
NEW TECHNOLOGIES OF THE 1980S: THEIR IMPACT IN GEMOLOGY

By Emmanuel Fritsch and George R. Rossman

The 1980s witnessed great development in the application of new technologies to gemology. These technologies provided new or better ways to grow synthetic gem materials and to treat natural ones. They also permitted numerous breakthroughs in gem identification, in areas where classical gemological methods were no longer sufficient to make a positive identification. In particular, various types of spectroscopy proved to be of important practical value, for example, infrared absorption, X-ray fluorescence, Raman scattering, and cathodoluminescence.

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The decade of the 1980s was marked by the development of existing technologies for crystal growth and gem treatment, and by the introduction of a number of new technologies to the field of gem identification (figure 1). Improvements in melt and hydrothermal growth techniques put new and better synthetic gemstones on the market, while new applications of irradiation technology resulted in a dramatic increase in the availability of different hues of irradiated blue topaz and other gem materials. Even as well known a technique as the oiling of emeralds reemerged in another application as the filling of surface-reaching fractures in diamonds. Prior to this decade, traditional instruments—such as the refractometer and the microscope—were the principal means of gemological study. Only a few advanced testing techniques were used, and only in major gemological laboratories, to supplement traditional methods: U.V.-visible spectrophotometers recorded quantitative absorption data, and the electron microprobe provided detailed, nondestructive chemical analyses. For the most part, the combination of these methods proved adequate to fully characterize natural gemstones as well as the limited variety of synthetic and treated gem materials then available.

In the 1980s, however, new challenges required more advanced techniques for examining gem materials. These challenges arose for several reasons:

1. The range of new technologies available to science markedly increased. For example, the early 1980s witnessed a dramatic rise in the use of computers and microprocessors. These smaller, more powerful, faster machines helped improve both the design and performance of all types of equipment, including those used for growth, treatment, and characterization of gems.
2. A new generation of synthetic gem materials was developed that provided additional challenges in gem identification.

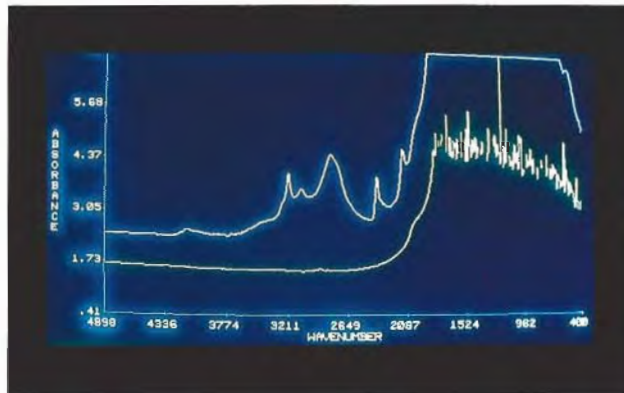


Figure 1. Significant advances in the technology of Czochralski pulling led to the increased production of Czochralski-pulled gem-quality synthetic alexandrite during the 1980s. In the Czochralski-pulling melt growth process, a furnace such as the one illustrated at left is used to grow synthetic alexandrite similar to that produced by Kyocera Corp., shown here to the right of its natural counterpart from Tanzania. Natural alexandrite (top spectrum) can be separated from its synthetic counterpart (bottom spectrum) using infrared spectroscopy, which was applied much more broadly in gemology during the 1980s. Furnace photo courtesy of Union Carbide; stone photo by Robert Weldon.

3. The spectacular growth in the volume of the colored stone market acted as an incentive both to treat natural materials and to improve synthetic ones.
4. The emergence of consumerism, and in particular the increased knowledgeability of the general public, created a demand for more and better information on gem materials, especially in areas such as radioactivity that assumed new importance with the increased volume of treated stones.

In this article, we review new technologies that had a demonstrable impact in gemology during the 1980s. Several of these technologies existed prior to 1980, but at that time had little effect on gemologists or on the gem-buying public. Although most of the recent advances in technology for gem identification are used almost exclusively in sophisticated gemological testing laboratories, it is important that the jeweler-gemologist understand the principles of these techniques and the new capabilities they represent. We will not dwell on those technologies demonstrated during the

search has been conducted into the growth of diamonds with a minimum of defects or inclusions (Kanda et al., 1989). The result is larger crystals of better quality and in greater numbers (Shigley et al., 1987). For example, in 1989, Sumitomo Electric Industries was producing 5-ct gem-quality synthetic yellow diamond crystals on a routine basis using high-pressure technology (Yazu, 1989). At present, however, these are grown for industrial purposes only and are not marketed to the jewelry trade. Major problems are still encountered in the production of large, gem-quality, colorless and blue synthetic diamonds.

