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# TECHNOLOGICAL DEVELOPMENTS IN THE 1990s: THEIR IMPACT ON GEMOLOGY

By Mary L. Johnson

*In the last decade, technology has improved how we synthesize, process, identify, and otherwise study gem materials. Significant trends include: the widespread availability of computerized communication; the application of synthesis techniques to gem treatments; the increased prominence of treated synthetics; the greater need for expensive instrumentation to solve gem problems in general, and the broader availability of small dedicated instruments to solve specific problems; and the adaptation of techniques from other sciences. Potentially applicable technology must be evaluated critically to assess its usefulness and appropriateness to solve a particular gemological problem.*

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Acknowledgments: Jacquelyn Haahr and Barak Green consolidated references; Dr. James Shigley, John Koivula, Kim Cino, and Mark Parisi engaged in discussions that much improved this manuscript. Robert Kane, Dr. Ilene Reinitz, and Thomas M. Moses also had helpful comments on the manuscript. Dr. James Shigley, Elizabeth Schrader, Dr. Karl Schmetzer, Brendan Laurs, and Stuart Overlin obtained illustrations.

Gems & Gemology, Vol. 36, No. 4, pp. 380–396  
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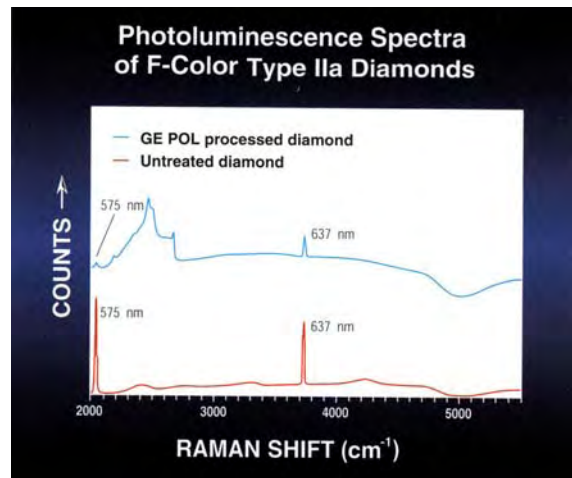
The other articles in this retrospective issue address tangible subjects: localities, synthetic gem materials, treated gems, and jewelry. In contrast, this article is about a concept, *technology*—about *how* we perform tasks differently in the gem trade than we did 10 years ago. A moment's reflection will convince the reader that the changes in the last decade have been enormous, and pervasive. This broad overview is intended to examine the many technological innovations—new methods, new instruments, and new applications for known instruments—that characterized the last decade, and to point the reader to further information. Three trends have been especially important: (1) the rise of computer-related communication; (2) the increasing sophistication of gem treatments, to the point of blurring the boundaries between treatment and synthesis (e.g., high pressure/high temperature-processed diamonds [figure 1] and flux-like fillings and new growth in heat-treated rubies); and (3) the growing need for instrumentation-based identification for gem treatments (e.g., B-jade) and synthetics (e.g., synthetic amethyst). Some widely heralded developments, however, such as the introduction of (doubly refractive) synthetic moissanite as a diamond simulant, reinforce the continued importance of “classical” gemology in this increasingly technological industry.

#### BACK TO THE BEGINNING: WHERE WERE WE IN 1990?

The Spring 1990 issue of *Gems & Gemology* (Fritsch and Rossman, 1990) described four key challenges from the previous decade that stimulated technological developments in gem testing: (1) the availability of smaller, more powerful computers for instrument control; (2) the introduction of new synthetic gem materials; (3) the advent of new treatments to meet the growing market for colored stones; and (4) the need to produce more and better information about gem materials to



Figure 1. At the end of the 1990s, Raman spectrometry became an important technique for detecting HPHT-processed type IIa diamonds, such as this 0.45 ct E-color GE POL diamond (inset). Photoluminescence spectra (below) obtained with this technique show features—particularly the ratio of the 575 to the 637 nm peak—that can be used to separate the vast majority of HPHT-processed from untreated type IIa diamonds. To achieve high-resolution spectra, the diamonds are cryogenically cooled in a specially designed sample chamber using liquid nitrogen (left). A laser beam is focused through the optical microscope, and a mirror is used to deflect the beam into the sample chamber. Photos by Elizabeth Schrader (inset) and Joe Duffy (left).



meet demand by consumers for better disclosure, especially concerning possibly radioactive gem materials. Greater computer power meant that more data could be processed faster; computer controls also lowered the cost of synthesis by providing better reproducibility and yield. New or improved synthesis techniques included Czochralski pulling, hydrothermal growth, “image furnaces” and skull melting (e.g., for YAG), and HPHT synthesis, especially for diamonds. Chemical vapor deposition (CVD) was used to produce thin-film coatings of (synthetic) diamond and diamond-like carbon; other important treatments of the ‘80s were irradiation (especially of topaz), fracture filling of diamonds and emeralds, and the treatment of synthetics to resemble treated natural materials.

By 1990, established analytical techniques with gemological applications included: electron microprobe analysis, X-ray fluorescence (XRF) for bulk chemical analyses, infrared and Raman spectroscopy, and cathodoluminescence (especially of

diamonds). Other experimentally used detection techniques were: nuclear magnetic resonance (NMR), electron spin resonance (ESR), and proton-induced X-ray emission (PIXE).

Technological changes since 1990 have had an impact on virtually every aspect of the gem business, but most importantly for the gemologist they have affected communication, cut evaluation, development of new gem treatments (and proliferation of synthetics), and growing reliance on more “advanced” techniques. Some of the procedures we use today have been developed within the last decade, but many are refinements of, or adaptations from, earlier technologies. This review does not attempt to achieve completeness, but is intended as a summary of those technological developments that were most important to the gem trade in computers and communication, gemstone cutting, synthesis and treatment, and gem identification (both classical methods and advanced techniques).

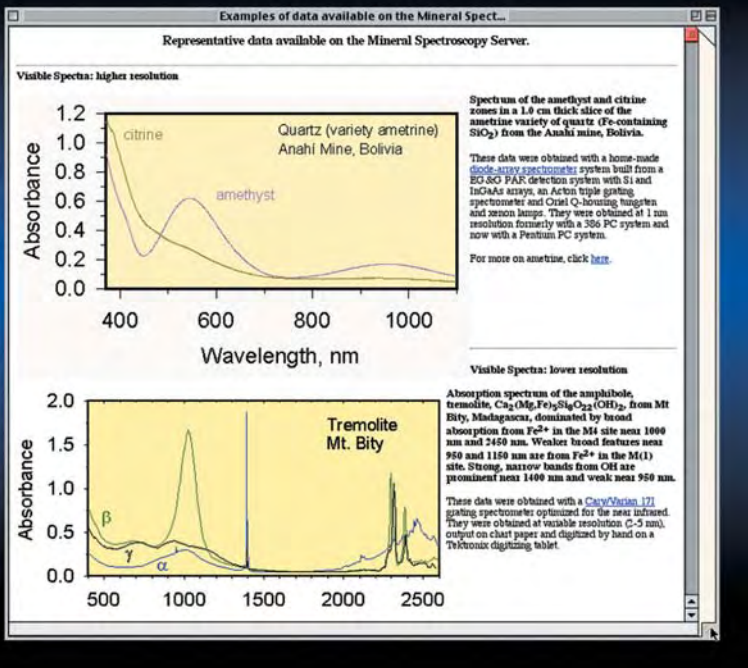


Figure 2. Today, researchers can easily access an abundance of information on the Internet, such as the spectroscopy data on Dr. George Rossman's Mineral Spectroscopy Server at <http://minerals.gps.caltech.edu>.

