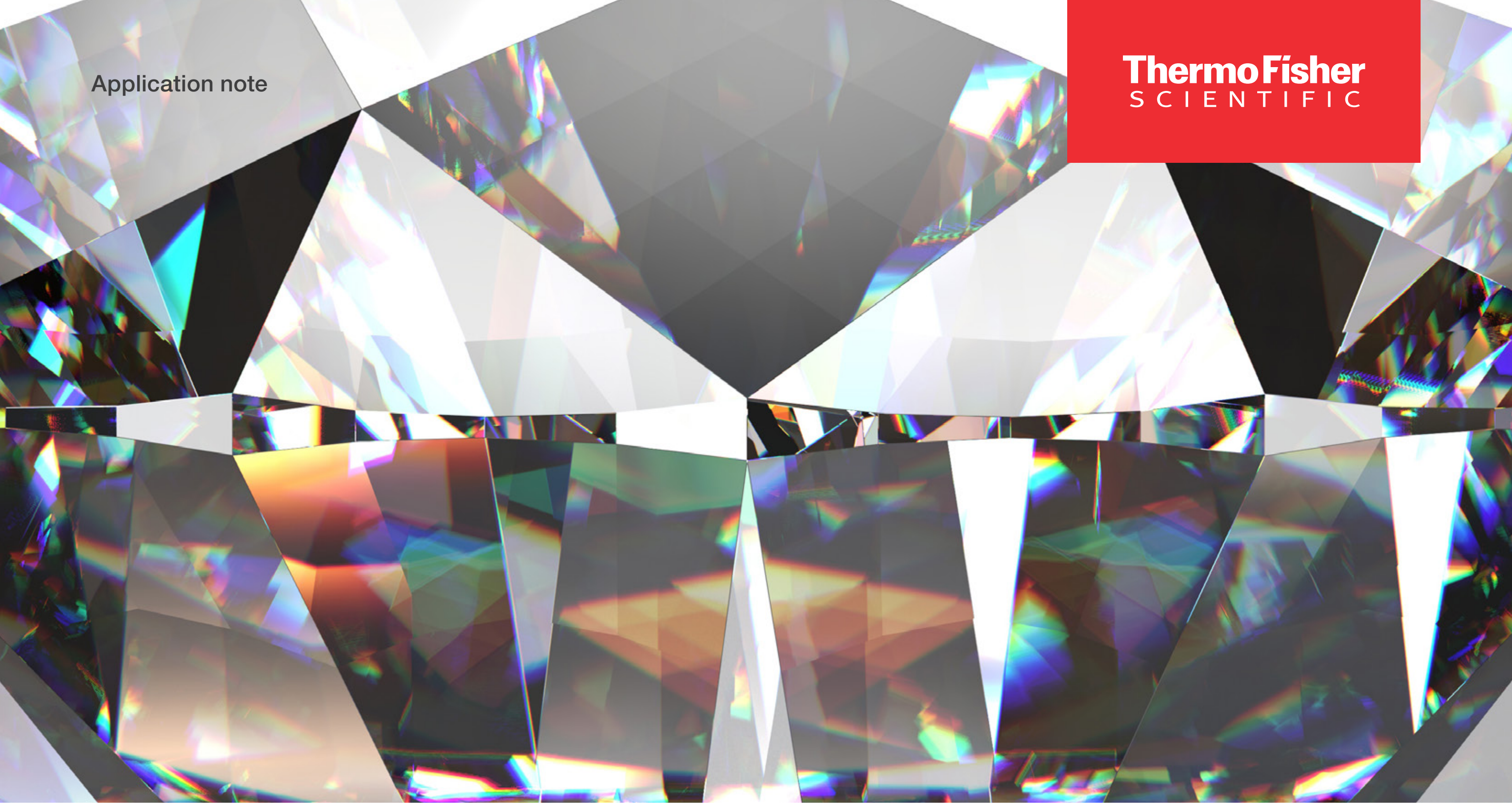

Analysis of diamonds by FTIR spectroscopy

Authors

Stephen Lowry, PhD
and Suja Sukumaran, PhD

Thermo Fisher Scientific,
Madison, WI, USA

Introduction

Diamonds are by far the most popular gemstone in America and many other parts of the world. Because of this popularity and the high price of quality gemstones, there is a large market for cheaper stones that resemble diamonds, as well as diamonds that have been treated to improve their appearance. Detecting these stones can be a significant problem for diamond buyers if they are represented as natural diamonds by unscrupulous sellers.

