ANALYTICAL TECHNIQUES IN GEMOLOGY: A HISTORICAL OVERVIEW

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Gemology has become an increasingly technical field, driven by the need for more advanced analytical methods and instruments to test gem materials (figure 1). This shift reflects the evolving challenges of gem identification, a trend that has been well documented in major gemological journals. This special issue of *Gems & Gemology* will survey the testing instrumentation currently employed by GIA's laboratories, reviewing their applications, limitations, and the vital information each technology provides. Important aspects of their use for gem testing will be discussed. Note that equipment developed specifically for the GIA diamond quality grading system or other laboratory activities will not be included. This article opens the Winter 2024 edition by briefly examining the introduction and role of scientific instrumentation in gemology to address identification challenges in the marketplace.

HISTORICAL BACKGROUND

The use of scientific instruments for testing gems at GIA began a few years after its founding in 1931 (figure 2), as reported in early editions of this journal. Prior to that, there were few gem testing instruments designed specifically for jewelers. In the early 1900s, G.F. Herbert Smith at the British Museum in London championed the optical refractometer for gem testing (Herbert Smith, 1907). He and his contemporaries discussed additional practical tests such as dichroism, absorption spectra, density, and hardness. GIA founder Robert M. Shipley sought to expand the de-

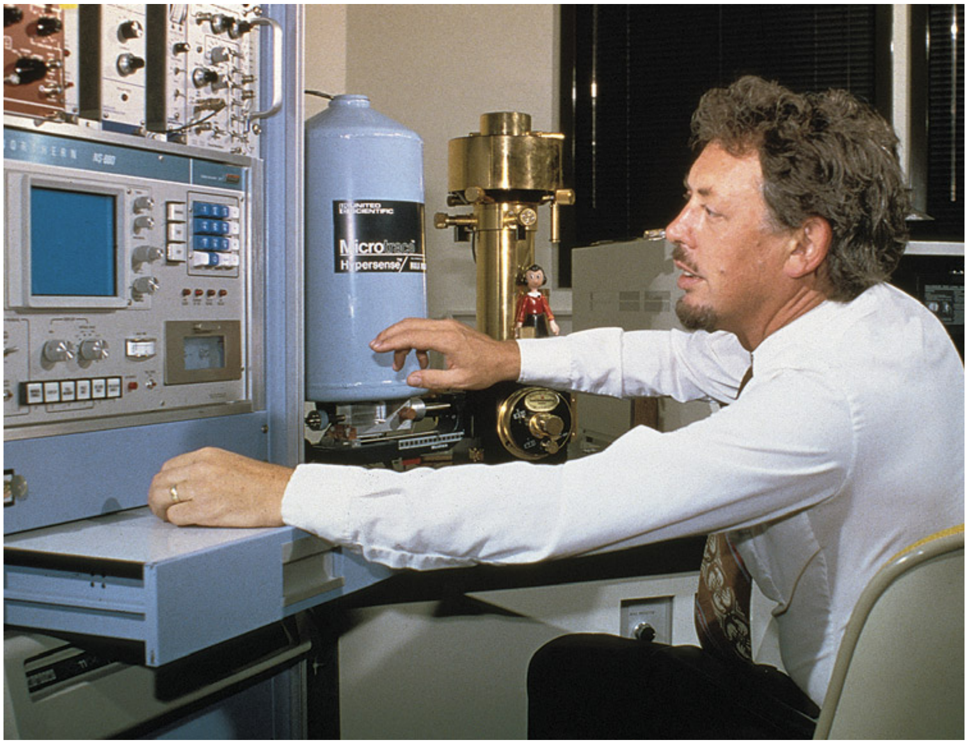


Figure 1. Dr. Vince Manson using GIA's first scanning electron microscope, acquired in 1976.

Figure 2. Robert Shipley Jr. working with gem testing equipment at GIA's early headquarters in Los Angeles. This 1937 photo is from a three-minute color film by Paramount Pictures for its "Popular Science" series, produced between 1935 and 1949 and shown in movie theaters nationwide.

velopment of testing instruments tailored to meet the needs of jewelers and gemologists, while educating them on these tools (figure 3).

In the late 1930s, GIA began evaluating and teaching the proper use of devices such as the loupe, microscope, hand spectroscope, refractometer, and

Figure 3. Standard testing equipment produced or distributed by GIA during the 1980s and 1990s.

