

# Gems & Gemology



SUMMER 1969



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# ON THE NATURE OF MINERAL INCLUSIONS IN GEMSTONES

by

Dr. E. Gubelin, CG, FGA

Among the many kinds of inclusions encountered in natural gemstones, where they are regarded as unequivocal criteria of genuineness and often as welcome identification marks of locality, mineral inclusions are of particular interest. Just as, externally, the mineralogical environment of a gemstone in a specific habitat will furnish valuable evidence of that deposit's evolution, the inner paragenesis (i.e., the various mineral inclusions) will yield valuable information about the chemical and physical conditions that must have existed during the formation of their precious host crystal.

In a great number of cases, such crystalline formation has been rare enough to be considered unique. Witness benitoite: the chemical constituents, apart from only once being present simultaneously, gathered together at the same time in the proper relation to the relevant crystal lattice. During the growth of a gem, older minerals, having crystallized during an earlier phase and having temporarily ceased

to grow (because of a shortage of substance or a phase displacement), were overgrown or fully enclosed by the younger gemstone, which may have grown even more rapidly (*Plate I-A*). W. F. Eppler (9) has named such minerals of an older formation *preëxistent mineral inclusions*.

*Syngenetic mineral inclusions* are those that have crystallized together with the formation of the gem, from a complex solution, or melt, in which a number of chemical components were present in a dissolved state. Separated molecules, in their function of building blocks of one mineral or another, may link up with one another or with other molecules of their own kind, disregarding completely the crystal structure of other minerals forming at the same time. Or they may, with the atoms at fairly equal distances apart (structural affinity), enter into a mutual isomorphic relationship (mixed crystals) or else grow epitaxially (from the Greek *epi*, meaning *upon*, and *taxi*, meaning *arrangement*) upon their growing faces (oriented intergrowth).

In the process, one of the mineral species may cease to grow, due to a lack of substance supply.

Where such a mineral species has developed rapidly, greedily amassing its components in one direction of growth, as it were, slim, stalky crystals have resulted, such as the rutile (*Plate A-2*) and tourmaline (*Plate A-3*) encountered in many quartzes, or the byssolite fibers included in demantoid (*Plate A-4*). Occasionally, these elongated, prismatic or fibrous mineral inclusions have grown independently; more often than not, however, they have formed coincidentally with the host crystal. But the crystallizing process of the gem now including them had not ceased by the time the guest minerals suspended growth; in fact, it frequently continued to exist in solution. Simultaneous or subsequent solidification (i.e., crystallization of any such substance remaining in solution) ultimately led to the inclusion of foreign minerals from a mixed crystal in which they had coexisted in solid solution until the balance of energy was ultimately upset. Precipitation of minority components has usually been caused by changes in temperature (cooling or heating, with or without change of pressure) and has led to crystalline differentiation in that microscopic crystals were formed, being mostly or nearly always coordinated according to the crystallonomic directions of the major component.

Typical examples of such exsolved, epigenetic inclusions are provided by the silk in corundum; i.e., the fine rutile needles with their sagenitic pattern of coordination in accordance with the three main directions of the

base and rhombohedron (*Plate A-5*); and the small slabs of lepidocrocite coordinated parallel with the main axis in iolite (*Plate A-6*); also, the dense patches of goethite scales in sunstone (aventurine feldspar) (*Plate A-7*). These slabs are not goethite, as originally stated, but lepidocrocite ( $\text{FeOOH}$ ) or ( $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$ ), which has the same chemical composition as goethite. The difference is a morphological one, in that the goethite inclusions are fibrous to acicular and lepidocrocite, tabular.

Alternatively, foreign mineral substance from outside may have penetrated into the interior of a mineral by way of fissures and channels, where it is now present predominantly in the form of skeleton crystals. Most prominent among the minerals involved in the formation of such subsequent inclusions are iron hydroxide, manganese hydroxide (psilomelane) and manganese oxide (pyrolusite). In the fissures filled by them, all of these have frequently produced magnificent dendritic forms or a host of other beautiful patterns. Patterns thus formed are particularly prized when occurring in kinds of quartz such as agate and rose quartz (*Plate A-8*); also, in turquoise, rhodonite, etc.

This deliberately brief excursion into the genesis of mineral inclusions in gemstones was intended to point out just how close a relationship exists between mineral inclusions and their host crystals and what interesting conclusions on the formation of gemstones may be drawn from a thorough knowledge of the inner paragenesis.

Mineral inclusions have been the most important witnesses of the

mysterious process of crystallization that were involved in the growth of mineral deposits at great depths below the earth's surface. Bearing in mind their immense analytic value for a reliable reconstruction of the evolutionary processes underlying their formation, and thus to the science of mineral deposits, over the past 100 years scientists have devoted more and more attention to the exact determination of mineral inclusions. As long as thin sections could be obtained, or when in any case the object to be tested could be destroyed, the task was easy enough. But being concerned primarily with studying the cut specimens of gemstones, gemologists have usually had to confine themselves to crystalloptical scrutiny.

Despite an almost inconceivable variety of shapes and forms, and an all but unlimited multiplicity of possible combinations, a rather reliable criterion is furnished by the characteristic crystal form of a mineral. It is because of this, or of the relevant habit of an idiomorphic crystal inclusion, that its true nature has often been identified. It is thus that in the relatively early stages of crystalloptical research, detection and identification were definitely not only of the aforementioned mineral inclusions, but of such diverse members of the amphibole family as actinolite shingles (*Plate A-9*), amianthus felt (*Plate A-10*) and hornblende needles in rock crystal (*Plate B-1*), bamboolike canes of actinolite in Ural emerald (*Plate B-2*), blades of tremolite in Habachtal and Sandawana emeralds (*Plate B-3*), irregular structures of green chlorite (*Plate B-4*), brown stalks of epidote (*Plate A-1*) and tetragonal bipyramids of anatase in rock crystal

(*Plate B-5*), radiating sheaths of goethite in amethyst (*Plate B-6*), small pseudo-hexagonal slabs and flakes of brown biotite in Transvaal and Ural emeralds, brassy-yellow pyrite crystals in both Chivor emerald (*Plate B-7*) and fluorite (*Plate B-8*), well-developed spinel octahedra in Mogok ruby (*Plate D-3*), euhedral octahedra of chromite in williamsite (*Plate B-9*), mica platelets in peridot, hematite in diamond, euclase, corundum, quartz and topaz (*Plate B-10*), and diamond and garnet in diamond.

Because of the slanting position of the angles incurred by the facets of a cut gemstone, even the well-defined forms of a crystal will often appear grossly distorted, and this may lead easily to misjudgment. Furthermore, ideal cases of idiomorphic crystal forms are extremely rare. Very frequently, crystals viewed under the microscope will not give any elucidation on the nature of the mineral inclusion under observation, since the solid body's shape is irregular, contorted or completely undefined. Not infrequently, such inclusions will be constituted by resorbed or abraded granules, ill-defined slabs, irregularly-edged or spiky aggregates, twisted flakes, splintery chips, or other such indefinable fragments of rudimental structures that would no longer permit morphological assessment. They might at best be identified by virtue of their color or the difference between their R.I. and birefringence and those of their host crystal. Nevertheless, such fragmentary mineral inclusions, being characteristic of certain gemstone species or particular localities, are indeed of some diagnostic value. Pending, discovery of adequate methods of

identification, gemologists therefore contented themselves with giving such exact descriptions of them as they possibly could. Although for the purposes of determining genuineness and distinguishing differences in origin gemological practitioners could reconcile themselves to the mere availability of a phenomenological descriptions of inclusions, this failed to satisfy the gemologist with a bias towards mineralogy and, even more so, the mineralogist interested in gemology.

During the past twenty years, ever-growing numbers of scientists have devoted themselves to painstaking research into the field of mineral inclusions in gemstones, and it is to the outstanding merit of W.F. Eppler that he has dedicated himself to this task systematically and consistently. Applying the orthodox method of crystalloptics in some cases, and extricating the minute mineral inclusions for subsequent microscopic and radiographic examinations in others, he has succeeded in identifying beyond any doubt a host of solid, foreign bodies included in gemstones. This has not only added a number of enlightening findings to the field of genetic observation, but it has also augmented considerably the diagnostic techniques that are so important to gemology.

