by blue arrows in Fig. 28). These planar defects extended from top to bottom in the cross-sectional FIB TEM foil. Significantly, the portion of the TEM foil that represented the quartz surface revealed jagged undulations that must have corresponded to the ridge-and-valley surface structures observed by SEM and AFM. The planar defects clearly controlled the surface morphology. Specifically, the TEM foil was notched at points where the planar defects intersected the surface.

DISCUSSION

The SEM and AFM images revealed that the z faces of iris quartz are marked by parallel grooves that act as a diffraction grating and give rise to the iridescence exhibited by these Indian quartz samples. Our TEM results showed further that a periodic layered
structure underlies the \( z \) faces, but these layers are oriented parallel to the \( c \)-axis and intersect the \( z \) \{011\} face, rather than being oriented parallel to the \( z \) faces, as Raman (1950) hypothesized. Despite our efforts to find electron optical evidence for a change in phase across the defect boundaries, as would be the case for Brazil twins, we observed no phase reversal.

In light of our imaging results, and also of previous investigations showing that the Brazil composition planes generally lie parallel to \{101\} rather than the \( c \)-axis, we conclude that the defects are not Brazil twin boundaries. Our close inspection of the planar defects suggests the presence of nanoscale fluid inclusions or other impurities that are disrupting the lattice periodicity. Since the iridescence does not precisely disappear at the edge of the \( z \) face, but is visible in narrow zones closely surrounding the \( z \) face (Raman 1950), we believe that iridescence is a combination of diffraction by both surface ridges and the regularly oscillating layers. Our study did not constrain the thickness of the diffracting surface structure. Polishing the iridescent face with a diamond polishing compound (8 micron grit) at first intensified the iridescence and then destroyed it, and we observed that broken portions of \( z \) faces were not iridescent. Thus, we estimate the thickness of the stratified medium as less than 500 \( \mu m \) but greater than the 5 \( \mu m \) depth of our FIB TEM foil, consistent with Raman’s estimate of 250 \( \mu m \).

Crystal morphology can indicate growth rate anisotropy, because growth velocity is a key factor in determining the relative size of crystal faces. Specifically, fast-growth directions generate smaller crystal faces than do slow-growth directions (Zoltai and Stout 1984). In quartz, the \( c \)-axis is usually the fastest growth direction, and \{001\} faces are
very rarely developed in natural crystals. The terminal \( z \{011\} \) face is typically less well expressed than is the \( r \{101\} \) face, and often the \( z \) face is not even apparent, indicating that the direction normal to \( z \) is a faster growth direction than that normal to \( r \). Thus, the periodic layering that gives rise to iridescence develops along a growth direction that is intermediate between the \( (001) \) and the \( r \) face normals.

We infer that the regularly spaced planar defects that characterize the \( z \) faces represent a late-stage crystal growth episode, in light of the fact that the iridescent region occurs as a sub-mm coating on the crystals. It seems possible that the periodic layering records a highly episodic injection of silica-rich aqueous fluids into the cavities that hosted the iris quartz clusters. During periods of solution input, quartz deposition ensued, and the crystal faces grew. These episodes were followed by periods of solution quiescence and, therefore, crystal growth stasis. We speculate that the planar defects formed by the incorporation of fluid inclusions (Fig. 29) on crystal faces at the onset of a new growth cycle (Ihinger and Zink 2000). It seems likely that the \( z \) faces formed first, and the \( r \) faces filled in later, so that, the planer defects are concentrated in the \( z \) faces. The grooved surface of the \( \{011\} \) faces may reflect a final episode of surface etching, with preferential dissolution occurring where defect planes are concentrated (Fig. 30). Whether the rhythmic injection of fluids resulted from wet-dry cycles of meteoric fluids or from geyser-like pulses of geothermal fluids might be resolvable by O isotope geochemistry, since \( \delta^{18}O \) is higher in geothermal fluids than in meteoric fluids (Criss and Taylor 1986).
CONCLUSION

Our study reveals that the iridescent appearance of hematite from João Monlevade, Minas Gerais, Brazil is caused by an ordered microstructure that consists of spindle-shaped hematite nanocrystals with minor Al and P. The nanorods are 200-300 nm in length and 50-60 nm in width, and they are arranged in three orientations rotated by 120° with respect to each other and stacked layer by layer to form the bulk crystal. The distances between adjacent parallel spindle-shape particles in the same layer are in the range of 280 – 400 nm, generating a diffraction grating for visible light. The sub-structure is apparent on all freshly fractured surfaces, indicating that it is not merely an exterior surface coating. Rather, we interpret the periodic sub-structure as the result of crystal growth by oriented aggregation of hematite nanorods.