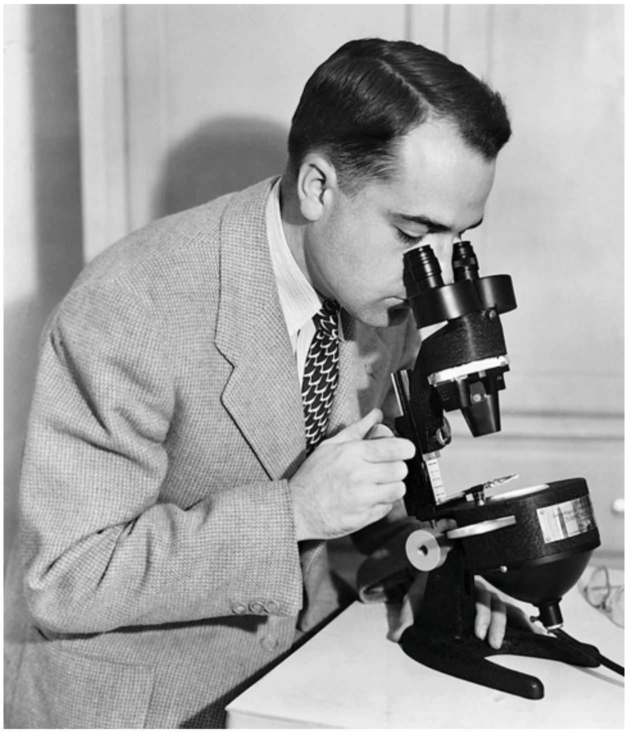


Figure 4. Richard Liddicoat using a stereomicroscope (the GIA Diamondscope) with darkfield illumination. Introduced in 1938, it provided improved viewing of inclusions and clarity features in gemstones.

dichroscope. These were produced by various manufacturers, including GIA. Some commercially available scientific instruments were useful for gem studies, but the special needs for holding, manipulating, and illuminating gems often required modifications (such as light source additions to the microscope). Some methods were adopted from the field of mineralogy. By the end of the decade, GIA was the exclusive U.S. distributor for several gemological instruments made abroad and had begun manufacturing its own specialized equipment, including a commercially available stereomicroscope fitted with darkfield illumination, introduced in 1938 (figure 4).

With the establishment of the GIA Gem Trade Laboratory in New York in the fall of 1949, gem testing for trade clients and the use of gemological instruments became more routine at GIA. This use increased with the introduction of the first GIA diamond grading reports in 1955. GIA Gem Instruments was established in 1966 to develop new equipment and refine existing tools. The acquisition of more advanced scientific instrumentation accelerated following the creation of GIA's research department in 1976. Investment in research staff and analytical instrumentation has continued to the present day, as evidenced by the articles in this special issue.

SCIENTIFIC INSTRUMENTS FOR GEM TESTING

Instrument development for gem testing often resulted from the need to address new identification challenges in the marketplace when existing instruments and methods were no longer sufficient. The introduction of a new piece of scientific equipment often inspired gem researchers to adapt it for their own needs. In recent decades, the increasing sophistication of gem synthesis and treatment techniques has accelerated the need for more advanced scientific instrumentation.

Several aspects of advanced instrumentation are worth mentioning. The testing procedure for gems submitted by laboratory clients must not result in visible damage or destruction to the samples, because these samples tend to be rare and highly valued by their owners. For testing, they must fit into and be held within the sample compartment of the instrument. Modification of the sample compartment may therefore be necessary. When recording spectra or conducting a chemical analysis, the testing location on the gem sample must be positioned so that it is accessible. For example, it may be more effective to bring the light signal directly to the gem using some type of flexible optical cable to capture a visible spectrum. In a high-volume production environment, both the analysis time and the ease of positioning and removing gem samples from an instrument are important factors.

The value of any data gathered from gems using scientific instruments becomes much more significant and useful when the results are stored in a searchable database and when they are collected on samples of known natural, synthetic, or treated origin. Many gems submitted to a laboratory for testing are of less certain origin, so the analytical data obtained from them can be problematic.

MICROSCOPY AND PHOTOMICROGRAPHY

Although glass lenses have been employed for magnification for many centuries, the use of what today would be called a microscope began in the mid-1600s. Scientists at the time were able to study materials at magnifications of up to about 300×. Over the following centuries, their application became extensive in many scientific fields (Kile, 2003). Microscopes using polarized light specifically designed for petrographic studies in geology became available in the 1870s. According to Gunter (2004), over the past