The low-pressure synthesis of diamond also received considerable attention in the second half of the 1980s. Using variations of a long-known growth technology called chemical vapor deposition (CVD), very small synthetic diamond crystallites (0.5 to 20 microns in diameter) are grown either isolated to produce powders, or in a continuous layer to produce thin films. Plagued at first with serious limitations, such as poor adherence of the film to its substrate, this new growth technique is now used to place synthetic diamond films on consumer products such as drill bits; free-standing synthetic diamond thin-film tweeters have been produced by several companies (Obata and Okamura, 1989). Also, although these films were initially thin and opaque, rapid advances are now being made to produce thicker, colorless and transparent films (figure 2). This same process is also being used to produce another material, diamond-like carbon, a type of amorphous carbon with high hardness and low deposition temperature. This new low-pressure technology may have a number of applications in the gem trade, as discussed in Fritsch et al. (1989).

Treatment Processes. Treatment of gem materials has been practiced for literally hundreds of years (Nassau, 1981a). Over the last decade however a number of efforts were made to improve known treatment methods or develop new ones.

Irradiation technologies, in particular, played a major role in this area. The use of gamma rays to enhance the color of tourmaline became more prevalent during the last decade (Ashbaugh, 1988; figure 3). Although the first commercial electron-irradiation operation for gemstones began in 1976 (J. Borden, pers. comm., 1989), only a small portion of this early production was seen on the U.S. market. During the 1980s, however, electron irra-



Figure 2. By the end of the 1980s, free-standing transparent, near-colorless synthetic diamond thin films as large as 2 cm ($3/4$ in.) in diameter were being grown by the new low-pressure technology. Such thin films are now used in masks for X-ray lithography (bottom) or as windows for X-ray fluorescence spectrometers (top). Photo courtesy of Crystallume.

diation facilities (figure 4) were responsible for dramatic increases in the amount of irradiated material in the U.S. market, especially blue topaz (Schmetzer, 1987). Worldwide, there was a spectacular increase in the production of neutron-irradiated blue topaz. During this same period, gemologists became aware of multistep enhancement processes, in which combinations of irradiation in either a nuclear reactor or a linear accelerator (or both) were used to produce materials called in the trade "American," "California," "Electra," or "Swiss blue" topaz (Ashbaugh, 1988; Fournier, 1988). Other gemstones, such as diamonds, are also treated in nuclear reactors to modify their color (Kammerling et al., 1990). Because reactor treatment can be less costly than electron irradiation, large volumes of gem material have been treated in this way.

Another relatively new gem-enhancement variation is the treatment of synthetic material to make it look more natural (Kammerling et al., 1990). In addition, the centuries-old practice of oiling emerald has been creatively revisited. Better documentation of optical properties of materials (a consequence of increased interest in materials science during the decade) has led to the develop-



Figure 3. Today, pale tourmalines (left) are commonly irradiated to darker pink to red colors (right) using gamma irradiation, a technology that became more widely used for gem enhancement during the 1980s. Some of these stones (largest is 5.91 ct) are courtesy of George Drake; photo © Tino Hammid.

ment of new filling materials to make surface-reaching fractures less apparent. The late 1980s provided us with two examples: Opticon, a product marketed as an industrial fracture sealer that

Figure 4. During the 1980s, great quantities of gem materials were irradiated in an electron accelerator for the purpose of inducing or enhancing color. This computer-generated image is the electron accelerator at the Rensselaer Polytechnic Institute, Troy, New York. Photo courtesy of Rensselaer Polytechnic Institute.



has been used to fill fractures in emeralds [Koivula and Kammerling, 1989]; and a low-melting-point lead oxychloride-based glass, with an index of refraction close to 2.4, that has been used to fill fractures in diamond [Koivula et al., 1989].