## COMPUTERS AND WORLDWIDE COMMUNICATION

Perhaps the most significant change in the last decade was the greater reliance on computers and computer networking. The widespread availability of vastly increased computational power and connectivity has revolutionized the way business is conducted, in the gemological laboratories as much as in the rest of the gem trade. For example, both the GIA Gem Trade Laboratory and the AGTA Gemological Testing Center use sophisticated databases to track gem identification and grading data within the laboratory (K. Cino, pers. comm., 1998; K. Scarratt, pers. comm., 2000). Within any business, computers can improve accounting and inventory practices (Golding, 1991), for which commercial software is available (see, e.g., Greig, 1999). The increase in computer power also led to smaller and less-expensive sophisticated instruments; for instance, both Sarasota Instruments (Osprey, Florida) and Adamas Gemological Laboratory (Brookline, Massachusetts) produced PC-based spectrophotometers (Kammerling et al., 1995c; Haske, 1999). P. Read and M. Haske produced new versions of gem identification software (Read, 1996).

Computers also have redefined the expected speed of communications. For example, although *Gems & Gemology* remains a quarterly journal, information on its web site ([www.gia.edu/gandg](http://www.gia.edu/gandg)) changes almost weekly. As another instance, on GIA's home page, a forged letter in Thailand was recently disavowed

worldwide less than two hours after it first came to GIA's attention (Alex Angelle, pers. comm., 2000). Computers connect a businessperson to an on-line community, with forums for discussion (Voorhees et al., 1999) and specialized commerce sites (business-to-business—B2B—and business-to-consumer—B2C; see, e.g., Diamond, 1999; Weinbach, 2000). Internet retailing is a new development, already going through boom and bust cycles; Janowski (1999) predicted that retailing on the Internet could represent 15% of jewelry business by 2005.

Online archives and databases continue to grow (see, e.g., "The geosciences in review," 1996), making fundamental information available for the price of a few mouse-clicks. A relevant example for the gemological community is Dr. George Rossman's Mineral Spectroscopy Server (<http://minerals.gps.caltech.edu>; figure 2), which contains an abundance of easily accessible spectroscopy data. Online gemology courses are also available on the Internet. Search engines scan the World Wide Web for relevant information, and "meta-search" engines search the search engines. The new user is cautioned, however, that nonsense on the Internet can be expressed as authoritatively as knowledge is, and that popularity is not a guarantee of quality, so the credibility of the source must always be considered in assessing information.

## NEW TECHNOLOGIES IN GEM CUTTING AND CUT EVALUATION

**Planning and Cutting Rough.** Diamond cutting and polishing (see, e.g., Caspi, 1997) involves many decisions, all meant to maximize the profit achievable from a particular piece of rough. Today, many of these decisions are being made with the help of computerized equipment: These machines can determine which cuts may be fashioned economically ("Sarin's new mapping machine...", 1999); or provide automatic centering, blocking, bruting (figure 3), polishing, and girdle faceting (Hourmouziou, 1996; see also Koivula and Kammerling, 1991d; Lawrence, 1997a, 1998). Laser cutting is another efficient way to fashion rough (Hourmouziou, 1996; Caspi, 1997).

A new theoretical model for the anisotropic behavior of diamond during polishing (that diamond polishes more easily in one direction than another) is based on local conversion of diamond to graphite on the surface during polishing (Van Bouwelen et al., 1997). A new machine, the Horizon 200, has been developed to measure the smoothness of a diamond's surface; results suggest that the more slowly material is removed in the last stages of diamond polishing, the

better the finish quality will be (Lawrence, 1997b).

Software for designing colored stone cuts is available (e.g., Gem Cad [<http://www.gemcad.net>]; see also Atwell and Hunt, 1993). Automation has also been applied to the fashioning of colored stones. The CSIRO in Australia developed equipment for robotic cutting of opals (Cody and Brown, 1992). In 1994, Golay Buchel demonstrated machine-cut calibrated synthetics and simulants (e.g., synthetic spinel triplets that resemble emeralds) as small as 1 mm in diameter (Koivula et al., 1994a). In 1995, Swarovski debuted machine-cut calibrated colored stones, with a possible production of 300,000 items per day (Kammerling et al., 1995h).

On a related note, many gems respond differently to different types of illumination, and the appearance created by the halogen lamps now generally used in retail jewelry displays is quite different from that seen by the consumer in a more typical at-home or workplace lighting environment. Therefore, Eickhorst (1999) recommended that other types of illumination, such as modern fluorescent lights, might be used to better purpose in selling gems.

**Evaluation of Diamond Cut.** Although many people “know” that the best cut for a round brilliant diamond was determined by Marcel Tolkowsky in 1919, research on proportions and cut began before then and has continued to the present day (see, e.g., references cited by Hemphill et al., 1998). All such models rely on assumptions about the source of illumination (spectrum, location, size, and shape) and the position of the diamond relative to the observer (e.g., Love, 1989, assumed that a diamond is more brilliant if it looks brilliant while tilted as well as face-up). Some modern analyses rely on computers to predict the optical performance of various cuts and sets of proportions (see, e.g., Hemphill et al., 1998; Gilbertson, 1999; <http://www.gemology.ru>); but geometric models continued to be produced, mainly in the Japanese literature (see, e.g., Kato, 1991). At this time, there is no consensus as to the “best” set of proportions for round brilliant cut diamonds.

## NEW TECHNOLOGIES IN SYNTHESIS AND TREATMENT

Innovations in synthesis and treatment, and the application of existing technologies to new starting materials, led to a variety of new gem materials in the 1990s. Because there are articles in this issue that address synthetics and treatments in depth (Koivula et al., 2000; McClure and Smith, 2000), the

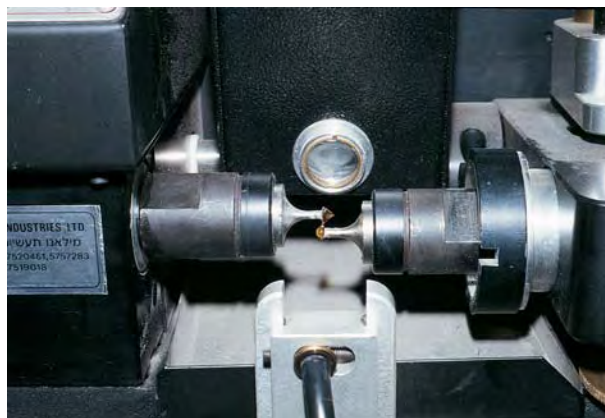


Figure 3. Automatic bruting machines were one of the many innovations in planning and cutting diamond rough in the 1990s. Photo by James E. Shigley.

discussion here will focus on synthesis and treatment technology rather than identification.