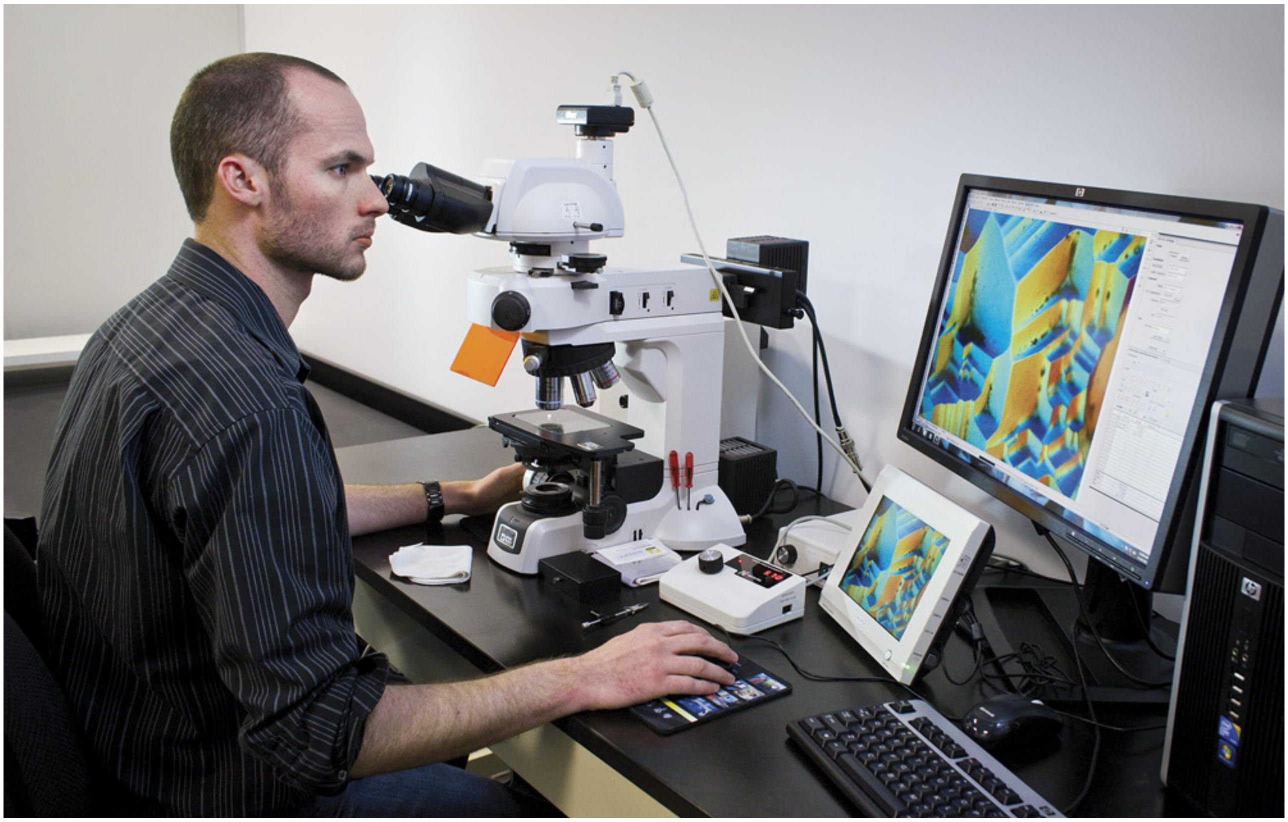


Figure 5. Nathan Renfro using the Nikon photomicroscope for observation and photography of inclusions and other micro-features at magnifications up to 1000×. Photo by Kevin Schumacher.

century these microscopes have "since contributed more to our knowledge of minerals and rocks than any other single instrument."

One of the first to describe gemstone inclusions observed with a microscope was the naturalist Isaac Lea, who published sketches of some of these micro-features (Lea, 1866, 1869, 1876, 1877). The geologist Henry Sorby pioneered the study of rocks and minerals using the microscope and also published works on mineral inclusions in gems (Sorby, 1869; Sorby and Butler, 1869).

The diagnostic value of inclusions in gem identification was first elucidated in the 1940s by the legendary gemologist Edward J. Gübelin (1945a,b,c, 1948; see also Kane et al., 2005). These micro-features could provide information on a gem's identity, quality grade, geologic and geographic origin, method of synthesis, and evidence of treatment. The three-volume *Photoatlas of Inclusions in Gemstones* (Gübelin and Koivula, 1986, 2005, 2008) remains the most significant reference work on mineral and fluid inclusions encountered in the major commercial

Figure 6. Photomicrography of inclusions: pyrochlore in a Cambodian sapphire (A), multiphase in a Colombian emerald (B), and sulfides in a Mozambique ruby (C). Photomicrographs by Nathan Renfro and Jonathan Muyal (C).