NEW TECHNOLOGIES IN GEM IDENTIFICATION: PROVIDING SOLUTIONS

When classical gemological methods do not provide enough clues to solve a gem identification problem, laboratory gemologists turn to more sophisticated techniques. During the 1970s, these were U.V.-visible absorption spectroscopy and electron microprobe analysis. While some progress has been made in these techniques, especially microprobe analyses, other new technologies are now routinely used in some gemological laboratories.

Electron Microprobe. The chemical composition of most gem materials can be determined with an electron microprobe (see, e.g., Dunn, 1977). In recent years, the gemological applications of this instrument have also benefited from the introduction of new technologies. One of the long-standing limitations of the electron microprobe was its inability to measure the concentration of light elements, in particular oxygen. The 1980s saw the development of detection systems, such as multi-layered diffraction crystals, that greatly improved the detection efficiency for light elements (Arm-

1980s for which there are no immediately apparent commercial applications to gemstone production, treatment, or testing.

The article is divided into two major sections. The first deals with new technologies in the field of gem material synthesis and treatment. The impact of these technologies on gemology can be estimated in terms of the volume, quality, or availability of material produced.

The second section discusses new technologies available for gem identification. We will briefly describe the new technologies being used, the types of information they provide, and their limitations. Examples are provided to illustrate how each technique has helped improve our means of identifying gem materials.

NEW TECHNOLOGIES IN SYNTHESIS AND TREATMENT: THE CHALLENGES

A major development that affected all techniques of crystal growth during the 1980s was the introduction of computer controls, especially through microprocessors and personal computers, for furnaces and other growth devices. This resulted in a better reproducibility and yield of crystals, thereby lowering the cost of synthesis (B. Chai, pers. comm., 1989). In the U.S. over the past 10 years, most industrial crystal growers have shifted their focus to the growth of thin films on silicon and other semiconductor materials for electronic applications, so the number of people involved in growing "bulk" transparent crystals has significantly decreased. In contrast, research groups in China have initiated an ambitious program to grow synthetic crystals for the jewelry industry, building on their experience in the growth of other types of oxide crystals (D. Elwell and B. Chai, pers. comm., 1989). Currently much effort is being devoted there to the growth of colored synthetic rare-earth garnets (see, e.g., Zhang and Liu, 1988).

The new synthetic gem materials that appeared on the jewelry market during the 1980s are described elsewhere in this issue (Nassau, 1990); therefore, we will concentrate here on the recent changes in technology that made their synthesis possible.

Growth Processes. The Czochralski pulling technique, invented in 1918, is certainly not a new technology (Nassau, 1980). However, important advancements have been made recently, centered

around furnace design and new refractory materials. These have resulted, for example, in the production of colorless sapphire crystals 15 cm in diameter, three times the size of the 5-cm-diameter oxide crystals typically grown during the 1970s (Heikkinen, 1989). Motivated by the demands of the laser industry for less light distortion, producers greatly improved the quality of the crystals as well. This was accomplished primarily by a better understanding and control of the thermal gradient in the furnace and by improved pregrowth treatment of the melt (K. Heikkinen, pers. comm., 1989). These improvements in growth techniques have led to the development of Czochralski-pulled alexandrite in the USSR (Bukin et al., 1981), China (Guo et al., 1987), Japan, and the United States (R. Kane, pers. comm., 1989). Although grown primarily for laser applications, some of these products have already appeared on the jewelry market (Trossarelli, 1986; again, see figure 1).

Hydrothermal synthesis of gem-quality materials such as quartz (amethyst) and emerald continued during the 1980s. In general, the high cost of hydrothermal synthesis makes it economically less attractive than Czochralski pulling when that alternative exists.

Image furnaces became commercially available during the last decade. Such furnaces focus intense light to provide the heat needed to grow synthetic crystals. Although a few synthetics grown by this technology have been faceted, especially synthetic corundum of different colors produced by Seiko, it is not yet appropriate for mass production. Nevertheless, image furnaces have been widely used experimentally to produce limited amounts of single-crystal spinel, ruby, cubic zirconia (NEC Corp.), and olivine (peridot; Hosoya and Takei, 1982), some up to 7 cm long. The decade has also witnessed significant progress in skull-melting technology, which has resulted in dramatic increases in the production of cubic zirconia (Nassau, 1990).