Thus, W.F. Eppler has managed to corroborate the occurrence of garnet in diamond and to prove the existence of olivine in diamond shortly after it had been observed by R.S. Mitchell and A.A. Giardini (36). He was the first to discover apatite as being a member of diamond endogenesis (8). He also reliably identified, in several other gemstones, certain mineral inclusions recurring frequently and thus

diagnostically significant; viz., the filling with granulated quartz, apatite and epidote of tubes parallel with the c-axis in aquamarine (*Plate C-1*); also the presence in aquamarine of individual specimens or batches of hematite, mica (fuchsite, muscovite, phlogopite), garnet, ilmenite skeletons and petalite; and other fascinating cases of quartz intergrown with apatite and epidote (*Plates C-2, C-3, C-4*), (10-15). The presence of shingles and needles of sphene crystals in star spinel, causing the latter's asterism, and having previously been detected by this author using purely crystalloptical methods, has also been confirmed by Eppler (*Plate C-5*), (6).

Since the discovery of the rich Siberian diamond deposits in Yakutia, Soviet mineralogists have exercised admirable exactitude in investigating the inner paragenesis of diamonds mined in the USSR. However, they, too, did not examine the solid bodies while still included; rather, they "freed" the mineral inclusions from their host diamond in order to subject these isolated microscopic crystals to crystalloptical and radiographic testing.

Proceeding along the same lines as Eppler, but independent of him, they, too, managed to recognize olivine and garnet as being the most significant and frequent accessory minerals in diamond. The olivine was identified by them as forsterite with approximately 6 percent fayalite (*Plate C-6*), and the garnet as pyrope with a high content of pyrope (*Plate C-7*). They also identified brown chrome spinel (picrotite) (*Plate C-8*), green chrome diopside, or chrome enstatite, and diamond (*Plate C-9*). An occasional incidence of serpentine, chlorite, biotite and phlo-

gopite was attributed by them to pseudomorphic changes suffered by mineral inclusions previously syngenetic. Reports of chromite, hematite, ilmenite, magnetite, pyrite, quartz, rutile and zircon—all minerals that in earlier works by different authors had been asserted as inclusions in diamonds—were considered doubtful or else refuted by the Soviet mineralogists on the grounds that the purely visual identification method used by earlier authors had given rise to confusion and erroneous identification or that, as in the case of quartz and zircon, it had by no means allowed for the genetic criteria pointing to their preëxisting or syngenetic formation.

The Soviet publications (14-22, 32, 39) furnish comprehensive information on the findings of research into mineral inclusions with reference to conditions of formation, orientation within the host diamond, R.I. and/or D.R., angles of optical axes, ratios of parameter, lattice constants and S.G. In places, these results are accompanied by illustrative photomicrographs and Laue diagrams. All these findings concerning mineral inclusions in diamond tell us that such alkaline minerals as olivine, garnet, chrome spinel and chrome diopside (or chrome enstatite) were formed simultaneously with the diamond, during the metamorphosis of olivine aggregate into what have been called griquaité nodules (as far as the main mineral constituents are concerned, griquaité is similar to eclogite; but although the first is of deep magmatic origin, the latter is of metamorphous origin) in intracrustal areas of formation (37). And graphite and serpentine are to be looked upon as pseudomorphoses after

diamond and olivine, the same as chlorite, biotite and phlogopite, being mutations of garnet, are encountered only as secondary fissure fillings, having penetrated into cracks and slits at a later stage.

Stimulated by these interesting findings of the Soviet mineralogists, W.E. Sharp of the Adamant Research Laboratory, Johannesburg, using the powder method, performed X-ray analyses on a great number of industrial diamonds. The diagrams thus obtained enabled him to identify as inclusions of ore minerals various iron oxides, such as goethite, hematite, magnetite and possibly even wustite. He, too, holds that the serpentine and chlorite observed are epigenetic alterations of syngenetic olivine or garnet crystals, respectively. In one isolated case, he even succeeded in obtaining the line diagram of graphite from material he had scraped off a fissure plane, of which he informed the author personally. He has thus, we may take it, corroborated the Soviet observation holding that graphite may be caused by graphitization of diamond in places where a carbon structure has been grossly disturbed (i.e., tension cracks or cleavage fissures), and also in the region of stress between foreign bodies included in the diamond substance enclosing them. Evaluating the findings of his elaborate X-ray tests, Mr. Sharp arrived at the definite conclusion that pyrrhotite (*Plate C-10*) is an ore inclusion occurring rather frequently in South African diamonds, where it is often found intergrown with other ore phases. Most frequently, it was found integrated with pentlandite; occasionally with pentlandite and pyrite. Such intergrowth probably



resulted from the exsolution of an earlier high-temperature phase that was later embraced by a growing diamond. Individual formations of ilmenite, pyrite or rutile were not found by Sharp, either.

An exceptionally interesting fact of identification was achieved in an entirely unorthodox manner by B.W. Anderson. During the examination of dense batches of yellowish-brown mineral inclusions in a Colombian emerald, his spectroscope revealed a spectrum of rare earths displaying an abundance of lines and evidencing parisite (47). Parisite is a rare fluocarbonate of the cerium metals  $(\text{Ce,La,Di})_2\text{Ca}[\text{F}_2/(\text{CO}_3)_3]\cdot\text{C}_3\text{F}_4$  that forms pyramidal to slender prismatic crystals varying in tint between brownish yellow or yellowish brown and lilac (*Plate D-1*). One of its scarce occurrences being Muzo, Colombia, its presence in emerald may well be considered a welcome locality feature.

Realizing the novel technique of combining microscopy with the X-ray powder method, P.C. Zwaan achieved definite analysis of a number of internally paragenetic minerals in a variety of gemstones. As early as 1964, several conspicuously hexagonal, prismatic inclusions in a lilac-colored Ceylon spinel were identified by him as apatite (*Plate D-2*), (48) and he has now announced the identification of apatite, corundum, phlogopite, pyrrhotite and spinel (the latter confirming later observations by this author) in corundum (*Plate D-3*), as well as of apatite, muscovite and rutile (the latter in concurrence with O. Mellis) in almandite (49).

This brief review of the new research techniques developed since the

middle of this century and of the highly noteworthy findings made possible by them with regard to mineral inclusions in gemstones has, it must be admitted, thrown light on merely a few of the milestones marking the long, often trying, but always fascinating path through this field of investigation. Yet it does show how in recent years the most remarkable findings have been made, yielding information that may contribute greatly to elucidating the genesis of gemstones.

Nevertheless, gemology, with its totally axiomatic approach, could not rest content with the present state of affairs, since it cannot, in the last analysis, tolerate the destruction of its objects and the great values they so often embody. Indeed, gemologists must continue in their search after the techniques and tools that may help them solve their problems without damage to, or even destruction of, their costly materials. In other words, if they wish to abstain from the often destructive methods of mineralogy, they must be concerned with devising their own instruments that will serve their needs more adequately.

The postulates of gemology have now been met with to a great extent by the recent invention of the electron-microprobe analysis, which has brought a revolutionary innovation to the field of analytic techniques. Providing the analyst with completely new ways and means of research, this highly impressive process is of the utmost importance to him by offering, as it does, an exact and nondestructive method of probing into the chemical constituents, and their quantitative ratios, of the substances being tested. It allows a qualitative and quantitative

analysis of solid bodies with volumes far beyond the faculties of the unaided human eye, thus obviously including mineral inclusions in gemstones.

This very promising instrument is called an *electron microprobe*, and is an elaborate device of considerable dimensions. It is thus highly unlikely that it will ever be included in the equipment of a gemologist; rather its installation will be restricted to the big research laboratories. More recently, this device has been employed for mineralogical-research tasks in general, and for investigating gemstones and their mineral inclusions in particular, and it is to be duly expected that this will be increasingly so in the future. For this reason, a report on the significant results achieved with the new system had better be preceded by a brief description of the device and its mode of operation.