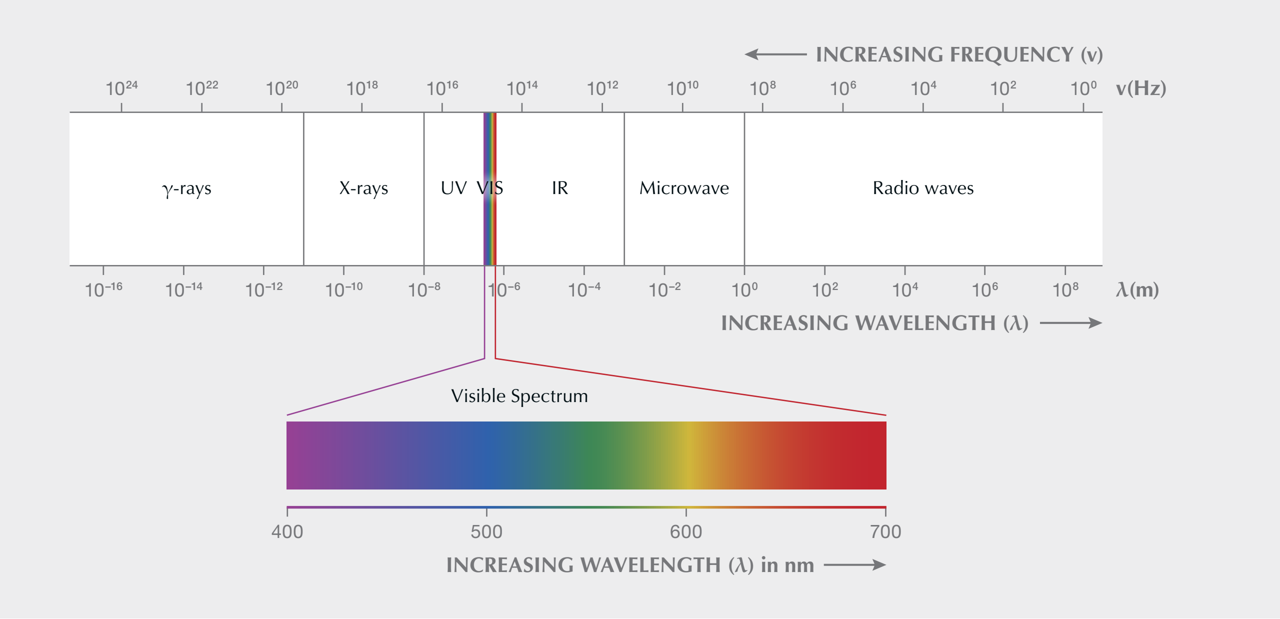


Figure 7. The electromagnetic spectrum encompasses the range of all possible frequencies of electromagnetic radiation. This form of energy travels through space in waves, which can be characterized by their wavelength and frequency. Note that as frequency increases (and wavelength decreases), energy also increases. Spectroscopic techniques are used to study gems in the ultraviolet, visible, and infrared regions of the spectrum.

gemstones. The past several decades have witnessed the introduction of polarizing and color filters, various kinds of illumination (brightfield, darkfield, reflected, diffused, indirect, and fiber-focused), and more recently digital photography and computer processing techniques to enhance photographic images (Koivula, 2003; Renfro, 2015a,b) (figures 5 and 6).

SPECTROSCOPY

Spectroscopy is the scientific study of how light interacts with materials (whether reflected, absorbed, or emitted). A spectrum is a graphical representation of that interaction as a function of light wavelength or frequency. The electromagnetic spectrum is the range of energy vibrations that correspond to wavelengths extending from many kilometers down to a fraction of the size of an atomic nucleus (figure 7). This wide range of frequency is divided into separate regions of interest.

Visible light is the limited portion of the electromagnetic spectrum that can be detected by the human eye. Each color of visible light corresponds to a particular range of wavelength and vibration frequency. When white light passes from the air into a triangular glass prism and then back into the air, the component colors propagate at different speeds. This causes the colored light rays to refract at different angles, dispersing into the familiar rainbow pattern.

Among the earliest uses of the spectroscope were observations of the sun's light emission spectrum and of the distinctive spectrum patterns produced when chemical elements were heated to incandescence by a flame. These breakthroughs demonstrated that visible spectra could be used to analyze the chemical composition of either nearby objects or very distant celestial bodies. When matter is heated to incandescence at high temperatures, it can emit a nearly continuous light spectrum. As this emitted light passes through a lower-temperature material, a pattern of narrow absorption and transmission bands appears. This pattern is based on the object's composition.

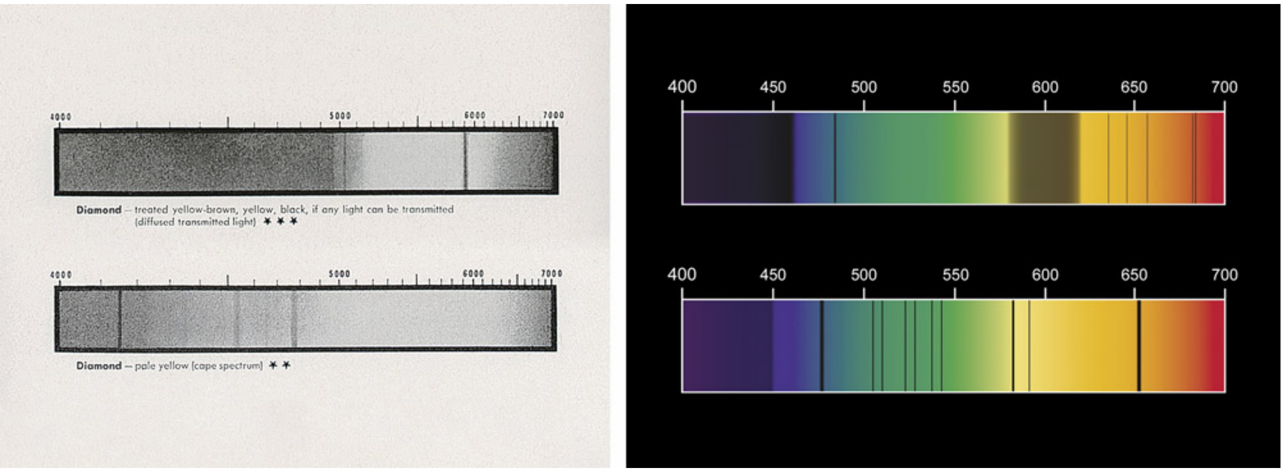
A visible transmission/absorption spectrum of a gemstone, as seen using a spectroscope (figure 8), reveals information on the causes of coloration. The hand spectroscope, championed by Anderson (1944a,b) and Crowningshield (1957; see also Moses et al., 2003), gives a visual representation of the wavelength regions of both selective light transmission and absorption by a gem. Each gem has its own characteristic pattern of narrow to wide, lighter to darker absorption bands, so viewing the spectrum pattern with a spectroscope can provide a quick means of identification for the gemologist (figure 9).