Of all the new technologies, the jewelry industry has probably paid closest attention to that which produced cuttable synthetic diamonds. Major strides made in high-pressure technology resulted in presses with an effective volume of one liter and holding pressures appropriate for diamond growth. Many crystals can be grown in the same cell, and several cells can be stacked up in one press (Yazu, 1985), improving considerably the productivity of this type of equipment. Moreover, extensive re-



Figure 5. Although not a new technology, infrared spectroscopy has recently provided solutions to many tough gemological problems. The GIA Research Department has used this Nicolet 60SX FTIR spectrometer to identify treated materials such as irradiated diamonds and plastic-impregnated turquoise, as well as to separate some natural from synthetic emeralds, diamonds, and alexandrites. Photo by Robert Weldon.

strong, 1988) and therefore the accuracy of the entire analysis.

Spectroscopy. The appeal of spectroscopic methods, which involve detecting and measuring the absorption or emission of electromagnetic radiation by a material, is that they can be used nondestructively on most gems. In the 1980s, the materials-science community adopted many techniques that had previously been used primarily in chemistry—such as infrared, X-ray, and Raman spectroscopy—to study a wider range of materials, including gems.

Infrared Spectroscopy. This technique probably brought the most new solutions to gem identification problems during the 1980s. Originally developed by organic chemists, infrared spectroscopy was first applied to mineralogy during the 1950s. Many applications to gem identification problems were developed by French and German scientists during the 1980s (Leung et al., 1983). Also at this time, Fourier transform infrared spectrometers (FTIR) became widely available (figure 5). Their speed of execution made them easier to use, and their greater sensitivity permitted the detection of weak features in the spectrum that were significant for many gem identification problems. The principles of infrared spectroscopy as they relate to gemology have been described by Fritsch and Stockton (1987).

Many materials can be identified rapidly by their infrared absorption spectra. For example, turquoise can be separated from its simulants by this technique (see, e.g., Arnould and Poirot, 1975),

amber can be distinguished from plastic simulants (Fraquet, 1987), amethyst can be separated from purple scapolite (Martin et al., 1989), and glass can be distinguished from nonphenomenal opal (C. Stockton, pers. comm., 1987; figure 6). These tests are difficult, even sometimes impossible, using classical gemological techniques.

In some cases, infrared spectroscopy can distinguish natural from synthetic materials. This is particularly important when the material contains few or no inclusions. In a number of cases, this distinction is possible because of differences in the way water is incorporated. Wood and Nassau (1968) demonstrated how flux-grown synthetic emerald could be distinguished from its natural equivalent: The incorporation of water during growth in natural stones causes absorption around 3600 cm^{-1} in the infrared spectrum, whereas the flux material uses no water so the corresponding synthetics lack absorption in this region. This technique has been refined to the point that not only can natural emeralds be distinguished from their synthetic hydrothermal equivalents, but also the products of various hydrothermal synthetic emerald manufacturers can be separated from one another (Leung et al., 1983; Stockton, 1987).

The distinction between natural and synthetic alexandrite is based on the same principle (Leung et al., 1983; Stockton and Kane, 1988). The fact that water was incorporated during growth in natural alexandrite results in an infrared spectrum that is different from that of most synthetic alexandrites (grown by a melt technique), which lack "water."

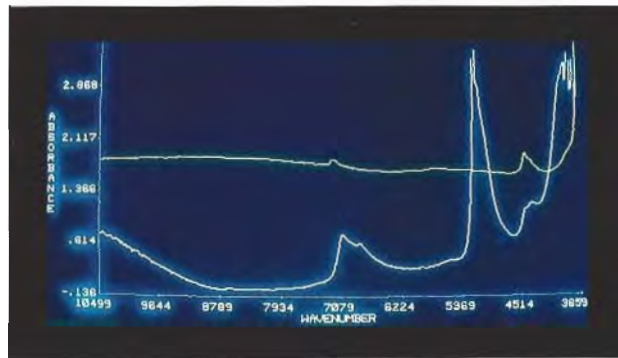


Figure 6. These orange and near-colorless opals (center) are difficult to separate from their glass imitations (either side). However, the infrared spectra of natural glass (top) and opal (bottom) differ significantly, providing a ready means of identification. Photos by Robert Weldon.