**Colored Stone Synthesis.** As discussed in the “Synthetics” article in this issue (Koivula et al., 2000), Russia and China were important centers of gem synthesis during the decade (see, e.g., Schmetzer, 1990; Bukin, 1992; Thomas et al., 1997; Johnson and Koivula, 1998e; Tauson et al., 1998; Balitsky et al., 1998, 1999a,b; and synthetic diamond references cited below). At the VNIISIMS facility near Moscow in 1994, about 500 researchers were investigating synthetic gem materials, using equipment such as the furnaces shown in figure 4 (Koivula et al., 1994c).

Anhydrous (without water) crystal growth techniques described include pulled-melt growth techniques for synthetic forsterite (Nassau, 1994); top-seeded solution growth and pulling from a melt with a continuously varied composition (applied to fluorides, but adaptable to other synthesis challenges; Koivula et al., 1992c); flux growth of synthetic alexandrite (Schmetzer et al., 1996) and synthetic spinel (Bukin, 1992); and growth of gem-quality synthetic moissanite by the Lely sublimation process (Nassau et al., 1997). Czochralski-pulled synthetic corundums made from naturally occurring starting materials were represented incorrectly as “recrystallized” rubies or sapphires (Kammerling et al., 1995g). Flux-grown synthetic emerald crystals grew larger if phosphorus was added to the flux (Kayama and Kuwano, 1998), or if grown from oriented seeds in a rotating crucible (Barilo et al., 1999). A study of yttrium-aluminum oxide melts explained why large YAG crystals cannot be grown from a flux: The melt separates into two viscous fluids that do not recombine



Figure 4. Although not new technology, furnaces such as these in Chernogolovka, Russia, were used to grow large quantities of synthetic gems in the 1990s. Photo by James E. Shigley.

easily (Aasland and McMillian, 1994). Growth zones in hydrothermal synthetics may be due (at least in part) to sporadic temperature fluctuations during the growth process (Thomas et al., 1999).

**Diamond Synthesis.** Throughout the 1990s, HPHT diamond synthesis employed a variety of large presses (see, e.g., figure 5). These included a belt-type apparatus (a piston/cylinder press with a special, strengthened central cylinder); multiple-anvil presses with

Figure 5. This BARS apparatus in Novosibirsk is typical of the split-sphere presses commonly used to manufacture Russian synthetic diamonds, such as the 0.04–1.07 ct samples in the inset. Shown here is the sample chamber as viewed from directly above. Photo of press by James E. Shigley; examples in inset courtesy of Alex Grizenko, photo by Maha Tannous.



tetrahedral, octahedral (Koivula and Kammerling, 1991b), or cubic symmetry; and split-sphere presses (sometimes called BARS for the Russian initials: see, e.g., Koivula et al., 1992d; Shigley et al., 1993b). Most HPHT synthetic diamonds are grown in a nickel-iron flux (see, e.g., Burns et al., 1999; Choudhary and Bellare, 2000); high-quality synthetic diamonds, with fewer defects than natural diamonds, can be grown using a temperature gradient in the HPHT cell (Pal'yanov et al., 1998). Efforts have been made to grow diamonds from graphite-carbonate (Akaishi et al., 1990), -hydroxide, -sulfate, -phosphate and -borate fluxes (Kanda and Akaishi, 1991), as well as from a silicate (kimberlite) flux (Arima et al., 1993), but so far the resulting crystals have been very small. However, as many of the characteristic features of synthetic diamonds—e.g., magnetism, types of inclusions, and luminescence spectra—are a result of the fluxes used, the development of alternative fluxes must be watched carefully.

**Gem Treatments.** Heat treatment of rubies and yellow and blue sapphires was performed in high-temperature furnaces (figure 6) with carefully controlled atmospheres; methods used during this decade were described in detail by Themelis (1992) and Emmett and Douthit (1993), with additional details gleanable from Themelis (1995) and Johnson et al. (1999a). Wang et al. (1992) used molten salt baths, at somewhat lower temperatures (900°C rather than 1200°–1800°C or so), to improve the color of blue sapphires. Diffusion treatment, which introduces chromophores at and near the surface of fashioned corundum, was described by Kane et al. (1990). As the decade progressed, the depth of possible diffusion treatment increased, and in some samples the need for repolishing was eliminated, minimizing the characteristic color concentrations at facet junctions (Emmett, 1999).

The most contentious emerald treatment was clarity enhancement (fissure filling); controversies raged as to whether disclosure rules should be different for emeralds that had been “oiled” than for those so treated with artificial resins such as Opticon and the new Gematrat and Permasafe treatments. Work by Zecchini and Maitrallet (1998) and Hänni and colleagues (see, e.g., Hänni et al., 1996a,b; Chalain et al., 1998) indicated that artificial resins and commonly used “essential oils” had different infrared and Raman spectra. However, using the same instrumentation on a greater number of known and potential fillers, Johnson et al. (1999c) showed that artificial

resins could not be distinguished from such oils with 100% certainty. Emerald treatment equipment (figure 7) became available in the early 1990s from several sources, including Israel (e.g., Koivula et al., 1993a; Koivula et al., 1994b).

Instances of stones rendered radioactive by treatment continue to be discovered. In the last decade, americium was used to irradiate diamonds (Ashbaugh and Moses, 1993; Reinitz and Johnson, 1994), some of which will be radioactive for thousands of years. Reactor-irradiated (and radioactive) ruby (Johnson and Koivula, 1998d) and cat's-eye chrysoberyl (to make "chocolate-brown" body color; Johnson and Koivula, 1997d) were discovered in circulation; in some cases, these could be quite hazardous.

Toward the end of the decade, new identification challenges emerged on two fronts: (1) the distinction of treated synthetics from synthetics, and (2) the use of synthesis methods to treat natural gem materials. Synthetics are treated for several reasons: to achieve colors not otherwise available (e.g., red treated-color synthetic diamonds: Moses et al., 1993), to disguise the synthetic nature of the starting material (e.g., quench-crackled or diffusion-treated synthetic corundum: Johnson and Koivula, 1997b, 1998b; Free et al., 1999), or to "stabilize" low-quality starting material (e.g., polymer-impregnated synthetic opal: Johnson and Koivula, 1998c). Some laboratories, such as the GIA Gem Trade Laboratory, now disclose certain treated synthetics on identification reports (e.g., treated-color synthetic diamonds, diffusion-treated synthetic corundum, and polymer-impregnated synthetic opal: Shane McClure, pers. comm., 2000).

In the past, synthesis methods applied to treatment have entailed creating overgrowths on existing gems (such as CVD overgrowths on diamonds and Lechleitner synthetic emerald overgrowths on beryl), and such techniques continue to be used. However, even techniques thought to be used simply for treatment can create new growth; for instance, Mong Hsu rubies show new crystal growth with heat treatment (Johnson and McClure, 2000). High-temperature techniques that can be used for both synthesis and treatment blur the line between treated gems and synthetics.