A spectrophotometer captures that same visible spectrum information but produces a graphical rep-

Figure 8. Robert Shipley Jr. using a spectroscope to examine the absorption spectrum of a gemstone at GIA for a 1937 Paramount Pictures film for its "Popular Science" series.

resentation with wavelength values along the plot's horizontal axis and the relative amount of light transmission or absorption along the vertical axis. The spectrophotometer offers several advantages over the spectroscope: a wider wavelength range of spectral features, the detection of very weak features not visible with the spectroscope, and the accurate wavelength locations of these features. Spectral features at wavelengths beyond the red end of the spectrum (the infrared) and those beyond the violet (the ultraviolet) are also of importance for gem characterization, and these can be captured with similar types of equipment. Although first developed several decades earlier, visible light spectrophotometers began to appear in gem testing laboratories in the mid- to late 1980s. This was followed by infrared and then other spectroscopy techniques (Nassau, 1981; Lind and Schmetzer, 1983; McMillan 1985, 1989; Fritsch and Stockton, 1987; McMillan and Hofmeister, 1988).

Figure 9. Left: Reproductions of Robert Crowningshield's hand-drawn spectrum patterns for colored diamonds, first published in the fifth edition of Richard Liddicoat's Handbook of Gem Identification *in 1957. Right: Artistic color renderings of the spectra for emerald (top) and zircon (bottom) currently used in GIA educational material.*



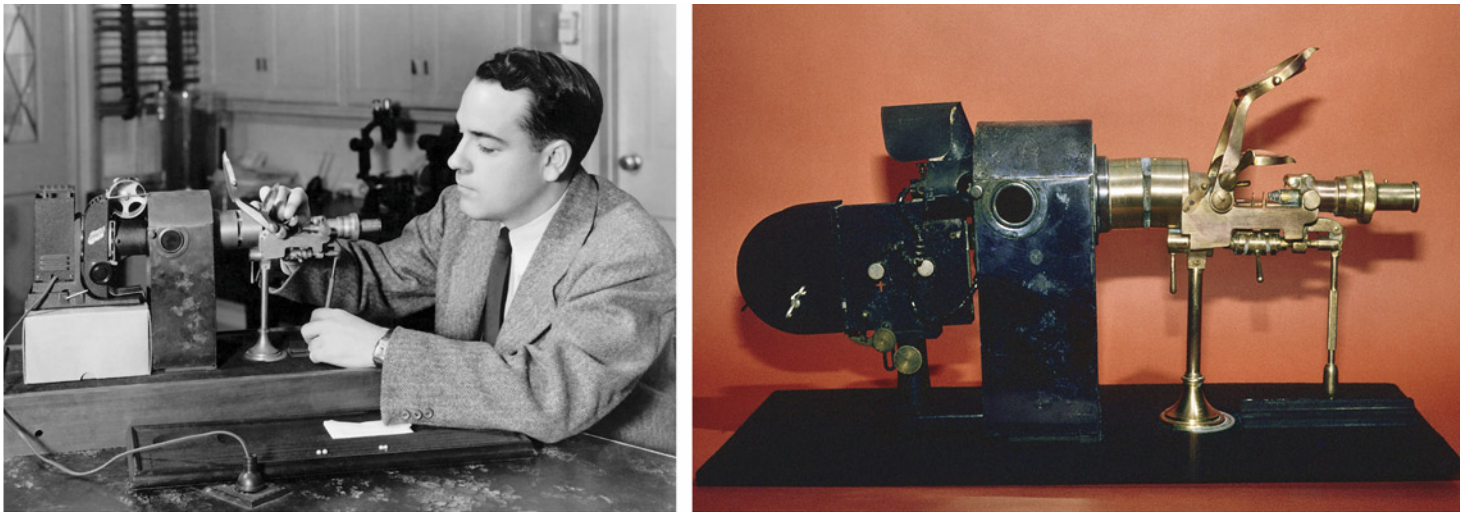


Figure 10. Left: Richard Liddicoat using an endoscope to examine the internal structure of a pearl. Right: A 1979 photo of the same instrument.