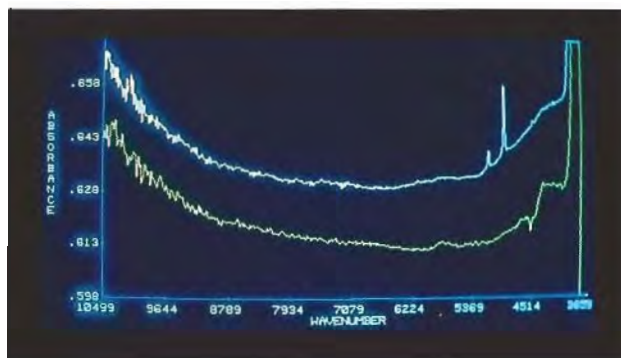
Numerous attempts have been made to separate natural and synthetic amethyst on the basis of their infrared spectra (Zecchini, 1979; Schmetzer and Bank, 1980; Lind and Schmetzer, 1983). In the vast majority of cases, the spectrum of synthetic amethyst will show an absorption band around 3540 cm^{-1} , probably related to a potassium-bonded hydroxyl group (Kats, 1962). This band is absent from the spectrum of most natural stones (Fritsch and Koivula, 1987).

Infrared spectroscopy can also detect the presence of polymers in materials that have been impregnated to improve their appearance or physical cohesion, such as opal (Fritsch and Stockton, 1987), turquoise (Dontenville et al., 1985), and jadeite (Hurwit, 1989). Other types of treatments are occasionally detectable; for example, sugar-

treated opals show a typical infrared spectrum (C. Stockton, pers. comm., 1987).

Quite possibly the most important application of infrared spectroscopy in the last few years has been the detection of treatment (irradiation followed by annealing) in yellow to brown nitrogen-containing diamonds. In 1957, Crowningshield demonstrated that the presence of a band at 5920 Å (since determined on more accurate instrumentation to be 595 nm) in the visible range is proof of treatment in such stones. It appears, however, that the defect creating this absorption can be annealed out at high temperature, so that the indications of treatment provided by the visible spectrum are less clear. The presence of two sharp peaks in the near infrared (at 1936 and 2026 nm , called H1c and H1b, respectively), can then be taken as a positive

Figure 7. These treated (by irradiation and annealing) yellow diamonds were subjected to a second heating to high temperatures to make the 595-nm band—considered proof of treatment in yellow diamonds—disappear. However, infrared spectroscopy helped identify two sharp bands in the infrared (top spectrum), caused by the annealing process, that are absent from the spectrum of a natural yellow diamond (bottom). Photos by Robert Weldon.



proof of treatment in yellow to brown (Collins et al., 1986; Woods and Collins, 1987; figure 7) and reirradiated green (Fritsch et al., 1988) diamonds.

X-ray Spectroscopy. Energy dispersive X-ray fluorescence (EDXRF) analysis is another relatively sophisticated technology that was first applied to gemology during the last decade (Hänni, 1981). In an XRF spectrometer (figure 8), an X-ray beam directed at a sample causes the individual chemical elements in the sample to emit X-rays of a characteristic energy. The instrument's solid-state detector and multichannel analyzer sort the X-rays, making it possible to detect which elements are present. When the instrument is carefully calibrated, the intensity of a given peak can be quantified to indicate the concentration of the corresponding element. One of the major advantages of this technique is that a cut stone requires no sample preparation; the gem is simply placed table down in a holder above the beam.

The chemical analysis provided by XRF can have important applications in gemology. Gemological papers early in the decade refer to the separation of natural from synthetic ruby and alexandrite on the basis of the presence of molybdenum from the flux (Stern and Hänni, 1982) and the presence or absence of trace elements such as gallium, thought characteristic of natural origin, (Ohguchi, 1981; Stern and Hänni, 1982). XRF has also been used to separate natural freshwater from saltwater pearls on the basis of the much larger concentration of manganese in freshwater pearls (Miyoshi et al., 1986; figure 9). Other examples are provided in Stern and Hänni (1982).