One of the most important recent developments in diamond treatments is the use of high pressure and high temperature for short amounts of time to either decolorize the diamond or give it a more attractive color. This treatment works by changing the aggregation state of nitrogen in the diamonds, introducing or modifying defects, and possibly chang-



Figure 6. High-temperature furnaces such as this one were used with carefully controlled gas mixtures to perform heat treatment of rubies and yellow and blue sapphires (see inset for stones before [top] and after treatment; average size 4.5 mm). Furnace photo courtesy of Linn High Therm GmbH; inset photos © GIA and Tino Hammid (top) and © John L. Emmett (bottom).

ing the degree of strain (see, e.g., Smith et al., 2000, pp. 194–195). Synthetic diamonds have been "annealed" (70–80 kbar pressure and 2000°–2200°C for 4–5 hours) to produce yellow and greenish yellow hues (Shigley et al., 1993a). Fancy yellow synthetic diamonds with type IaA aggregates can be produced by treating typical (type Ib) synthetic yellow diamonds at high pressure (Koivula et al., 1992d). HPHT processing of some brownish type IIa natural diamonds can remove color to the point of rendering the stone colorless (D–F); early experiments with this method were done in Russia (Teslenko, 1993), and recently General Electric has been processing diamonds in this fashion for commercial distribution (for further information on these GE POL diamonds, see, e.g., Johnson et al., 1999b; Moses et al., 1999).



Figure 7. In the early 1990s, emerald treatment equipment became available from several sources. The “Mini Oiler” shown here uses a hand-operated vacuum-pressure pump both to remove air and moisture from fractures and to create pressure for fracture filling. Photo courtesy of Colgem-Zamrot.

Other brown type Ia diamonds turn greenish yellow to brownish yellow (Henn and Milisenda, 1999; Reinitz et al., 2000—see figure 8). For a review of related technology, as revealed in recent patents for HPHT treatment, see Schmetzer (1999).

#### NEW TECHNOLOGIES IN GEM IDENTIFICATION

**Gemological Techniques.** “Classical” gemology is a relatively mature field; consequently, technical gains in this area over the last decade have for the most part been small refinements and discoveries, not major innovations. Among these were:

- Refinement of weight estimation formulas for mounted stones (Carmona, 1998a,b)
- A procedure to measure the specific gravity of mounted goods (Mitchell, 1992)
- Alan Hodgkinson’s “visual optics,” including a method to distinguish diamond from various substitutes by noting the pattern of dispersed flashes from a fashioned stone (Hodgkinson, 1989), as well as tests with simple equipment to estimate or determine optic character, birefringence, dispersion, pleochroism, and refractive index (Hodgkinson, 1995)
- Confirmation of appropriate tests to identify black opaque gem materials (Johnson et al., 1996), including examining the surface in polarized reflected light
- Use of polarized reflected light to help identify opaque inclusions at the surface of non-opaque gems (Crowningshield and Johnson, 1994)

- An explanation of the “Plato effect” in synthetic corundum (Johnson and Koivula, 1999), which is seen by looking edge-on at the irregular edges of mosaic crystallites
- Use of microscopic techniques from optical mineralogy by gemologists to identify small particles, such as minute scrapings from a gem (Hodgkinson, 1994)
- Obtaining the optic figure of a transparent inclusion in a transparent gemstone, using a “conoscope” (focusing lens on a microscope) in contact with the host gem (Koivula, 1993)
- Use of short-wave UV radiation to distinguish striae in synthetic sapphires (Kammerling et al., 1994)
- Use of pleochroism to tell diffusion treatment (pleochroism should exist) from coating (no pleochroism) for anisotropic gems (Koivula and Kammerling, 1991a)
- Use of a refractometer to measure dispersion, by calibrating results against benitoite and fluorite (Hanneman, 1992)
- Use of thermal conductivity to distinguish (most) synthetic emeralds from (most) natural emeralds—synthetics tend to have lower values (Read, 1990a)

**Gemological Instruments.** Innovations also continued in the development and improvement of instruments for basic gemology. The following are a few of the many advances in the decade. Reflectivity meters, including the Brewster-angle refractometer, compensate for their lower precision than standard refractometers by measuring a wider range of refractive indices (up to 3.3: Read, 1990b; Kammerling et al., 1995a). Dr. W. Hanneman produced many small gemological instruments, including an inexpensive replacement for a quartz wedge, which could be used with a microscope to determine the optic sign of a gem (Koivula et al., 1992b). Because darkfield illumination is generally needed to see small inclusions well, a darkfield loupe was developed by GIA GEM Instruments (Koivula and Kammerling, 1991c; figure 9).

Other new gemological equipment included two types of immersion cells to distinguish diffusion-treated corundum, one illuminated (Koivula et al., 1992a; Linton et al., 1994) and one for use with a penlight (Read, 1993). A modified “phosphoroscope” for short-wave UV testing was described by Elen and Fritsch (1999, pp. 36–37). A strong magnet on a convenient mounting could be used to distinguish (most) unmounted synthetic diamonds that

contain metallic inclusions from natural diamonds (Kammerling et al., 1995d; Hanneman, 1996; Hodgkinson, 1996). H. Linton and colleagues on the Gemmological Association of Australia Instrument Evaluation Committee reviewed many new gemological instruments in the *Australian Gemmologist*, including: the Bailey light source, an inexpensive, low-powered replacement for sodium D-line illumination for a refractometer (Linton et al., 1996); and the Meiji Technico GF-252 combination refractometer/polariscope (Linton et al., 1997).

**Growth Structure Analysis.** Although morphological crystallography—the study of a material by identifying its crystal faces and their relationships—predates the discovery of X-rays, during the 1990s a variation of this approach was developed to gain information about the internal structure of gem materials. Faces (and corresponding growth zones) are most easily identified by their shapes, but these shapes can change with relative size; a better technique is to use the angles between adjacent growth zones, which do not vary. Kiefert and Schmetzer (1991a,b,c) used a horizontal immersion microscope to measure these angles, and showed valuable applications (e.g., distinguishing Nigerian and Colombian emeralds from synthetic emeralds). Perhaps the simplest example of this technique is the identification of synthetic diamonds on the basis of cubic growth zones (not present in natural diamonds, which grow as octahedra; Welbourn et al., 1996 [figure 10]). Growth features also are useful to understand the growth histories of some rubies (see, e.g., Peretti et al., 1995; Smith, 1996) and blue sapphires (Schwarz et al., 1996).