In addition to their diagnostic value, spectroscopic methods are also generally nondestructive (although some types of higher-energy ultraviolet illumination can affect a gem's color or other features). However, these methods do present some practical challenges. A gem's facet arrangement and cutting style often affect the amount of light transmission. Faceted gems are optically designed so that incident light enters through the crown facets, is internally reflected by the pavilion facets, and ultimately exits upward back through the crown. To record a visible transmission/absorption spectrum, however, some light signal must pass from the light source through the gem to the instrument detector. Certain facet arrangements provide little if any light transmission. A longer light path within the stone results in stronger selective light absorption and therefore a more intense face-up color.

Polished gems are not parallel-sided flat "windows" with a fixed and straight light path length that can be directly measured—they are multifaceted objects within which light is often internally reflected. As a result, the visible spectrum plot of most gemstones simply shows increasing absorption along the vertical axis. Individual girdle facets can sometimes be parallel to one another, providing a straight light path for spectra collection. The measured spectrum signal results from the combined effects of selective light absorption by optical defects along the light path. The signal is sampling along the light path but not the entire bulk volume of the gem, except when the stone is quite small. Spectroscopic analysis of gems can be quite rapid (requiring only a few seconds), but the methods are performed on one gem at a time. As a result, samples must be placed and removed from the instrument chamber by hand or by an automatic loader.

The spectroscopic techniques most widely used today in GIA laboratories are described in this special issue. For a review of the various forms of spectroscopy and their applications, see Agnello (2021).

IMAGING

Several imaging techniques are used in gem testing, the most important of which is X-ray radiography for distinguishing natural and cultured pearls. Following the discovery of X-rays in 1895, experiments revealed they could penetrate solid material, and by the 1920s these energetic rays were being used for a variety of medical applications. Different materials vary in their relative transparency to X-rays, and this variation is captured as a light/dark pattern on a photographic image.

The introduction of Japanese cultured pearls into the European market by K. Mikimoto in the early 1920s posed a widespread challenge for jewelers, who could not distinguish them from rare natural pearls by simple visual means (Boutan, 1921; Lemaire, 1921; Eunson, 1955; Nagai, 2013). To address this challenge, the first gem testing laboratories were set up in London, Paris, and other locations during the 1920s. Testing was conducted on a larger scale (but only one sample at a time) by viewing a pearl's internal structure using an endoscope or by viewing internal banding of the bead nucleus within the drill hole (Wright, 1923; Szilard, 1925a,b; Michel, 1926;

Figure 11. Robert Crowningshield preparing to X-ray several pearls at GIA's New York laboratory in the early 1950s.

Jardine, 1931; figure 10). This technique was followed in the late 1920s by X-ray radiography (Dubois, 1907; Anderson, 1932; Alexander, 1941a,b; Barnes, 1947a,b,c,d; Webster, 1957a,b; Ogden, 2012; Scarratt and Karampelas, 2020; Ericson, 2021).

The X-ray method was of particular value because a radiograph image could be created for multiple pearls (loose or strung) at the same time. Responding to the jewelry industry's needs, Dr. A.E. Alexander set up a pearl testing office in New York City, which he operated from 1940 until 1949, when it was turned over to GIA and became the GIA Gem Trade Laboratory (Alexander, 1940, 1941a,b; Benson, 1951) (figure 11). Evaluation of pearl radiographs remains the most important imaging technique at GIA.

CHEMICAL ANALYSIS

A gem mineral's composition reflects the chemical elements and conditions in its formation environment. Minerals such as quartz (SiO_2) have a very simple chemical composition. Others have a complex and variable composition. One such mineral is tourmaline, which is actually a group of more than 40 distinct but related mineral species. It has an idealized chemical formula of $XY_3Z_6(T_6O_{18})(BO_3)_3V_3W$, where B = boron, O = oxygen, and the other six letters represent atomic positions or sites in the crystal lattice where various elements can be accommodated. The many possible element substitutions in these sites result in the large number of mineral species within the tourmaline group.

For discussion purposes, the chemical elements in minerals can be categorized by concentration: major elements above about 1 weight percent (10000 parts per million, or ppm), which are essential since they determine the nature of the mineral; minor elements varying from 0.1 to 1 wt.% (1000 to 10000 ppm); and trace elements below approximately 0.1 wt.% (1000 ppm). The minor and trace elements tend to be more variable in concentration. All these elements substitute for one another in the mineral's crystal structure.

Before the 1960s, the chemical analysis of minerals was a complex, time-consuming process. This involved powdering the material and then dissolving it by chemical reaction in an acidic solution, followed by a combination of "wet chemical" methods to detect the major and minor elements present in the solution. Major and minor elements were often precipitated from the solution as oxides, which could then be dried and weighed. These components were reported as weight percent oxides, a convention still used today. The task required a skilled analytical chemist and a sizeable volume of the mineral to be analyzed.