The first operating electron-probe microanalyzer was constructed by R. Castaing shortly before 1949. Designed as a combination of parts of the electron microscope and an X-ray fluorescence unit, it functions as an instrument for the quick and nondestructive qualitative and quantitative analysis of solid bodies with a volume of only a few cubic microns (1 micron equals 0.001 mm.). Samples to be tested need not be extracted from the substance enclosing them, but they will, of course, have to be exposed to the surface of it. It is on account of this improved facility of procedure that microprobe analysis proves to be such an advantageous method of investigating mineral inclusions in gemstones. It yields the solutions to many analytical problems that were previously difficult, if not impossible, to

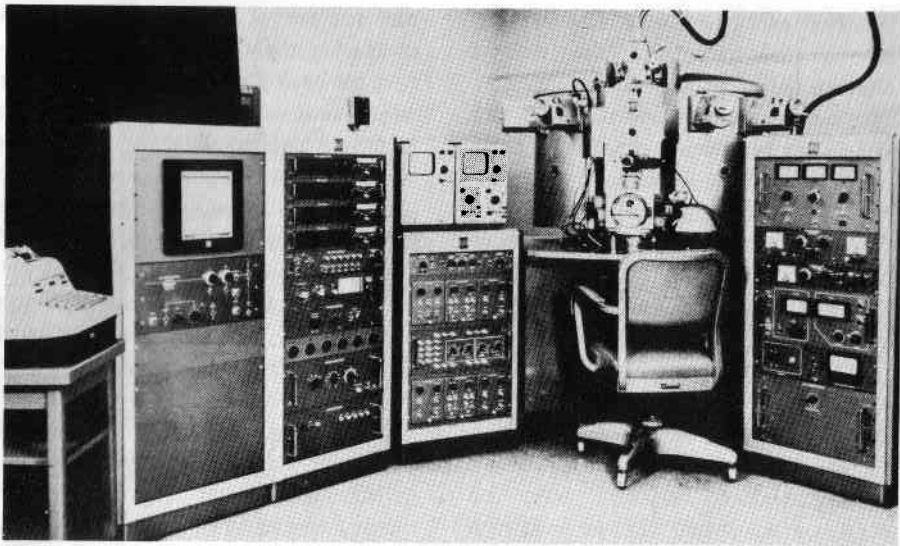
solve by conventional, let alone by optical, methods.

The electron microprobe emits a finely focused beam of electrons, directing it onto the surface of the sample and producing an X-ray reflection characteristic of the chemical elements affected by the electron excitation. The angle of reflection and the intensity and wavelength of the characteristic X-rays—all of these factors being determined by the structural elements of the material tested—will then be used to furnish the analytical data to determine the elements that are present in the target and their relative mass concentrations.

Currently, all such elements can be identified that have atomic numbers upward of 11. In typical cases, the spatial resolving power is between 2 and 3 microns; the sensitivity ranges from  $1:10^4$  to  $1:10^3$ , depending on the composition and expansion of the sample; and the relative accuracy amounts to between 1 and 2 percent, if the concentration is in excess of a few percent. The highly complex equipment consists of (a) an electron-beam column, (b) a number of X-ray spectrometers, (c) a light microscope, (d) a high-voltage power supply and control circuit, (e) one or more vacuum systems, and (f) a measuring-and-recording system for the analytical information.

*Figure 1* shows the equipment contained in a typical probe laboratory. The most important part of the whole apparatus is the electron-beam column housing the electron-gun assembly, apertures, magnetic lenses and deflection plates.

*Figure 2* depicts a typical example of such an electron-beam column. The

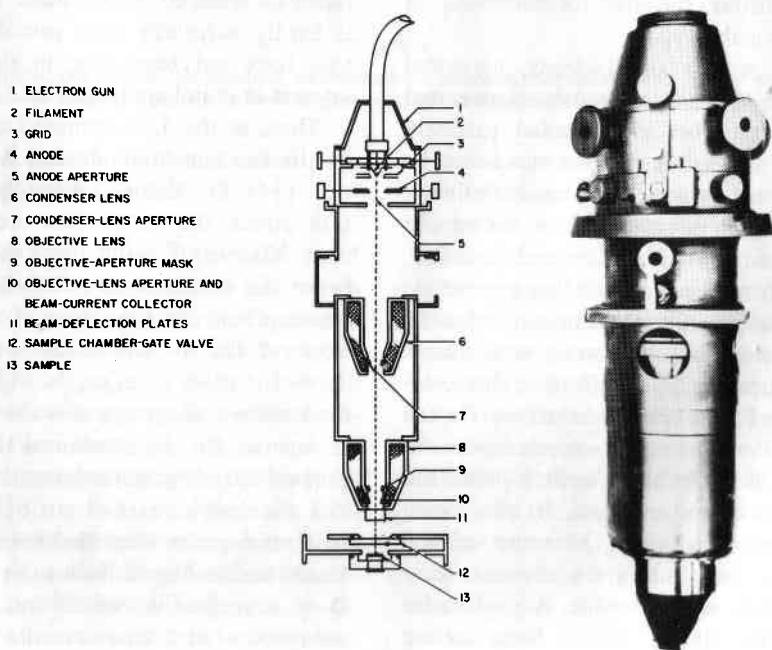


*Figure 1*

A complete electron-microprobe laboratory.

*Figure 2*

Electron-beam column with a schematic cross-section.



- 1 ELECTRON GUN
- 2 FILAMENT
- 3 GRID
- 4 ANODE
- 5 ANODE APERTURE
- 6 CONDENSER LENS
- 7 CONDENSER-LENS APERTURE
- 8 OBJECTIVE LENS
- 9 OBJECTIVE-APERTURE MASK
- 10 OBJECTIVE-LENS APERTURE AND BEAM-CURRENT COLLECTOR
- 11 BEAM-DEFLECTION PLATES
- 12 SAMPLE CHAMBER-GATE VALVE
- 13 SAMPLE

function of the column is to provide an electron beam of the desired characteristics at the sample surface. The beam must be adjustable in size and density; however, these parameters must be very stable, once established for any program. The heated filament provides a source of electrons that are accelerated by the field between the anode and source. The acceleration potential may be varied at any time from 2 to 50 kv. and can be selected by the operator. The first magnetic lens, or condenser, lens, focuses and diminishes the beam, usually at a point well above the objective aperture. The second magnetic lens, or objective lens, focuses the image of the first lens onto the surface of the sample and provides a further reduction of the image. The beam-current monitor, shown at the position of the objective aperture, is invaluable in duplicating analytical conditions for the measurement of beam stability.

A stream of electrons, narrowed down to a hair's breadth, is generated by the tube and directed precisely onto the surface of the sample to be analyzed, which, in the case of mineral inclusions in gemstones, is the minute cut surface of the mineral included, which has been exposed on the surface of the gemstone by the cutting of its facets. The electron bombardment produces an X-ray-reflection characteristic of the atoms encountered. On the one hand, an angle is formed between the electron beam emitted and the X-ray beam reflected, its size being determined by the structure of the atoms constituting the chemical composition of the sample. A goniometer may be adjusted to the X-ray leaving

the surface at various angles, thus measuring the typical angle of reflection for each element. In this way, the constituent elements of the sample may be determined. On the other hand, crystals of a suitable material are used to deflect the X-rays according to their wavelengths, and the deflected X-rays are picked up by X-ray detectors. The photons of these X-rays entering the detector are converted into oscillations of electrical energy. The frequency of these impulses is in direct proportion to the intensity of the X-ray radiation received, and this, in turn, is in direct proportion to the quantitative ratio of the elements producing it within the substance being analyzed (*Figure 3*).

During the relatively short period that has elapsed since the introduction of this revolutionary innovation in the field of chemical microanalysis, it has already established itself in a wide range of research applications. And it is hardly surprising that gemologists, too, have not been long in showing interest in it and applying it.

Thus, at the 43rd Annual Congress of the German Mineralogists Association (34), O. Mellis, delivering a lecture about the spectrolite occurring near Ylammaa, Finland, was able to prove the existence of a definite relationship between the chemical composition of the spectrolite and its labradorescent color changes, as well as to the number of opaque, needle-shaped inclusions. He also explained that the process of radiographic determinations and the analysis carried out by means of a microprobe identified these same black needle-shaped inclusions (*Plate D-4*) as magnetite, which was, itself, intergrown with fine ilmenite blades

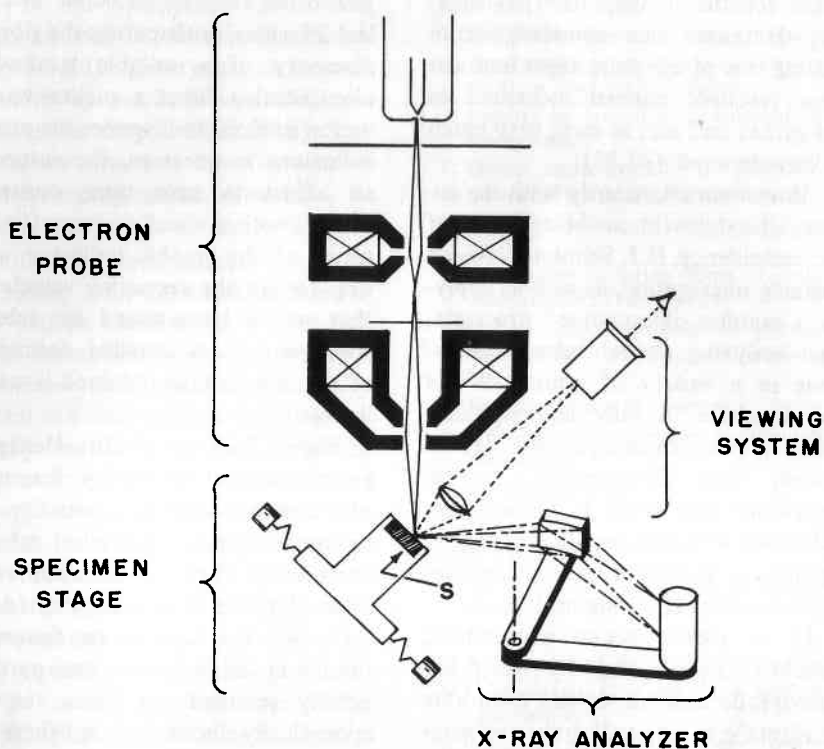


Figure 3

Schematic drawing of an electron microanalyzer showing the relationship of the electron probe, the specimen stage, the viewing system, and the X-ray analyzer to the specimen, S.