**Color Measurement.** Color comparison requires color standards, a choice of illumination (see, e.g., Yonick, 1999), a consistent (nondistracting) background, and trained color graders with proven excellent color perception (Brown, 1993; King et al., 1994). During the 1990s, machines were devised to measure color, especially for D-to-Z diamonds (e.g., Gran and Austron colorimeters; Shor, 1999). In a new application of old technology, existing measurement equipment, such as multichannel spectroscopy, was used to “grade” colored diamonds; however, these devices were of limited effectiveness due to differences in viewing geometry (compared to visual grading), the effect of gemstone fluorescence, and some reproducibility concerns (Peretti, 1995). Color can be communicated via computer by, for instance, collecting gemstone image spectra (Wagner, 1999); however, all such methods are accurate only if the sending and receiv-

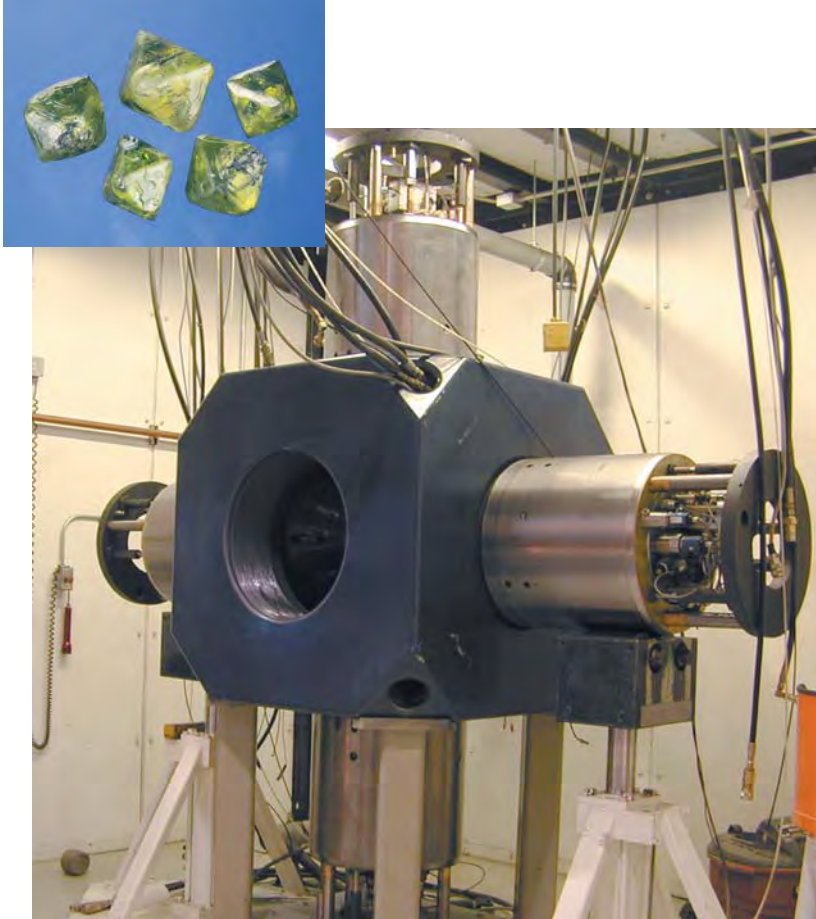


Figure 8. With presses such as this one, diamonds are exposed to high pressure and high temperature for short periods of time to give them a more attractive color. These five greenish yellow HPHT-treated diamond crystals (1.90–4.45 ct) were originally brown. Instrument photo courtesy of Novatek; inset by E. Schrader.

ing devices are properly calibrated and can detect and show the correct colors (which depends on the color “gamut” of each piece of equipment).

**“Black Boxes.”** A “black box” in this context is a relatively small piece of equipment that gives a choice of simple responses; it is not necessary to understand the technology to use such a device, although understanding prevents many interpretation errors (see, e.g., Liddicoat, 1996). The most common “black boxes” are diamond testers, to distinguish diamonds from (most) simulants. A new generation of these were introduced, from hardness pencils to UV-transparency meters, with the commercial development of synthetic moissanite as a diamond simulant (see, e.g., Nassau et al., 1997; Hammer and Stefan, 1999; Hanneman, 1999), because synthetic moissanite reads as diamond on most traditional diamond testers. The De Beers DiamondSure distinguishes synthetic diamonds



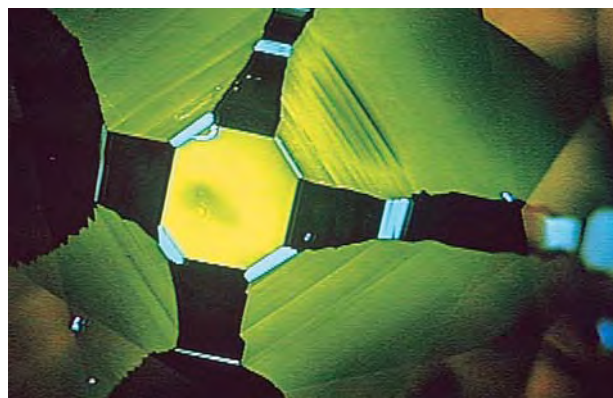


Figure 9. The darkfield loupe, shown here with a flashlight and tweezers, was developed by GIA GEM Instruments to help gemologists see small inclusions.

from most natural diamonds on the basis of the presence (in most natural diamonds) of the 415 nm optical absorption line (which is absent in synthetic diamonds), as described by Welbourn et al. (1996; see figure 11). Although this device was not commercially available at the end of the decade, it was in use in some major gemological laboratories.

Another important set of “black boxes” consists of measuring devices. Like a Leveridge gauge, the Presidium Electronic Gemstone Gauge measures the external dimensions of a gemstone (Linton and Brown, 1990). The Presidium DiaMeter-System Berger (again, see Linton and Brown, 1990), Sarin BrilliantEye (Lawrence, 1998), and OGI Megascop ( “New machine . . . ,” 1998) measure the propor-

Figure 10. The yellow-fluorescing octagon in the middle of the table of this 2.19 ct synthetic diamond represents growth on a cubic face. Cubic growth zones are not seen in natural diamonds. Photo courtesy of De Beers DTC Research Centre.



tions of fashioned stones, mainly diamonds, and provide “cut grades” according to predetermined criteria (often a choice of proportion ranges for “Ideal” cuts). According to its manufacturers, the GemEx BrillianceScope Analyzer quantifies “dispersion,” “light return,” and “brightness” (Schoeckert and Wagner, 1999), although no reviews of this equipment were available by the decade’s end.

## INNOVATIONS IN ADVANCED ANALYTICAL TECHNIQUES

Of increasing importance in gemology is the need to have recourse to techniques that require more sophisticated (and more expensive) instrumentation. Commonly referred to as “advanced testing,” these techniques were developed for—and used routinely in—other sciences, especially chemistry, physics, and geology (see, e.g., Hawthorne, 1993). They usually require considerable training both to use the instruments and to interpret the results. Applications of some specific techniques to gemology are described below.

**Improvements and New Vistas for Established Techniques.** Although techniques such as infrared and Raman spectroscopy were developed long before the 1990s, they became far more important to gemological laboratories during this decade. Such techniques were also modified or adapted to answer new challenges presented by treatments and synthetics.

*Ultraviolet-Visible (UV-Vis) Spectroscopy.* This was a mature technology, with a few new developments in the 1990s. Diamond spectra are generally run at low temperatures to improve resolution, but Lifante et al. (1990) noted that by taking mathematical derivatives of room-temperature spectra—that is, by examining the changes of slope in these spectra—better resolution could also be achieved. Diffuse UV-Vis reflectance spectroscopy can be used in combination with infrared techniques to identify gems, and a database of these spectra has been produced (Tretyakova et al., 1997, 1999).