Chemical analysis techniques vary in the sample size they require. Some methods sample the entire volume of the material, while others focus on small spots. Certain methods are better suited for detecting major and minor chemical elements, while others can detect trace elements at very low concentrations. These trace elements may be randomly distributed within the sample or concentrated along growth structures, so the analysis location is crucial for accurate results. Some analytical methods provide a *qualitative* chemical analysis (identifying which elements are present), while *quantitative* analysis measures the amount of each element. For quantitative analysis, a standard reference material must also be tested. This reference material must contain the elements of interest in known concentrations, which allows the element concentration in the gem sample to be calculated. The most effective reference materials are those that closely match the structural and compositional characteristics of the gem being tested. An example of such a "matrix-matched" material is synthetic corundum doped with elements of interest for the analysis of natural corundum.

Modern chemical analysis often involves focusing an electron beam (or some other type of energy) onto a small spot on the mineral's surface. This causes the elements to emit X-rays at characteristic frequencies, which can be measured by the instrument's detector. The electron microprobe, introduced in the early 1970s, was often used to obtain quantitative compositional data from rocks and minerals. However, the electron microprobe method required a polished surface for analysis. This surface had to be coated with a thin carbon film to conduct away any buildup of electrical charge, and the element measurement results had to be mathematically corrected to yield accurate composition data (Wilson, 1972; Reed and Ware, 1975; Dunn, 1977; Reed, 1989).

Two chemical analysis methods are used today by GIA. The first is energy-dispersive X-ray fluorescence, or EDXRF (Anzelmo et al., 2013). This provides qualitative analysis of the major and minor elements present in the material, although the detection limits vary by element. Elements with a lighter atomic weight than sodium cannot be detected using EDXRF. The only requirement is a polished surface representative of the material. The method produces a bulk analysis to depths of several millimeters below the sample surface.

The other important chemical analysis technique is laser ablation–inductively coupled plasma–mass spectrometry, or LA-ICP-MS (figure 12), which focuses a narrow beam of high-powered laser light on the gir-

Figure 12. GIA's laser ablation–inductively coupled plasma–mass spectrometry (LA-ICP-MS) system for chemical analysis of small spots on the surface of polished gemstones. Photo by Kevin Schumacher.

dle of the gem being tested. Pulsing of the laser energy physically ablates or removes submicroscopic particles to create a tiny pit on the surface that is approximately 50 µm across (which can be removed by minor repolishing). Because this is a point or spot analysis method, several locations typically along the girdle are selected for analysis to obtain a more accurate indication of the material's overall minor and trace element chemistry. The ablated particles are carried by a flowing inert gas into a very high-temperature plasma torch operating at temperatures of up to 10000 K (hotter than the surface of the sun). Passing through the torch, the particles are vaporized, ionized, and broken down into their constituent chemical elements, which can then be analyzed by the instrument's mass spectrometer. This provides a quantitative determination of the minor and trace elements present and their concentrations (Sun et al., 2019; Wang and Krzemnicki, 2021; Yu et al., 2022). LA-ICP-MS can quickly detect a wide range of elements down to a parts-per-billion level with no sample preparation.

LOOKING AHEAD

For gemological laboratories to maintain consumer confidence, identification and reporting on all types of gem materials must remain a central focus. As analytical techniques such as those discussed in this special issue continue to advance, new testing methods will emerge. The nature of gem identification challenges will also evolve, and the gems themselves will have varying degrees of detectability. Some future trends to watch for include:

1. Synthetic gem materials, some of which have been available for more than a century, will remain abundant, with continued improvements in growth methods. However, the development of entirely new synthetic gems seems unlikely. Almost all natural minerals that can be synthesized for gem use have already been produced, and the few that remain (e.g., topaz and tourmaline) are unlikely to be grown since there is no industrial demand for them beyond their use as gems.
2. Mining at most localities produces a range of qualities of gem materials that could find a place in the market if treated to improve their color, durability, or appearance. Gem treatments, which also have a long history, will likely proliferate and improve, with some processes creating less obvious appearance changes and fewer diagnostic features. Multistep treatments may be further developed. There is also a category of processes designed simply to remove evidence of prior treatment. Since most treatments begin with a natural gem material, they often provide fewer diagnostic clues than synthetic gems.
3. Surface coatings, one of the oldest treatment methods, could regain prominence with advances in coating materials. These developments include less noticeable visual effects, selective application to limited portions of the cut stone's surface, and colorless coating materials offering improved durability without being visible.
4. The emergence of new gem-like materials, such as faceted durable glasses or transparent polycrystalline ceramics, should pose limited testing challenges but may initially cause concern.
5. Country of origin reporting on colored stones will remain a focus for gem testing laboratories. The addition of new localities for high-quality colored gemstones, some occurring in geological settings similar to those of older localities, will require continued refinement of testing methods and determination criteria. Determining the origin of polished diamonds remains unlikely (Smith et al., 2022).

Modern gemological laboratories face significant challenges beyond the initial investment in scientific instrumentation. The equipment also requires trained operators, potential modifications for holding samples, costly maintenance contracts, and robust data collection protocols. Perhaps the greatest challenge is creating a searchable database for the vast amounts of data generated by this technology. The University of Arizona's RRUFF project provides a free database of high-quality spectra and other data on thousands of well-characterized minerals (https://rruff.info/; see also Lafuente et al., 2015; Culka and Jehlička, 2019). Similar online databases exist for other materials but often lack data on the gem samples tested (in particular, for synthetic and treated gem materials).