One must be cautious when interpreting XRF spectra, however, because of the abundance of diffraction peaks and other artifacts that may lead to expensive mistakes. For example, figure 10 shows the spectrum of a flame-fusion synthetic ruby, which has no gallium, with a diffraction peak exactly at the energy expected for this element. Some laboratories use XRF as a complementary technique, for example, to separate natural from synthetic ruby (C. Schiffman, pers. comm., 1988; S. McClure, pers. comm., 1990).

X-ray fluorescence instruments historically could not detect elements lighter than sodium. The recent development of extra-thin detector windows or windowless detectors has made light-element detection possible. Interestingly, one of the materials used for very thin XRF windows is a



Figure 8. XRF technology was used throughout the 1980s by a number of advanced gem testing laboratories. The GIA Research Department found this Tracor X-ray Spectrace 5000 energy dispersive X-ray fluorescence (XRF) spectrometer to be useful, for example, in the separation of freshwater from saltwater pearls (see below) and untreated from fracture-filled diamonds. Photo by Robert Weldon.

synthetic diamond thin film, produced by the low-pressure technology (Pinneo and Conner, 1989; see figure 2).

Raman Spectroscopy. Raman spectroscopy has seen increased application to gemological research during the 1980s. Like infrared spectroscopy, it is a nondestructive vibrational spectroscopy that requires expensive equipment, especially if data are obtained with a Raman microprobe. Raman spectroscopy is extensively used in mineralogy and geochemistry (McMillan, 1989). In gemology, it has been used to identify gemstones, to distinguish between crystalline and amorphous materials, and to separate natural stones—for example, diamond and jade—from their simulants (Nassau, 1981b;

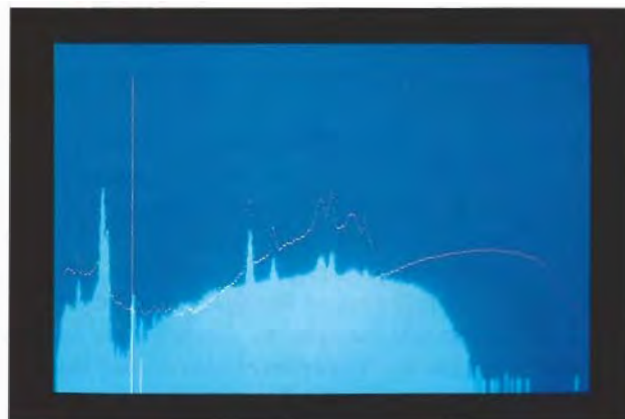


Figure 9. XRF technology can separate freshwater (left) from saltwater (right) cultured pearls on the basis of their chemical composition: Freshwater pearls (blue spectrum) contain significantly more manganese (indicated by the vertical red lines at left) than saltwater pearls (red dotted line). Photos by Robert Weldon.

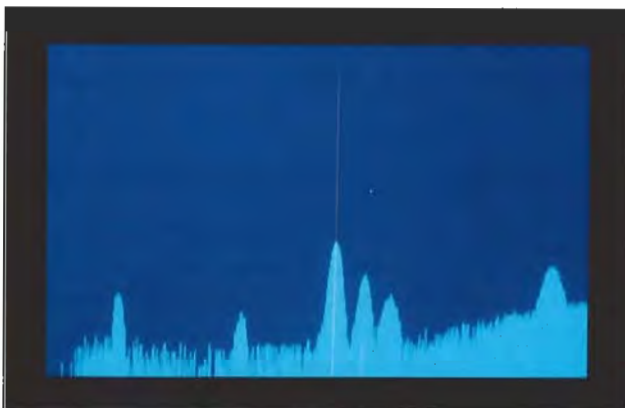
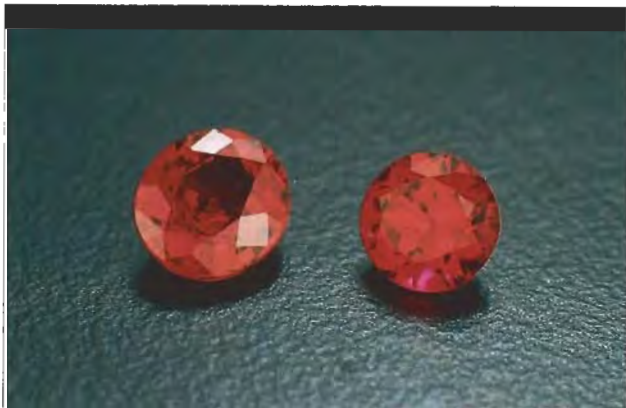
Dhamelincourt and Schubnel, 1977). It can also nondestructively identify pigments in gem materials (Délé-Dubois and Merlin, 1981).