*Infrared Spectroscopy.* During the 1990s, IR spectroscopy found some important new applications. This is the best technique to determine whether or not jadeite has been bleached and polymer-impregnated (Fritsch et al., 1992; figure 12), and IR analysis of jadeite has now become standard practice in some gemological laboratories. Smith (1995) showed that the IR spectra of heat-treated Mong Hsu

rubies were different from those of unheated rubies. Emeralds fall into five different groups based on IR spectra in the hydroxyl and water regions, although water-rich samples require emerald powder to be mixed into KBr pellets, which is somewhat destructive (Schmetzer and Kiefert, 1990). Most manufactured glasses can be distinguished from obsidian and other natural glasses based on their IR spectra (Owens, 1999). Yan et al. (1995) described an infrared microscope that works in much the same way as a Raman microspectrometer: Visible-light optics are used to focus on the region of interest, and an IR spectrum is then collected.

Diamonds are usually divided into four types (Ia, Ib, IIa, and IIb) based on the amounts of trace nitrogen and boron present, and the aggregation state of the nitrogen, as evident in their IR spectra (Fritsch and Scarratt, 1992; Weldon, 1999). Knowledge of diamond type can help separate natural from synthetic diamonds (see, e.g., Shigley et al., 1995); also, different diamond types react differently to HPHT processing.

Although transmission geometry (i.e., the beam is passed through the sample) is used in most gemological applications, IR spectroscopy is also useful in reflected beam mode. With the latter, the beam may be bounced off the sample's surface or focused within the sample (e.g., Johnson et al., 1999c). Although this technique is sometimes called DRIFT (diffuse reflected infrared Fourier transform) spectroscopy, for gemological purposes the beam generally is transmitted through the sample and then reflected off a mirror, rather than being reflected off the sample surface. Reflectance IR spectroscopy has been used to detect fillers in emerald (see, e.g., Zecchini and Maitrallet, 1998; Chalain et al., 1998) and jadeite (Quek and Tan, 1997), as well as to distinguish synthetic from natural emeralds (Johnson and Koivula, 1996a). Tretyakova et al. (1997) noted that three reflected-radiation techniques (reflected IR, reflected UV-visible spectroscopy, and reflected Raman) have their individual limitations, but can be used together to derive more complete results.

*Raman Spectroscopy.* Perhaps the single most important "new" instrument in gemological research in the last decade was the laser Raman microspectrometer (again, see figure 1). This sensitive luminescence technology found widespread use in the nondestructive identification of inclusions (and fillers) in various gem materials, even under the gem's surface, as well as of the gem materials themselves (see, e.g., Koivula et al., 1993b; Hänni et



Figure 11. Based on the absence or presence of the 415 nm absorption line, De Beers's DiamondSure refers synthetic diamonds and some natural diamonds for further testing while "passing" most natural diamonds. The fiber-optic probe is mounted vertically (as here) for testing loose stones, and can be removed for testing mounted stones. Photo by M. J. Crowder.

al., 1997; Johnson and Koivula, 1997c). Because Raman spectra can be gathered on mounted gems, this is a convenient technique for examining artifacts (see, e.g., Hänni et al., 1998). Toward the end of the decade, it promised to play a key role in the identification of HPHT treatment in diamonds.

Figure 12. IR spectroscopy has now become standard practice in some gemological laboratories for determining whether jadeite has been bleached and polymer-impregnated. The strong absorption in the 2900  $\text{cm}^{-1}$  region of the mid-infrared spectrum is particularly useful for detecting treated jadeite.

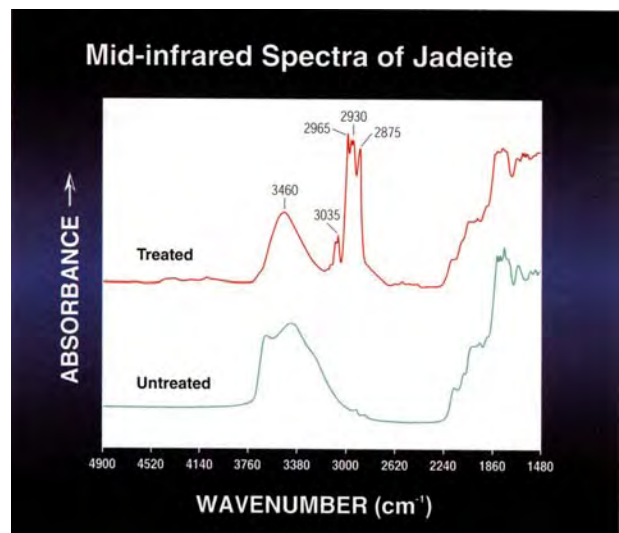




Figure 13. The De Beers DiamondView images a diamond's fluorescence to radiation below 230 nm in wavelength. The blue fluorescence of the natural diamond shown on the monitor is typical of most natural diamonds. Photo courtesy of De Beers DTC Research Centre.

This technique provides a "fingerprint" of the material tested: Raman spectral peaks of crystalline substances are sharp and occur at fixed energies; less-organized materials such as opal, resins, and oils have broad but often distinctive spectral features. Today, most major gemological laboratories have a Raman microprobe (microspectrophotometer).

Spectral libraries were needed to apply Raman analysis easily to gemology; one of the first in this field was produced by Pinet et al. (Schubnel, 1992), while others are available from manufacturers (e.g., Renishaw) and on the Internet (e.g., <http://minerals.gps.caltech.edu/files/raman>). Given a reliable spectral library, a Raman spectrum can provide within minutes an identification that otherwise might require several hours for both chemical analysis and X-ray diffraction.

Some gem materials luminesce to the laser excitation, and this photoluminescence can swamp the Raman signal (Kammerling et al., 1995f), or itself be captured and evaluated. Luminescence spectra have been collected at room temperature (Chalain et al., 1999) and low temperatures (Fisher and Spits, 2000; Smith et al., 2000; again, see figure 1) from diamonds suspected of HPHT treatment, in the hope of developing robust identification criteria. At the turn of the millennium, this is an active area of significant research.

*Luminescence Spectrometry and Imaging.* This topic includes luminescence to UV radiation (fluorescence) and to visible light (photoluminescence;

see discussion of "Raman spectroscopy," above), as well as time-delayed emission of light (phosphorescence). A prototype "microspectrofluorimeter" was developed to collect emission spectra from small spots on samples (Dubois-Fournier et al., 1989); it is also possible to see photoluminescence by placing a sample between two filters that together block out all light (the "crossed-filter" technique: Hoover and Theisen, 1993). The De Beers DiamondView (Welbourn et al., 1996) images samples using their fluorescence to radiation below 230 nm in wavelength (i.e., at higher energy than short-wave UV). At these energies, all diamonds fluoresce, and synthetic diamonds show different patterns of growth than natural diamonds (figure 13).

Many studies in the '90s used cathodoluminescence, collecting images and spectra from samples that glow on exposure to an electron beam (see, e.g., Ponahlo, 1989; Johnson and Koivula, 1998a). Sunagawa et al. (1998) used cathodoluminescence images to confirm that two faceted diamonds came from the same piece of rough. Pulsed cathodoluminescence has yielded new spectral features in corundum and spinel (Solomonov et al., 1994).