As with iridescent hematite, the iridescence observed in quartz from the Jalgaon region of India is of natural origin and is not the result of a treatment process. Our characterization using SEM, AFM, FIB and TEM clearly revealed periodic ridge-and-valley structures on the iridescent $z$ faces but not on the non-iridescent $r$ faces. As was inferred by Raman (1950), the ridges are associated with a stratified medium under the iridescent surface, but the orientation of the layers is parallel to the $c$-axis rather than to $\{011\}$. Bright- and dark-field imaging with the TEM offered no support for the prevailing hypothesis that the iridescent grating is associated with Brazil twins. Based on the nature of the defect boundaries, we interpret the stratified medium as an assemblage of growth planes. The distance between the defect planes is on the order of 437 nm to 629 nm, consistent with the diffraction of visible light. Thus, the iridescence in iris quartz is a combination of diffraction by surface ridges and by regularly oscillating growth layers.
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http://www.mindat.org/article.php/1481/A+story+about+color+rock


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Appendix A

Tables

**TABLE 1.** Final Rietveld refinement parameters for iridescent hematite

<table>
<thead>
<tr>
<th>Space Group</th>
<th>$\bar{R} \bar{3} c$</th>
<th>Refinement</th>
</tr>
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<tr>
<td>Unit Cell</td>
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<tr>
<td>$a$ (Å)</td>
<td>5.0500(2)</td>
<td>No. of reflections</td>
</tr>
<tr>
<td>$b$ (Å)</td>
<td>5.0500(2)</td>
<td>Diffraction Range (2θ)</td>
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<tr>
<td>$c$ (Å)</td>
<td>13.7903(6)</td>
<td>No. of variables</td>
</tr>
<tr>
<td>$\alpha$ (°)</td>
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<td>$R(F^2)$</td>
</tr>
<tr>
<td>$\beta$ (°)</td>
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<td>$R_{wp}$</td>
</tr>
<tr>
<td>$\gamma$ (°)</td>
<td>120</td>
<td>$\chi^2$</td>
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</table>
**TABLE 2.** Atomic coordinates and occupancy of iridescent hematite after refinement

<table>
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<tr>
<th>Atom</th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>Occupancy</th>
<th>$U_{iso}$</th>
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<td>Fe</td>
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<td>0</td>
<td>0.35530(6)</td>
<td>0.879(7)</td>
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<tr>
<td>O</td>
<td>0.2996(5)</td>
<td>0</td>
<td>0.25</td>
<td>1</td>
<td>0.004</td>
</tr>
<tr>
<td>Al</td>
<td>0</td>
<td>0</td>
<td>0.35530(6)</td>
<td>0.121(7)</td>
<td>0.004</td>
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</table>
**TABLE 3.** Surface composition of the iridescent hematite

<table>
<thead>
<tr>
<th>Element</th>
<th>Line Type</th>
<th>Wt%</th>
<th>Atomic %</th>
</tr>
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<tr>
<td>O</td>
<td>K series</td>
<td>30.78</td>
<td>59.23</td>
</tr>
<tr>
<td>Al</td>
<td>K series</td>
<td>4.01</td>
<td>4.58</td>
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<tr>
<td>P</td>
<td>K series</td>
<td>0.54</td>
<td>0.54</td>
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<tr>
<td>Fe</td>
<td>K series</td>
<td>64.67</td>
<td>35.65</td>
</tr>
<tr>
<td>Total:</td>
<td></td>
<td>100</td>
<td>100</td>
</tr>
</tbody>
</table>
Appendix B