Gem characterization studies featuring the types of analytical data mentioned above are scattered in the gemological literature and vary in usefulness. Gemological laboratory staff often find themselves creating their own in-house databases using their own instrumentation and the best obtainable gem samples. To confront new identification challenges,

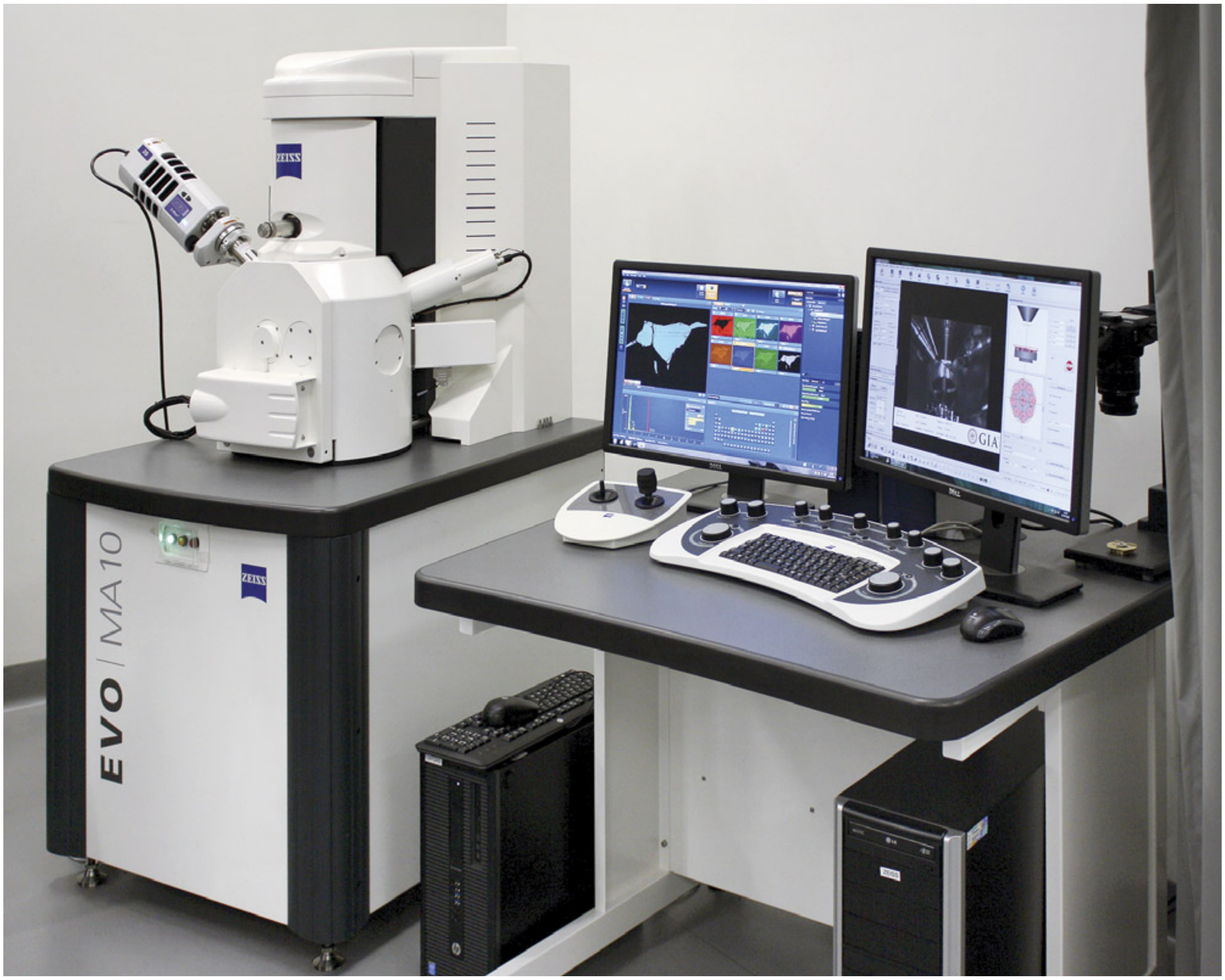


Figure 13. The Zeiss EVO 10 scanning electron microscope used today at GIA in New York. The instrument is capable of magnification up to 100,000×. Photo by Jian Xin (Jae) Liao.

laboratory staff continually search for new analytical techniques and instrumentation that can be adapted for their purposes.

This article began with a photo of the first scanning electron microscope acquired by GIA in 1976 at its former Santa Monica headquarters. Figure 13 shows the newer instrument acquired in 2015 for GIA's New York laboratory. These two photos illustrate the decades-long evolution in analytical instrumentation in major gemological laboratories. The current state of the art in analytical instruments and techniques is described in this special issue.

ABOUT THE AUTHOR

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