*Electron Microprobe Analysis and Scanning Electron Microscopy.* Again, these technologies should be considered mature (even in gemology), and were routinely used for chemical analysis and imaging of gem materials during the decade (see, e.g., Raber, 1996). Two types of detection systems are employed for chemical analysis: (1) energy-dispersive spectroscopy, which is quicker; and (2) wavelength-dispersive spectroscopy, which is much more accurate (Nikischer, 1999). "Windowless" detectors and layered crystal detectors (both of which became more common in the 1990s) have made it possible to measure the light elements boron, carbon, oxygen, nitrogen, and fluorine (see, e.g., Hänni et al., 1994; Johnson and McClure, 2000). SEM detectors became so efficient that it was possible to show the opal structure in Kyocera impregnated synthetic opals without first carbon- or gold-coating the samples (Kammerling et al., 1995b).

*X-ray Fluorescence Spectroscopy and X-ray Diffraction.* XRF was applied routinely to many identification problems in this decade. This technique is used to get an approximate bulk chemistry of various materials, including colored stones, diamonds (e.g., to detect fracture filling), and pearls (to distinguish freshwater from saltwater pearls and to detect some treat-

ments). There have been a few new applications in this decade. For instance, since the penetration depth of the X-ray beam can be less than the sample thickness, XRF has been used to demonstrate diffusion treatment in a star sapphire (Johnson and Koivula, 1996b). Energy-dispersive (EDXRF) systems with element-mapping capability now permit researchers to observe the distribution and concentration of chemical components within a sample (e.g., the Kevex Omicron: Sam Muhlmeister, pers. comm., 2000).

X-ray diffraction is another "mature" field, with few relevant innovations to applied gemology (versus, e.g., characterization of new gem species) in the last decade. One notable development is the availability of diffraction reference spectra on CD-ROM, with computer search capability (from the International Center for Diffraction Data, Newtown Square, PA).

*Metals Testing.* In one way, metals are easier to test than gems, as the use of destructive testing is usually less of a concern. Four metal-testing techniques—density, chemical reactivity, capacitance decay, and chemistry by EDXRF—were reviewed by Mercer (1992) and found to be insufficiently accurate to meet U.S. legal standards. However, five years later, nondestructive "X-ray assay" was considered reliable enough to substitute for fire assays for testing metals (Reilley, 1997). LA-ICP-MS (laser ablation–inductively coupled plasma–mass spectrometry; see below) is more precise, allowing gold samples to be tracked from specific mines by their trace-element contents ("Fingerprinting'...", 1995; see also Guerra et al., 1999). Particle accelerator–based techniques such as PIXE (see, e.g., Demortier, 1989) are even more precise, but access to such machines is limited, and they have been used mainly on archeological artifacts (e.g., Calligaro et al., 1998; Demortier et al., 1999).

**Promising Newcomers: Academic Techniques Being Adapted to Gemology.** *Isotopic Studies and ICP-MS.* These tests are innately destructive, on one scale or another: Conventional isotope studies require that the elements being studied be ionized as a liquid or gas, while microbeam techniques use a laser or ion beam to blast ionized particles from small (i.e., tens of microns in diameter) spots in the sample (see, e.g., Günther and Kane, 1999). The resulting trace-element and isotopic data can yield extensive information about gems, such as clues to formation ages and parent material. Ion probes and LA-ICP-MS can measure light elements such as hydrogen, lithium, and beryllium in gems ("The geosciences in review,"

1996), providing a practical micro-equivalent to wet chemistry as well as isotope chemistry. However, the (micro) destructive nature of these techniques, as well as their limited availability and expense, will likely restrict their gemological applications in the near future. In addition, as the spot size (and resulting crater) gets smaller, reproducibility may suffer.

There have been several isotopic studies of emeralds, especially by Giuliani and co-workers (see, e.g., Cheilletz et al., 1994; Giuliani et al., 1997, 1998, 2000). The oxygen isotopes of Colombian, Afghan, Brazilian, Zambian, Tanzanian, and Nigerian emeralds may be related to their source rocks and temperatures of formation (Johnson and Koivula, 1997a; Giuliani et al., 1998), which may provide clues to the locality of origin.

Conventional ICP-MS has been used to distinguish elephant from mammoth ivory, based on strontium-to-calcium ratios (Sato et al., 1991). Together with Fourier transform infrared (FTIR) spectroscopy and optical techniques, Pulz et al. (1998) used ICP-MS to distinguish Campos Verdes (Goiás, Brazil) emeralds from those from Colombia, Swat (Pakistan), Itabira (Minas Gerais, Brazil), and Franqueira (Spain).

*X-ray Imaging Techniques.* X-ray topography—creating images from diffracted X-rays—was described by Sunagawa et al. (1998), who used it (with cathodoluminescence) to determine that two fashioned diamonds had been cut from the same piece of rough. Synchrotron Laue patterns of rough Argyle diamonds have shown that they are more likely than diamonds from other sources to contain slightly misoriented crystallites; because of this mosaicity, Argyle diamonds have a high wear resistance for industrial uses, but are harder to polish as gems (Clackson and Moore, 1992). Coatings, as well as mosaic crystals, can be imaged using X-ray diffraction tomography (Liangguang et al., 1999).

*Other Physical and Chemical Techniques.* Various other instrumental methods have been applied to the analysis of gem materials through cooperative programs at universities and other institutions, which make otherwise prohibitively costly, experimental, and/or restricted equipment available to researchers. Using computer-aided tomography (CAT scanning), a pearl was shown to be attached to the shell in which it had grown (Wentzell, 1995). Scanning tunneling microscopy (STM) is one of a family of related micro-surface techniques that have many applications in computer-chip testing but are

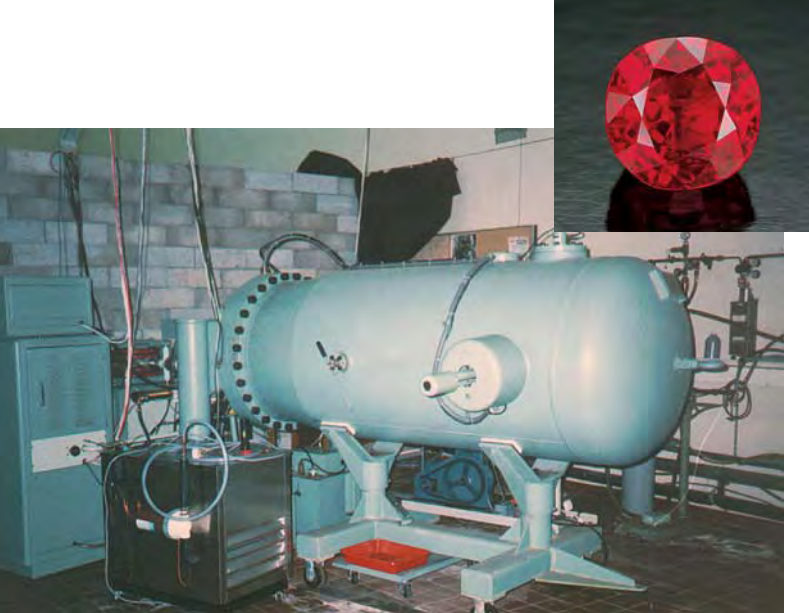


Figure 14. Proton-induced X-ray emission (PIXE) analysis has been used to nondestructively measure the trace-element concentrations in gems and metals. The Van de Graaff accelerator shown here is one method used to generate the proton beam for this technique; photo courtesy of Tay Thye Sun. Several PIXE studies have been performed on rubies (see inset of Burmese stone; courtesy of Amba Gem Corp., photo © Tino Hammid).