(probably secondarily exsolved from the magnetite—author).

Notwithstanding the efforts of eminent mineralogists, it had previously been impossible to determine the true nature of the needles intersecting each other (*Plate D-5*) in red garnets of the pyralspite family. However, the combined application of optical and radiographic methods by O. Mellis, in conjunction with electron-beam analysis, has since achieved a definite identification of these needle-shaped inclusions as rutile. He is convinced that its formation

is not to be attributed to exsolution but to syngenetic epitaxy and thus represents a phenomenon of growth. These rutile needles are predominantly oriented parallel with the edges of the rhombododecahedron. It is only in Madagascar garnet that they have been observed to be coordinated also to the cube. If parallel to the edges of the rhombododecahedron, the needles are invariably twinned; if aligned, however, with the edges of the cube, they are not pure  $\text{TiO}_2$ , but are invariably mixed crystals, which is easily recognized by the difference in interference colors when viewed in polarized light.

These reliable findings have considerably increased our knowledge concerning one of the most important and most frequent mineral inclusions in red garnet and are, as such, very much to be welcomed. (33,35).

Almost simultaneously with the author, although without being aware of the coincidence, H.J. Schubnel, using a Castaing microprobe, as well as applying a number of combined processes, was analyzing several mineral inclusions in a variety of gemstones. He found calcite in ruby and sapphire, spinel in ruby, chloroapatite in Ceylon spinel, and chloroapatite, pyrite, pyrrhotite and rutile in Ceylon sapphire (40, 41), thus producing findings happily in accordance with those described in Part II of this study.

In a more recent publication, Schubnel reports that by using the microprobe analysis he has been able to identify as primarily unmixed magnetite the black, needle-shaped inclusions responsible for the asterism in Indian star diopside. These are, themselves, intergrown with secondarily exsolved blades of ilmenite and hercynite (42).

After other analytic methods had failed to detect the coloring of chrome-jadeite during the examination of jade-albite (27,28,29,30) by the author, successful analysis was finally achieved by means of the electron probe. Following the kind suggestion of M. Weibel, Professor of Geochemistry at the Institute of Crystallography & Petrology of the Federal College of Advanced Technology in Zurich, the author subsequently obtained permission to use the Institute's AMX electron microprobe to identify the mineral inclusions in a variety of

gemstones he had collected over the last 25 years, anticipating the possible discovery of a suitable method of identification. Since a qualitative evaluation sufficed to diagnose the mineral inclusions in question, the author, in an effort to save time, contented himself with a visual assessment of the travel of the graphic indicator, omitting to use the recording attachment that would have traced the relevant spectrograms. A detailed description of the results thus obtained is set out below.

*Plate D-6 to D-10:* Hexagonal prisms of slim to stubby shapes are often encountered in almandite, kornepine, spinel and Ceylon ruby. In most cases, they are euhedral with a base of medium size. Dipyramids are rare, but the crystals are frequently double ended, yet sometimes partly or grossly resorbed. At times, they are sporadically distributed; at others they occur in groups. The microlites are either colorless (i.e., mimicking the color of the host crystal) or they are olive green (notably in kornepine). When viewed between crossed Polaroids, they are conspicuous, owing to straight extinction and vivid interference colors. Their R.I. is distinctly lower than that of their host gems. Examination of the crystal inclusions exposed on the facet surface, by means of the electron microprobe, produced readings definitely indicating the presence of Ca and P.

Consequently, these minute inclusions have been identified as apatite  $[Ca_5(F,Cl)(PO_4)_3]$ . The presence of F cannot be revealed by the microprobe, because its atomic number 9 is below 12, the minimum number detectable by this instrument. Apatite crystal-

lizes in the hexagonal system and occurs in long to short columnar or thick tabloid single crystals, or in small crystal groups (as inclusions, they prefer columnar dipyrarnidal habits). Apatite shows a marked bias for variable chemical composition; therefore, its physical properties fluctuate between the following mean indices:  $n = 1.642$  and  $1.646$ ,  $S.G.=3.18$  and  $hardness=5$ .

Owing its existence to a great variety of formation processes, apatite is widespread as a secondary accessory mineral in a multitude of deposits. It is scarcely ever absent from magmatic rocks and is often to be encountered in pegmatite and pneumatolytic formations, but is also occasionally found in hydrothermal sources (veins and alpine crevasses). In the pegmatite phase, apatite is a primary mineral of early crystallization, although it may also be formed at a later stage of this genetic phase. Accordingly, apatite may occur in gemstones either as a preëxistent or as a syngenetic mineral inclusion.

### Descriptions of Color Photographs

#### Color Plate A:

1. Apatite needles embraced by quartz (rock crystal).
2. Rutile needles in rock crystal.
3. Rods of tourmaline in rock crystal.
4. Radiating byssolite fibers in demantoid.
5. Exsolved rutile needles in ruby.
6. Exsolved leipdicrocite tablets in iolite.
7. Dense concentration of goethite flakes in aventurine feldspar (sunstone).
8. Secondary, epigenetic formation of psilomelane in dentritic agate.
9. Blades of actinolite in rock crystal.
10. Dense felt of amianthus fibers in quartz, causing chatoyance.

#### Color Plate B:

1. Rods of hornblende in rock crystal.
2. Bamboolike canes of actinolite in emerald from the Ural Mountains.
3. Needles of tremolite in emerald from Sandawana.
4. Dense concentration of chlorite in quartz (often producing moss agate).
5. Clearly euhedral anatase crystals in rock crystal.
6. Divergently radiating fibers of goethite (caxenite) in amethyst.
7. Well-developed crystals of pyrite in emerald from the Chivor Mine, Colombia.
8. Euhedral pyrite in fluor spar.
9. Well-defined octahedron of chromite in williamsite.
10. Hematite in topaz.

#### Color Plate C:

1. Growth tubes filled with quartz, apatite and epidote in aquamarine.
2. Phologopite in aquamarine.
3. A skeleton of ilmenite in aquamarine.
4. Combination of quartz and apatite in aquamarine.
5. Oriented arrangement of twinned spines in star spinel.
6. Pseudotetragonal olivine prisms epitaxially settled on the octahedron face of a diamond.
7. Garnet crystal in diamond.
8. Combination of distorted octahedra of chrome-spinel in diamond.
9. Emerald-green diopside crystal in diamond.
10. Group of pyrrhotite crystals in diamond.

#### Color Plate D:

1. Slender prisms of parisite in emerald from the Muzo Mine, Colombia.
2. Idiomorphous prisms of apatite in mauve spinel from Ceylon.
3. Euhedral octahedron of spinel in Burma ruby.
4. Tiny needles of black magnetite in spectrolite from Finland.
5. Rutile needles oriented along the edges of the rhombododecahedron in almandite.

6. Group of idiomorphous prisms of apatite in almandite from Ceylon (these apatites proved to contain lanthane and thorium).
7. Double-ended apatite crystals in korne-rupine.
8. Stumpy prisms of apatite in ruby (from Ceylon?).
9. Slender stalks of apatite in ruby (from Ceylon?).
10. Partly xenomorphous, partly resorbed apatite crystals in mauve spinel from Ceylon.