The most important use of Raman spectroscopy in gemology is the identification of inclusions (figure 11). The Raman microprobe can focus on and identify an individual inclusion even when it is beneath the surface of a stone. This is particularly valuable in the separation of natural from synthetic materials (Délé-Dubois et al., 1986). To help with these identifications, the Gemology

Laboratory of Nantes University, France, is preparing a catalog of Raman spectra for all gem materials and their most common inclusions (B. Lasnier, pers. comm., 1989).

Cathodoluminescence. First reported by Crookes in 1879, this technology was not applied to gemological problems until the late 1970s, when a simple commercial instrument became available (Gaal, 1976–1977). Its use became more widespread during the 1980s (Ponahlo, 1988). Cath-

Figure 10. XRF has been used to separate natural (left) from synthetic (right) rubies on the basis of their trace elements. However, the gemologist must be careful of the artifacts in the XRF spectra when making a determination. For example, the XRF spectrum of a flame-fusion synthetic ruby shows (at the far right) a band at the position expected for gallium, an element presumed to be typical of natural rubies, and (closest to the first, on the left) a band at the exact position expected for iron, an element generally not present in flame-fusion synthetic rubies. Both peaks are actually due to diffraction, not gallium or iron. Photos by Robert Weldon.



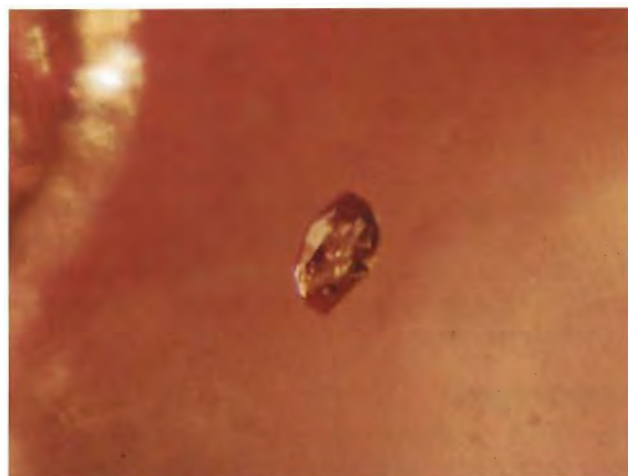
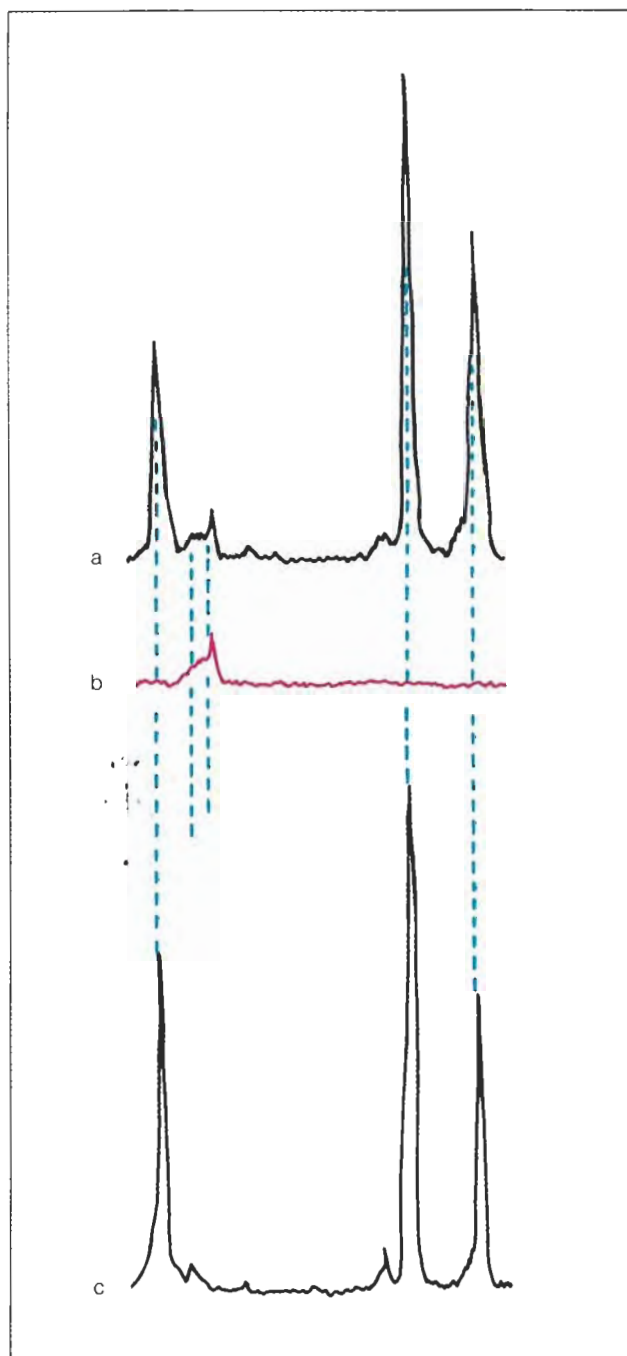