only beginning to be applied to the study of gems. STM revealed that diamond polishing proceeds by chipping in the hardest directions, and by plastic deformation and subsequent graphitization in other directions (Van Enckevort et al., 1993; Van Bouwelen and Van Enckevort, 1999). PIXE analysis (figure 14), an ion-beam technique, has been used for the nondestructive measurement of trace-element concentrations in gems and metals in Egyptian jewelry (Koivula et al., 1993b; Querré et al., 1996), as well as in rubies from Myanmar and Thailand (see, e.g., Kammerling et al., 1995e; Sanchez et al., 1997), and diamond inclusions (Ryan and Griffin, 1993). X-ray photoelectron spectroscopy (XPS), an analytical technique for surfaces, can determine elemental compositions and oxidation states, and gives similar results to FTIR in testing for surface waxing or polymer impregnation in jadeite (Tan et al., 1995).

Nuclear magnetic resonance (NMR), a nondestructive technique that probes the local environment of specific elements such as hydrogen and fluorine, can separate some natural and synthetic emeralds, and may be able to detect heat treatment in aquamarines (again, see Koivula et al., 1993b); it also shows promise for being able to separate synthetic from natural pink-to-orange sapphires (Troup et al., 1992). Electron paramagnetic resonance (EPR; also called electron spin resonance, or ESR—see,

e.g., Hutton and Troup, 1994b) has been used to investigate the valence state and bonding properties of certain transition elements in gems, including: manganese in pink tourmaline (Petrov, 1990), vanadium in emeralds and green beryls (Hutton and Troup, 1994a), chromium in natural and synthetic alexandrites (Rager et al., 1998), and nickel in annealed synthetic diamonds (Mashkovtsev et al., 1999). Laser tomography has been used to image small defects in heat-treated sapphires (Shida, 1990) and synthetic rubies (Koivula et al., 1993b). At this time, these techniques are more academic than practical, but each of them could make useful gemological determinations.

*Statistical Analysis.* The estimation of errors is important in the assessment of the validity of any experimental study, regardless of the technique used. Recently, however, “demographic” studies have been introduced, which use (often simplified) statistical techniques to study the properties of data populations, rather than individual samples or observations. For example, the diamond fluorescence research of Moses et al. (1997) was mainly a statistical study of human perception of diamond color and transparency as a function of intensity of blue fluorescence. Although individual responses were quite variable, and sometimes even contradictory, overall trends in appearance aspects could be discerned. This study was unusual for gemology in that it considered general population trends, instead of individual responses and observations of individual items.

#### SUMMARY: INNOVATIVE TRENDS IN THE 1990s AND SOME PREDICTIONS

In their 1980s review, Fritsch and Rossman (1990) suggested the following as significant remaining challenges for the gem trade: determining natural versus treated colors in natural gems (especially green diamonds, blue topaz, and red tourmaline), instrumentational determination of heat treatment (especially corundum) and dyes (e.g., “lavender” jade), and the reproducible measurement of a gem’s color. Progress has been made on some of these issues in the last decade—notably on determining the origin of color of green diamonds, perhaps the most economically important of these issues—but no definitive methods or solutions have yet been published, and newer crises have relegated the rest of these issues to a much lower priority.

This last decade was one in which technology brought changes to nearly every aspect of gemology

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and gem manufacturing. To this author, the following trends best summarize the current need for, and spirit of, innovation:

- *The Internet is for everybody.* Widespread availability of information (of all levels of quality) means that sophisticated consumers may know more—or think they know more—about gems than their retailers do.
- *Computers calculate proportions and cut designs.* To the degree that a gemstone's optical performance can be modeled accurately, cuts can be planned and optimized without wasteful trials. Verification is still required, however.
- *Robots fashion gems.* Used at first for calibrated goods, robotic cutting creates relatively uniform results.
- *Lighting concerns are increasingly important in the retail store and the laboratory.* The optimal lighting environments for viewing gems for purchase as well as gemological evaluation will likely be a major research focus over the next decade.
- *The use of high temperatures and synthesis methods for treatment has begun to blur the line between treated gems and synthetics.* The classic 1990s examples were the (very) high temperature heat treatment of Mong Hsu rubies, which grows new material, and the use of high pressure/high temperature both to produce synthetic diamonds and to decolorize or otherwise improve the color of natural diamonds.
- *However, treated synthetics are now considered a category in their own right.* The GIA Gem Trade Laboratory, for example, currently identifies diffusion-treated synthetic corundum and polymer-impregnated synthetic opal.
- *"Black boxes" provide specific solutions to certain gemological problems.* In some cases, as with synthetic moissanite testers (used to distinguish diamond from a doubly refractive substitute), these devices may substitute for a lack of basic gemological skills, or the time needed to practice them.

- *More treatments and synthetics require advanced testing.* For example, a piece of jadeite may show no obvious signs of polymer impregnation when viewed with a microscope, but this treatment may be readily apparent when the sample is examined with FTIR.
- *Statistical studies and computer databases may lead to "profiling" of gem materials by expert systems.* We have now reached the point where computers are better able than individuals to keep and compare relevant observations; in the future, these systems should be able to spot goods representing new mines, synthetics, and treatments.
- *New and old techniques from other sciences continue to be adapted to solve gemological problems.* Mineralogy, chemistry, and physics have been fruitful sources of advanced techniques with gemological applications, but other sciences (such as biology) may provide more techniques in the future.

It is difficult to know which of the many technologies reviewed in this article will make the greatest impact in the future. Most of the technical innovations that affect the trade today have their roots in the technologies of previous decades. The most significant development has been in the rate at which changes reach us. We have less time to react than we did in the past: Crises come on top of crises, challenges on every scale hit at once. Continuous education is therefore a key to success.

Although it is true that gem identification is becoming increasingly difficult (a trend that will certainly persist), often requiring sophisticated laboratory instrumentation and techniques, it is also true that the competent gemologist can still ascertain a tremendous amount of information using classical gem-testing methods that are routinely available. By staying on top of the gemological literature, and applying this newly gained knowledge to classical gem-testing methods, competent gemologists can accurately identify many gem materials and, as importantly, determine when they need to submit a gem to an independent, recognized laboratory.

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Key to abbreviations: *Australian Gemmologist* = AusG; *Diamond and Related Materials* = DRM; *Diamond International* = DI; *Gems & Gemology* = G&G (Gem News = GN, Gem Trade Lab Notes = GTLN); *Industrial Diamond Review* = IDR; *Journal of Crystal Growth* = JCG; *Journal of Gemmology* = JoG; *Journal of the Gemmological Society of Japan* = JGSJ; *Nuclear Instruments and Methods in Physics Research B* = NIMB; *Revue de Gemmologie* = Rev. de Gem.; (*Gemmologie*:) *Zeitschrift der Deutschen Gemmologischen Gesellschaft* = (Gem.) Z. Dt. Gemmol. Ges.

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