#### Color Plate E:

1. Tabular biotite parcels in almandite.
2. Brown biotite platelets in korne-rupine from Ceylon.
3. Pseudo-hexagonal and spotted biotite platelets in peridot.
4. Dense concentration of biotite in pink sapphire from Ceylon.
5. Dark-green flakes of biotite in emerald from the Transvaal.
6. Irregular concentration of biotite flakes from the Habachtal.
7. Red-brown phlogopite in sapphire (Ceylon?).
8. Tabular phlogopite parcel with broken borders in spinel.
9. Microlites of chromite in the center of a residual fluid drop in peridot.
10. Irregular aggregates of chromite in emerald.

#### Color Plate F:

1. Green fragment of chrome-diopside in diamond.
2. Feldspar crystals in sapphire.
3. Hair-fine, fox-red fibers of goethite and individual quartz crystals (bright patches) in topaz.
4. Dense cluster of quartz grains surrounded by hairlike fibers of goethite in topaz.
5. Tiny octahedra of hercynite arranged in parallel lines in spinel.
6. Concentration of hercynite octahedra arranged in parallel array in blue spinel.
7. Dense concentration of ilmenite in almandite.
8. Idiomorphous calcite crystal displaying a rhombic pattern of lines caused by intertwined twin lamellae in Burma ruby.
9. Euhedral crystals, partly well preserved and partly as cleavage fragments,

enclosed in emerald from the Muzo Mine, Colombia.

10. Xenomorphous, pseudo-octagonal calcite in red spinel from Burma.

#### Color Plate G:

1. Xenomorphous intergrowths of dolomite and calcite, partly filling octahedral cavities in blue spinel.
2. Enlarged area of Photograph G-1 exhibits the difference between dolomite and calcite in one of the filled cavities.
3. Two tabular fragments of molybdenite in emerald.
4. Well-developed microlites of niobite in sapphire.
5. In the center of the photograph there is a slightly damaged pyrite accompanied by flakes of biotite (brown) and fine silk of rutile needles in sapphire from Ceylon.
6. Pyrrhotite of ideal development in ruby. Diagonal lamellae of some exsolved minority component are distinctly seen.
7. Resorbed pyrrhotite grain in sapphire.
8. Grains of pyrrhotite and pentlandite of varying sizes and irregularly dispersed through an emerald.
9. A crystal of olivine flush with the table facet of a brilliant-cut diamond.
10. A fragment of chrome-diopside exposed on the surface of the table facet of a brilliant-cut diamond.

#### Color Plate H:

1. Irregular conglomerations of a black and red mineral, whose chemical composition of  $TiO_2$  indicates either anatase or rutile, in kyanite.
2. Huge prisms of red rutile in sapphire from Ceylon.
3. Black rutile resorbed into a ball-like shape and surrounded by a stress halo in sapphire from Montana.
4. Well-developed crystal of sphene in ruby from Burma.
5. Group of pale-yellow sphene crystals in ruby from Burma.
6. Brilliant-red, euhedral crystals of uranpy-rochlore (hatchettite) in sapphire (the bright spots are feldspars).
7. Rounded grains of metamict zircon surrounded by tension cracks in korne-rupine from Ceylon.

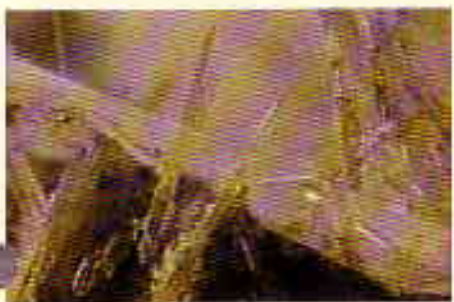


8. Bright zircon surrounded by tension cracks that contained trace impurities of anhydrite and hematite in sapphire.
9. Idiomorphous, yet slightly resorbed, high zircon in mauve sapphire from Ceylon.
10. Crystal of high zircon, somewhat irregularly developed, in kornerupine from Ceylon.
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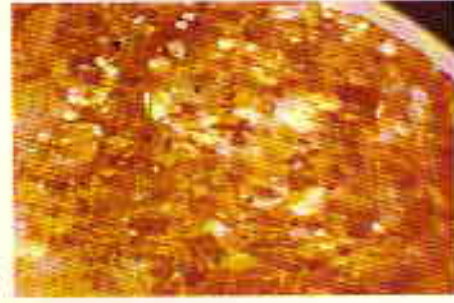
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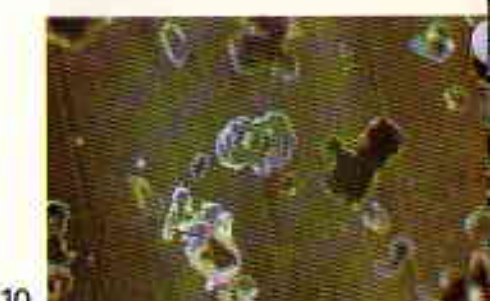


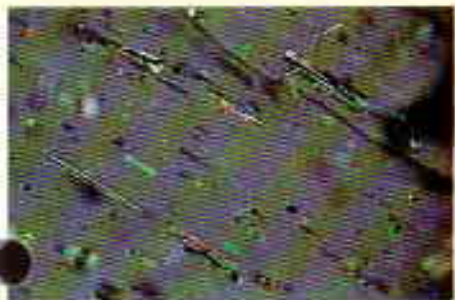
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**B**



**D**

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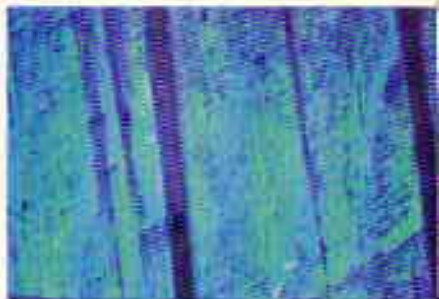
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7



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9



10



# Developments and Highlights at the

## Gem Trade Lab in New York

*by*

Robert Crowningshield

### **X-Ray Bombarded Sapphires**

It has long been known that yellow sapphires can be temporarily darkened in color by irradiation with subatomic particles or even X-rays. Unlike diamond, the color change is not permanent, and a few hours exposure to sunlight will cause the color to fade. It is also known that natural dark-yellow sapphires are more sensitive to heat than other colors of natural sapphires and also ruby.

As an experiment, we X-ray treated a natural-yellow sapphire that had been overheated accidentally during manufacture. We were able to match its X-ray-treated color with other stones from the original lot. At the same time, we X-ray treated four light-yellow stones belonging to the Laboratory. We taped all the stones and one other from the original lot to a south-facing window over the long

July 4th weekend. When they were removed from the window, the Lab's stones had faded to their original color, whereas the other stones remained unchanged. It is hoped that the X-ray-bombarded stone will retain its color. We have heard of one natural-yellow sapphire that faded due to heat but that regained its color after having been left outside the window of a manufacturer's shop for an entire winter.

### **Brown Conch Pearl**

We had the pleasure of examining a 43-carat pearl from the brown conch, or conchiglia, shell. The flamelike texture of the surface closely resembled that of the more common pink conch pearl. The brown pearl we examined was reported to have been discovered by a housewife preparing one of the animals for a stew.

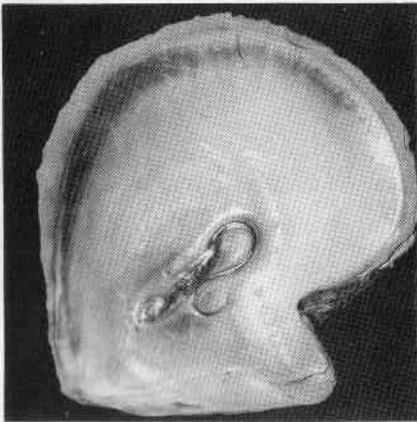
### Entombed Fish

Through the good offices of Gem Trade Lab member Maurice Shire, we are pleased to reproduce another "conchological" oddity: an entombed fish in a pearl-oyster shell owned by Max Kniel of Zurich, Switzerland (*Figure 1*).

### Emerald-Green Grossularite

In the last issue we acknowledged a gift of rough, near-emerald-green grossularite. Now, we can report that similar material makes truly exciting and beautiful gems. Jerry Call, our staff lapidary, cut a fine emerald-cut stone weighing 1.12 carats. We have since seen two others and have heard of at least 12 more, the largest in excess of three carats. In color, they resemble the best demantoids but without their high dispersion. We have been unable to detect chromium in the absorption spectrum of the stones we have examined, although they do appear red under a color filter.

*Figure 1*



### Dyed Howlite

*Figure 2* is a photograph of an intense-blue carving of dyed howlite. The color was presumably to imitate turquoise, but it was much too intense. Since the material had the same R.I. as turquoise and is frequently too large to obtain a reliable S.G., two other tests are helpful. First, a drop of acid in a hidden area will etch the surface, exposing the ends of crystals. Secondly, a hot needle will melt the surface much as borax used in jewelry repair does.