Figures

Figure 1. Light rays that reflect from the upper boundary and the lower boundary of the thin film interfere constructively or destructively and form a new wave. (Top) Constructive interference: light wave 1 and 2 are in phase. (Bottom) Destructive interference: the two waves are out of phase.
Figure 2. The tarnish of bornite is a thin film that is composed of iron hydroxide and copper sulfide producing the “peacock” color. (From Leon Hupperichs, 2007)
Figure 3. (Top) A freshly fractured face of fire obsidian. (Bottom) SEM image of magnetite thin layers in the obsidian matrix. The scale bar is 1 µm. (From Ma and Rossman, 2007)
Figure 4. Simplified configurations of diffraction caused by a reflective diffraction grating.
Figure 5. (Top) Australian black opal. (Bottom) SEM image shows opal consists of well-arranged and uniform silica spheres. The size of the silica spheres is about 400 nm. (From Rossman, 2013).
Figure 6. Labradorite. (From Dorr, 2015)
Figure 7. Iris agate. (From Krupsaw, 2015)
Figure 8. Iridescent hematite sample in this research. Sample is approximately 2.5 by 2.5cm
Figure 9. An outcrop in the Andrade Mine (Brazil) shows iridescent hematite layers (From Currier, 2012)
Figure 10. The location of Andrade Mine (marked by a pink tourmaline crystal).
(From mindat.org and the Hudson Institute of Mineralogy, 2015)
Figure 11. (A) Geologic Map of the Quadrilátero Ferrífero. Major tectonic structures: DBS - Dom Bosco Syncline, MS - Moeda Syncline, GS - Gandarela Syncline, IS - Itabira Synclinorium, JMS - João Monlevade Synclinorium. Andrade ore is denoted as AN. (B) Location of metamorphic and structural domains in the Quadrilátero Ferrífero. (From Rosière et al., 2001)
Figure 12. Stratigraphic column of Quadrilátero Ferrífero. (From Mendes et al., 2014)
Figure 13. Reflected light binocular microscope image of the iridescent hematite used in this study.
Figure 14. SEM image of hematite sample from Andrade Mine showing hypidoblastic to idioblastic granoblastic texture resulting from post-tectonic recrystallization. (From Rosière et al. 2001)
Figure 15. Rietveld refinement of iridescent hematite. The data and fits are represented by: observed data (black cross), calculated (red line), differences (blue line), and background (green lines).
Figure 16. FESEM images of a freshly fractured iridescent hematite surface from lower to higher magnification.
Figure 17. (a) Optical microscope image taken with the AFM before landing the probe on the surface. The dark triangle is a cantilever. The hematite platelets reflect a strong blue color in this particular direction. The particle beneath the cantilever was scanned by AFM probe. (b), (c) Iridescent hematite surfaces in different scales. (d) Three dimensional image of (c) constructed by NanoScope Analysis software.
Figure 18. Iridescent quartz from Jalgaon, India used in this study. The quartz crystals exhibit strongly expressed terminal faces and iridescence is only visible on smaller $z \{011\}$ faces and not on $r \{101\}$ faces. Sample is approximately $5 \times 4 \times 3.5$ cm.
Figure 19. An iridescent face. Note that the iridescence does not precisely disappear at the edge of the z face, but continues in narrow zones closely surrounding the z face. (From Tanaka, 2011)
Figure 20. SEM images of iridescent surface during FIB milling process. (a) Deposition of amorphous carbon strap. (b), (c) Trenches are formed on both sides of the foil during milling.
Figure 21. Iridescent face vs. non-iridescent face. (Top) SEM micrograph contains both iridescent area (left, marked by z) and non-iridescent area (right, marked by r). The boundary between the r and z faces appears as a white light from top left to bottom middle. (Bottom) Higher magnification of etch pit area. The etch pit of z face contains parallel ridges. The surface of the etch pit from the r face is relatively smooth.
Figure 22. SEM image of etch pit and unetched surface of an iridescent face. The “thick” layers on the unetched surface (right portion of image) are actually composed by multiple thin layers with the same thickness of the layers in the etch pit (left part of the image). The striations shown on the etch pit’s wall (marked by arrows) indicate the orientation of these “thick” layers is the same with the thin layers in etch pits.
Figure 23. EDS spectrum of iridescent face. Data were collected at 20 kv (yellow), 10 kv (red) and 5 kv (blue). Higher voltage spectra reflect chemical composition at greater depths.
Figure 24. AFM images show the topography difference between an iridescent face and a non-iridescence face. The height varies from 500 nm to -500 nm on an iridescent face, and the layer structure can be clearly observed. The height of non-iridescent faces has a much smaller range. The bright spots are dirt on sample’s surface, and the black holes are etch pits. Therefore, the surfaces of non-iridescence faces are fairly flat.
Figure 25. Fast Fourier Transform (FFT) analysis of iridescent face AFM result using MATLAB. (a) AFM raw data. We cut 256 cross sections from top to bottom and the blue line is one of them. (b) The topography information of the blue cross section in (a). (c) FFT transform of (b) to calculate the most dominate frequency of the ridge oscillation.
Figure 26. Final FFT result of the iridescent face in Fig. 25(a). The first few high-amplitude peaks are caused by signal leakage in FFT, and the most dominant frequencies that relate to the distances between two adjacent ridges on z faces are in the range of 1.59- 2.29 μm\(^{-1}\). Therefore, the wavelength of the oscillation is on the order of 437 nm to 629 nm.
Brazil Twins

Figure 27. A TEM micrograph shows Brazil twins in amethyst used for comparison (USNM #R1453). Their locations are marked by blue arrows.
Figure 28. This FIB section was extracted from an iridescent face, which contains periodic planar defects with fluid inclusion parallel to c axis (marked by blue arrows in Fig. 27). Those planar defects cross all the way from top to bottom in the FIB section. The surface’s topography is lower where the planar defects are present, leaving a zigzag pattern shown on the iridescent faces. The lighter striations marked by red arrows are FIB artifacts.
Figure 29. Possible nanoscale fluid inclusions in the planar defects.
Figure 30. Simplified iridescent quartz model.