Figure 11. This sulphur inclusion in a Burmese ruby (right) was identified using Raman spectroscopy (left), one of the technologies new to gem identification in the 1980s. Spectra: a = inclusion in matrix, b = matrix alone, c = reference sulphur. This is the first time sulphur has been identified as an inclusion in ruby. Photo and spectra courtesy of J. Dubois-Fournier, Gemology Laboratory, Nantes University, France.

odoluminescence is the emission of visible light by a material excited with an electron beam in a vacuum chamber. It does not require any sample preparation, and does not affect most common gem materials. Visual observation of the color (or distribution of color) of the emitted light is enough, for example, to distinguish clearly between natural and synthetic yellow diamonds on

the basis of the distribution of their various growth sectors (Shigley et al., 1987). Emission spectra and quantitative measurements of the emissions can also be recorded and used for some gem identification purposes, such as the separation of natural from synthetic rubies, emeralds, and alexandrites (Ponahlo and Koroschetz, 1986; Ponahlo, 1988, 1989).

A number of additional technologies have been used experimentally in the characterization of gem materials. These include nuclear magnetic resonance (NMR), electron spin resonance (ESR), and proton-induced X-ray emission (PIXE). Thus far, none of these methods has helped solve gemological problems that could not be handled in a simpler fashion. Research into the gemological applications of these methods is still in its infancy, however, and the potential for future important contributions exists.

CONCLUSION

The 1980s witnessed greater opportunities for more and better synthetics and enhancements, which provided new challenges for gem identification. For example, improvements in Czochralski pulling and high-pressure diamond synthesis brought new and better products into the commercial scene. The application of various irradiation techniques resulted in the processing of large amounts of blue topaz, in a number of different shades; developments in filling materials produced new treatments in emeralds and diamonds. Several of the technologies described in this article have provided new approaches to meet these challenges. Even so, at the end of this decade the following challenges still remained:

- The distinction between natural colors and those created by laboratory irradiation. Prominent problems involve green diamonds, blue topaz, and pink and red tourmalines.
- The detection of heat treatment in gem mate-

rials such as sapphire or beryl when microscopic characteristics do not provide conclusive evidence.

- The detection of dye in gem materials.
- The determination of the geographic origin of gem materials.
- The reproducible measurement of the color appearance of a faceted gem.

New technologies developed in the 1990s will undoubtedly help address some of these outstanding problems. It is equally as likely that new technologies will generate new problems for the gemologist. We will probably see more synthetic gem materials, possibly large synthetic hydrothermal emeralds developed for laser applications. We will also need to follow closely developments in the fields of diamond synthesis, optics (especially coating technology), electro-optics, and electronics that traditionally have provided materials of potential use in the gem market.

During the past decade, gemologists tried to respond to changes imposed on them by the outside world. In the years to come, gemologists must learn to anticipate these changes. At the end of the 1980s, much of the available technology and the vast network of technological information was under-utilized by gemologists. Although the equipment required is usually expensive and may not be readily accessible, there is nevertheless a tremendous potential for new developments by exploiting techniques already available in materials science and building the data bases that will make their results useful to the jeweler-gemologist.

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