### Garnet Inclusions

*Figure 3* shows a patch of needle-like inclusions oriented in three directions and resembling a trout fly. It is in a truly beautiful red-orange garnet with the properties of spessartite-pyrope-almandite.

### Glass Jade Imitation

The center stone in *Figure 4* is the first mounted "meta-jade," or "Iimori

*Figure 2*





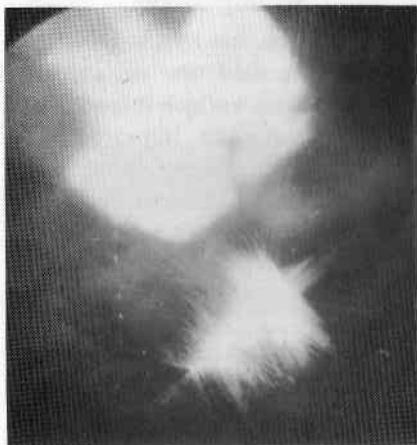


Figure 3

-jade," we have encountered. It is actually a glass manufactured in Japan and erroneously called "synthetic jade." The 18K ring contained numerous diamonds, six pear-shape green synthetic-spinel triplets and 42 synthetic sapphires.

#### Facet-Junction Wear

Figure 5 shows one of many stones in a ring, all of which showed facet-junction wear on both crown and pavilion and were suspected of not being diamonds.

#### Black-Star Sapphire

Through the courtesy of Dr. A. E. Alexander, we show two views of a remarkable 277-carat black-star sapphire. On the back, a very symmetrical concavity was polished in which the star was exceptionally brilliant (Figures 6 and 7).

#### Pink Smithsonite

Pink smithsonite, which we had never encountered before, was made available to us for study by Astro Minerals, New York City. The piece



Figure 4

we examined was pink shading into gray, resembling a pink-crested cockatoo. Figure 8 illustrates the absorption spectrum of the pink areas.

#### Unusual Idocrase

Figure 9 is the absorption spectrum of a new, to us, form of idocrase. The rough specimens we studied were transparent yellow-green. The R.I. of 1.728-1.732, with very low birefringence and an S.G. of 3.36, are normal for idocrase. Analysis of interference figures of several of the specimens indicated uniaxial, but with the quartz wedge some appeared negative and some positive, although the figures were hard to obtain and our observation may have been inaccurate. The stones were highly dichroic, and two small specimens had areas of an almost emerald green. In these areas, the absorption spectrum showed indications of chromium. Several of the stones contained numerous transparent, seemingly colorless low-relief crystals that looked like partly melted ice cubes (Figure 10).

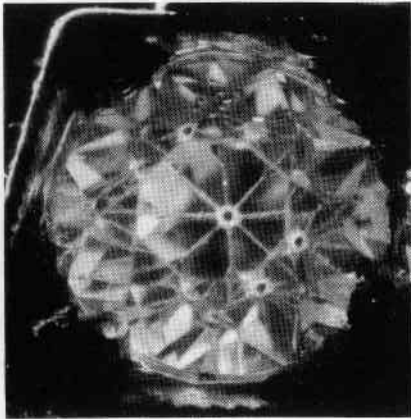


Figure 5

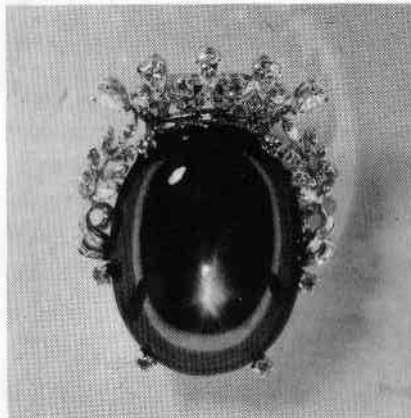


Figure 6



Figure 7

### Hydrogrossular

X-ray diffraction by the Los Angeles Lab identified for us a highly polished cushion-antique-shaped tablet resembling malachite. It proved to be chrome-bearing, massive hydrogrossular. We are happy to acknowledge it as a gift from Max Schuster, New York City gem dealer.

### Heat-Treated Zoisite

We have been examining carefully all zoisites before and after heat treatment, and feel that we may have a partial answer to the question of why the stones mentioned in the last issue of *Gems and Gemology* fractured following the use of ultrasonic. Stones with needlelike inclusions or small fractures and cleavages were either unaffected by the heat treatment or showed only a slight extension of the fractures. However, small fingerprint inclusions, such as the ones illustrated under 45x in *Figure 11* developed fractures around the small elements of the fingerprint, resulting in a continuous fracture plane. It is possible that ultrasonic aggravated one of these heat-induced fractures, resulting in the damage reported by Graduate John Fuhrbach.

*Figure 12* illustrates the same fingerprint inclusion after regular heat treatment for two hours at 700°F. A heat-induced fracture extends considerably beyond the original limits of the inclusions. This loose stone was later subjected to ultrasonic for a considerable length of time with no further spreading of the flaw. Most heat treatment of zoisite, as well as other commonly heated stones, is done after the stones are cut, rather

PINK SMITHSONITE (Astro)

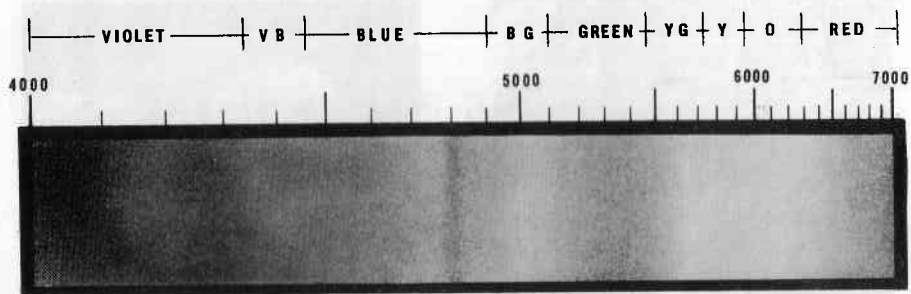


Figure 8

YELLOW-GREEN IDOCRASE (?) (NY 43741)

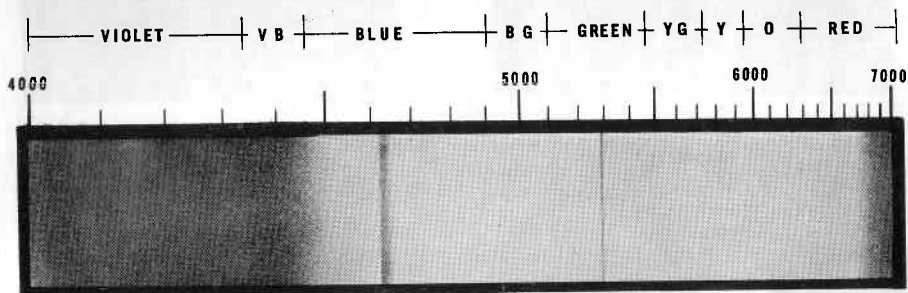


Figure 9

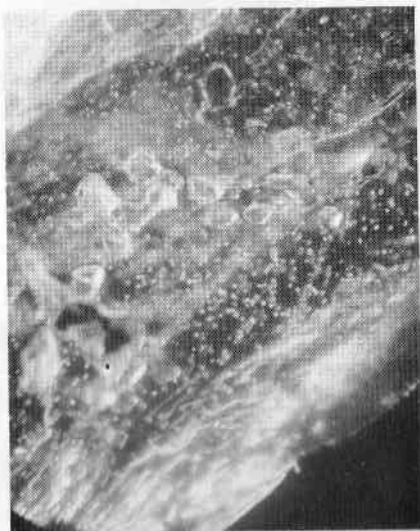


Figure 10



Figure 11



Figure 12

than in the rough, since insignificant flaws can spread in the rough and spoil considerably more than their size would suggest before treatment.

#### "Faceted" Emerald Crystal

Figure 13 illustrates the termination of an unusually flawless natural-emerald crystal submitted by gem dealer George Bruce, Stone Mountain, Georgia. So clear the stone and so well "polished" the faces, it was at first assumed that the stone had been artificially faceted.

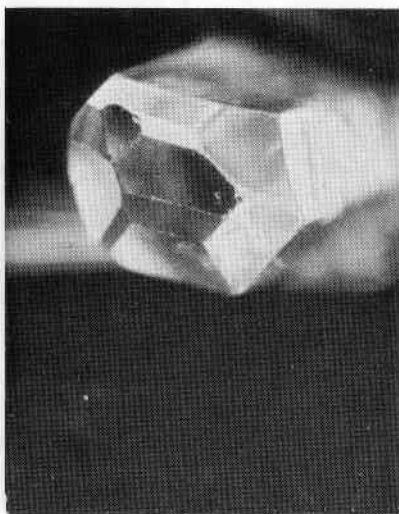


Figure 13

#### Acknowledgements

We wish to express our sincere thanks for the following gifts:

To **Mr. Jean Naftule** of Nafco, New York City, for a selection of emerald-green tourmalines for use in study and student collections.

To Graduate **Howard Rubin** for a selection of many types of stones.

To the **M. & L. Singer Co.**, New York City, for a selection of boules and cut synthetic sapphires.

To student **Murray Darvick**, New York City, for a fine specimen of opalized wood.

# Developments and Highlights at the Gem Trade Lab in Los Angeles

by

Richard T. Liddicoat

## Russian Emeralds

Some time ago we received a large number of Russian emeralds as a gift from Arnold Baron, CG, Billings, Montana. Recently, we studied them under magnification, resolving some very interesting inclusions. One faceted stone contained hundreds of two-phase inclusions; in almost every one the bubble moved as the stone was turned. As many as a dozen bubbles could be seen moving at once. In none of these did we encounter a third phase. *Figure 1* shows a number of tiny two-phase inclusions in one of the stones. At the center of *Figure 2* is a two-phase inclusion that we thought at first was three-phase; this was taken at 105x. *Figure 3* also shows some two-phase inclusions, the one at the center of the field in the roughly triangular cavity is the most prominent.

## Fluorite in Emerald

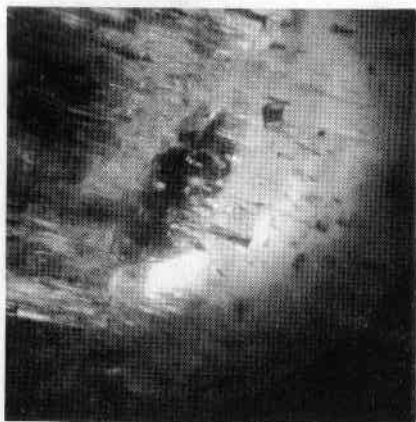
*Figure 4* shows a crystal inclusion with distinct color banding evident. The included crystal reached the surface, so Charles Fryer, GIA Lab Supervisor, scraped it and by X-ray diffraction proved it to be fluorite. We do not recall having encountered fluorite inclusions in emerald before.

## Diamond Wheel Marks

A diamond with rather heavy wheel marks in two different directions on the same facet surprised us, so we photographed it (*Figure 5*). We do not expect to see running lines in more than one direction.

## Nephrite Absorption Spectrum

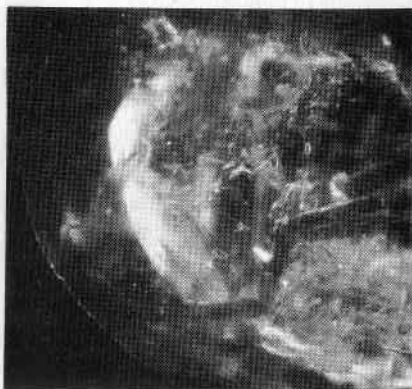
Recently, we have seen two or three yellow-green nephrites of unknown origin that showed a distinctive absorption spectrum (*Figure 6*).



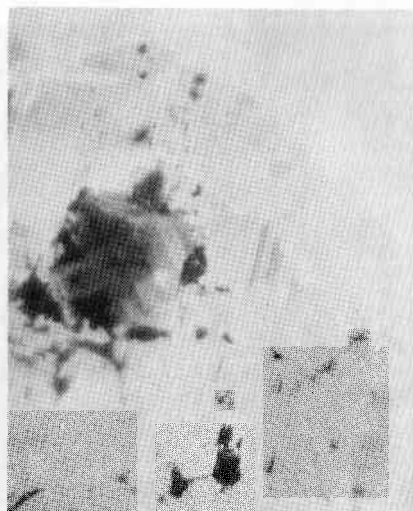
*Figure 1*



*Figure 2*



*Figure 3*



*Figure 4*



*Figure 5*

### **High Indices in Synthetic Emerald**

We have seen some interesting synthetic emeralds again during the period since the last Lab report. We received a ring for identification in which were set three Lechleitner synthetic-emerald overgrowths on beryl. The center stone had indices of 1.59 plus to almost 1.60. We have never encountered anything approaching this in the past. As a matter of fact, we have not seen many of these Lechleitner types in jewelry since their

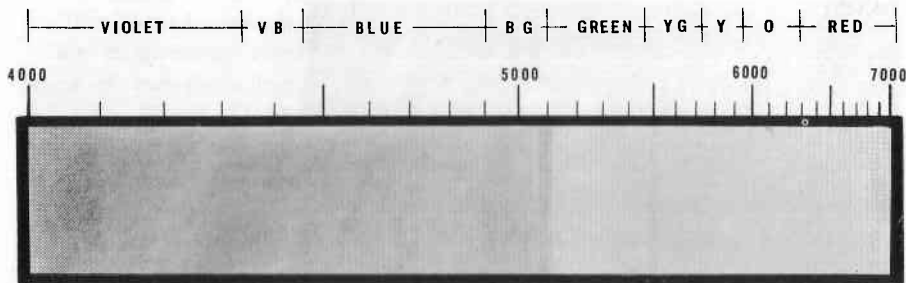


Figure 6

hey-day a few years ago. We were very surprised to encounter such high indices. Only one of the three stones could be read on the refractometer, because they were set below the center stone.

#### Linde Hydrothermal Emerald

Recently, we had the opportunity to examine the latest Linde hydrothermal emerald and found it distinctly different from the initial product. The refractive indices were approximately 1.571-1.578 and the specific gravity, 2.678. There was much less fluorescence to both long- and short-wave ultraviolet than in the earlier hydrothermal product, but it was still pronounced. It still shows red to a transmitted light beam. The daggerlike spaces topped by phenakite crystals were still present, and there were wispy two-phase inclusions. These are shown in *Figures 7* and *8*, taken at 210x.

#### More Synthetic Emerald

A flux-fusion synthetic emerald with quite a number of inclusions also contained a beautiful large phenakite crystal (*Figure 9*).

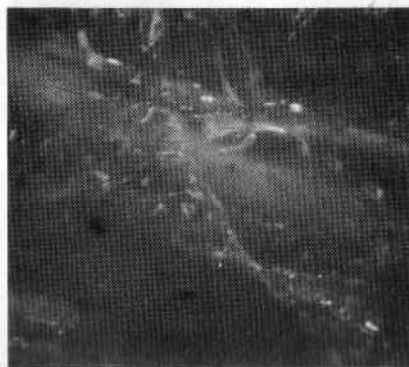
#### Inclusions in Phosphophyllite

Within the last month we had occa-

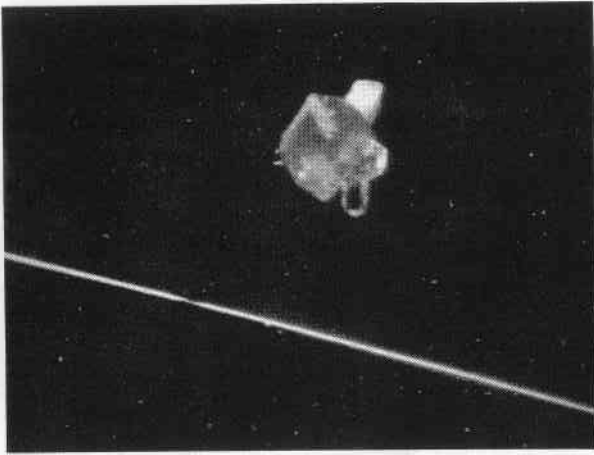
Figure 7



Figure 8



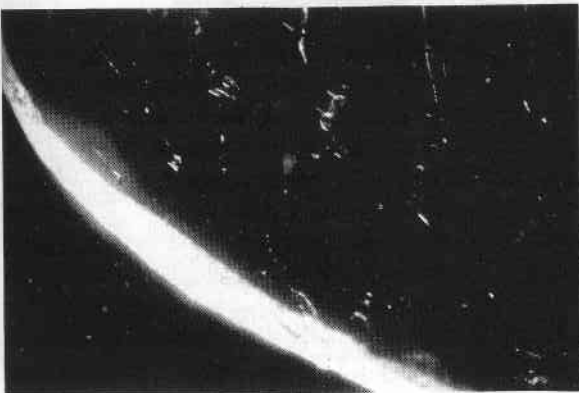
sion to test a large stibiotantalite and the largest phosphophyllite we have ever seen. *Figure 10* shows a two-phase inclusion in the latter taken at 63x.



*Figure 9*



*Figure 10*



*Figure 11*



### Two-Phase Inclusions in Garnet

We have never encountered, nor heard of, two-phase inclusions in garnet, but *Figure 11* shows such an inclusion under 63x. The stone in question was a spessartite-almandite. The manganese lines were stronger than the almandite lines.

### Synthetic-Rutile Fraud

We had a greenish-yellow synthetic rutile that had been sold as sphen. The interesting fact about it was that it had some very natural-appearing inclusions of a type we have seen before in synthetic rutile. The inclusions were tiny, present in large numbers, and imparted a cottony appearance to the stone (*Figure 12*).

### Coated Amber

We had sent in to the Lab for identification a cabochon that proved to be amber, but we were disturbed by its appearance. In places, it appeared to have something on the surface. We were able to prove that it did have. The odd appearance is obvious in *Figure 13*. When it was turned to the side, the coating on the base showed

up as a dark line just above the tweezer arm (*Figure 14*). We were unable to determine the exact nature of the coating, but it was coated to darken the color.

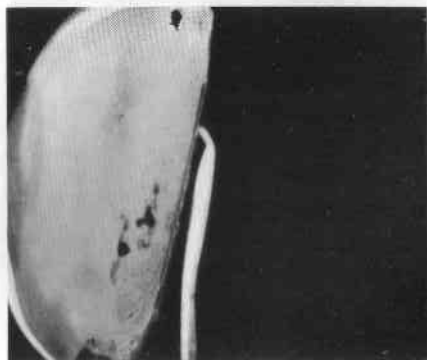
### Chrysocholla Opal

Graduate Gemologist Felix Chang of Taipei, Taiwan, was in a few months ago and brought a very interesting blue-green cabochon that had been brought to him as he was about to depart for the United States by a gem dealer in Taiwan. The dealer had

*Figure 13*



*Figure 14*



*Figure 12*



assumed that it was chrysocolla quartz and had noted that when it was cold it was translucent, but that when it became warm, it showed much less translucency and a number of cracks. Upon checking its identity, it was interesting to learn that it was not quartz but chrysocolla opal, the first we had encountered. We discovered that it was not a matter of cooling and heating but of the amount of moisture it contained, because this acted much like hydrophane opal, in that with moisture added it was translucent, as shown in *Figure 15*. When it dried, the fracture became visible and it became more opaque, as shown in *Figure 16*.

#### Banded Glass

Another rather unusual identification involved a green stone thought by the client to be an emerald. It proved to be glass but had very unusual strong banding, as shown in *Figure 17*. When the same stone was examined in a polariscope, the same

strong banding showed up in an odd strain effect that resembled twinning, as seen between cross polaroids (*Figure 18*).

#### Free-Form Cutting

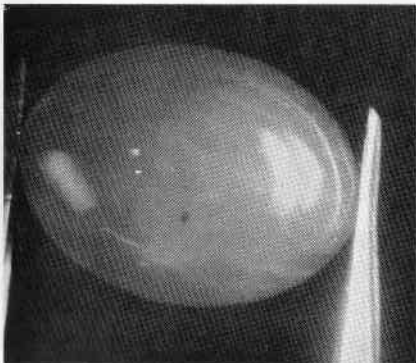
Warren Jones, now associated with the Venice Lapidary Guild of Southern California, donated to the Institute ten examples of the free-form cutting that his group is doing (*Figure 19*). These included aquamarine, chrysocolla quartz, nephrite, turquoise, jadeite, lapis-lazuli, morganite, etc. They are very interesting shapes for today's market—something new in the *avant-garde* gem-cutting field.

#### Acknowledgements

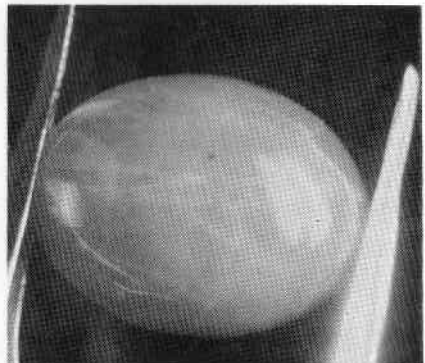
We wish to express our sincere thanks for the following gifts:

To M. Charbonneau of Ammolite Minerals, Ltd., for an assembled stone consisting of a base of highly iridescent fossil ammonite shell and a colorless quartz top.

*Figure 15*

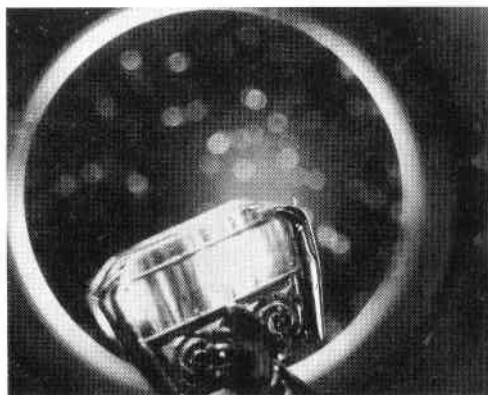


*Figure 16*





*Figure 17*



*Figure 18*



*Figure 19*

To **Ben Gordon**, Gordon Jewelry Co., Houston, Texas, for a large assortment of colored stones for our Gem Identification classes.

To **Arnold Schiffman**, Schiffman's, Inc., Greensboro, North Carolina, for a green, transparent, faceted grossularite

and a pink, pear-shaped, Lake Biwa fresh-water pearl.

To the **Washington, D. C. Guild of the American Gem Society** for a large assortment of colored stones for our identification sets.

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# Book Reviews

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*THE ENGRAVED GEMS OF THE GREEKS & THE ETRUSCANS*, by Gisela M. A. Richter. Published by Phaidon Press, New York City, 1968. 339 pages. Cloth-bound. 13" x 9" format. Illustrated with 1500 black-and-white photographs and line drawings. Price: \$45.

This monumental work is destined to become a standard reference in the field of classical archeology. It is the most important book of its kind since Furtwängler's preëminent treatise published in 1900. It will be followed by a companion volume on the engraved gems of the Romans.

Dr. Richter presents a representative selection of 695 Greek and 181 Etruscan examples. They were chosen from all major American and European collections, to emphasize their beauty and interest. In most cases, an illustration of the carved gem in its original, minute size is accompanied by an enlarged illustration of the impression, thus showing the design as it was intended to be seen when used as a seal.

The material is arranged chronologically, with an introductory chapter for each period, preceded by a general introduction dealing with the uses of engraved gems, the choice of designs, the materials used, and information about the artists. The book thus presents a history of Greek and Etruscan art in miniature.

Etruscan gems are treated in the same way. They, too, are of great interest both artistically and historically, for they shed much light on the relationship between the Greeks and Etruscans, and many of them preserve Greek legends that have not otherwise survived.

*The Engraved Gems of the Greeks & the Etruscans* will be a work of vital interest for

all those who appreciate this ancient and honorable craft and who wish to have the most definitive work available today on this always fascinating and sometimes complex subject.

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*CHINESE JADE OF FIVE CENTURIES*, by Joan M. Hartman. Published by Charles E. Tuttle Co., Inc., Rutland, Vermont, and Tokyo, Japan, 1969. 172 pages. Cloth-bound. Illustrated with black-and-white and ten color plates. Price: \$12.50.

Unlike many other books on jade, which treat the subject from its earliest beginnings as an art form to modern times, often in a cumbersome and technical style, *Chinese Jade of Five Centuries* is confined to the Ming through the Ch'ing dynasties — the period when the art of jade carving was at its height.

The author points out that most of the truly superb jade carvings were executed during these five centuries. She describes a wide sampling of outstanding pieces from American museums only, tracing in a popular and straightforward style the Buddhist influences from the Ming through the Ch'ing dynasties as a very definite transition in style and workmanship, while demonstrating the inherent symbolism in the decorative motifs of these jades.

Mrs. Hartman has conveyed the traditional Chinese philosophy and way of life by including brief descriptions of the court, pertinent historical data, and an interpretation of the symbolism that appears on these jades.

Written for the layman, the text should satisfy the more discerning and knowledgeable reader, the student and collector.

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