Fourier-transform infrared (FTIR) spectroscopy can be a useful tool for buyers and sellers to determine whether diamonds are natural. Diamonds are unique among gemstones because they are composed of a single element (carbon), while virtually all other gems contain multiple elements, including significant amounts of oxides. The infrared spectrum of diamond is equally unique and can be used to easily confirm that a stone is actually a diamond.

The extreme conditions required to create a diamond provide a way for trace amounts of other elements to be trapped in the carbon crystal matrix. The most important trace element is nitrogen, which can be found in different forms in a diamond crystal. These nitrogen aggregates create unique features in the infrared spectrum and are key in classifying diamonds. Hydrogen, boron, and carbonates are also found and have equally identifiable features in the infrared spectral region. While it is easy to confirm that a stone is a diamond, it is much more difficult to determine if it is synthetic or treated. Laboratory grown high-pressure high-temperature (HPHT) diamonds are now commercially available and their identification is one of the major challenges for the gem industry today. The infrared peaks corresponding to the nitrogen aggregates, or presence of other elements, can provide valuable evidence that a stone is not natural. In this application note, we will describe several relevant FTIR techniques that can be used for this identification in gemological laboratories.

Figure 1. Nicolet Summit X spectrometer with Collector II accessory.

The Thermo Scientific™ Nicolet™ Summit™ X FTIR Spectrometer, equipped with a Thermo Scientific™ Collector™ II Diffuse Reflectance Accessory, has the sensitivity to rapidly acquire high-quality spectra from faceted diamonds. When the system is configured with Thermo Scientific™ TQ Analyst™ Software, automated workflows can be created to analyze spectral data and quickly report results. Once the stone has been properly positioned in the instrument, a complete analysis can be completed in less than a minute with no effect on the stone itself.

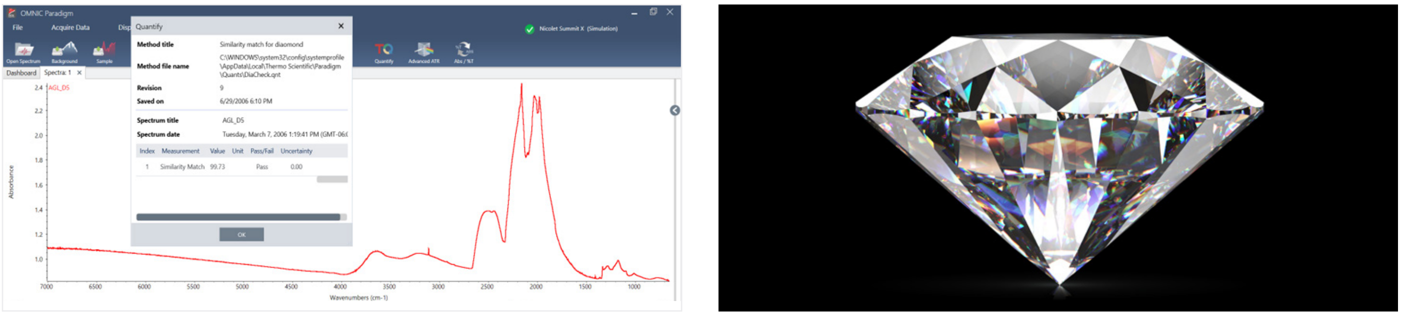

Figure 2. Diamond confirmation based on Similarity Match in TQ Analyst Software.

Experimental

Rapid confirmation that a stone is a diamond

Automatic material confirmation is a technique that has been extensively used in the pharmaceutical industry to ensure that incoming materials are correctly labeled. Reference spectra are acquired from all the materials that are used in the product and then a computer algorithm is employed to calculate the similarity between the incoming material and the specified reference spectrum. This same method can be applied to diamond identification; in this case, a reference spectrum from a Type IIa diamond was used to develop a similarity match method using TQ Analyst Software. The computed match value will be 100 if the spectrum from the sample is identical to the reference and zero if there is no similarity between the two spectra. (Since the spectra for all diamonds are slightly different, the match value will always be less than 100.) Generally, setting the acceptable threshold at 80 gave no false positives while allowing for a certain amount of variance in the sample spectra. The major exception was Type IIb diamonds with high boron levels, which severely distorted the phonon region.

The only features observed in a spectrum of pure diamond are the phonon bands from the crystal lattice structure in the 1500 cm^{-1} to 4000 cm^{-1} spectral region. There are technically three phonon bands, but one is not infrared active. A typical diamond analysis is shown in Figure 2.

Classifying diamonds

Classical least squares (CLS) is an extensively used quantitative analysis technique that assumes the spectrum from a sample that is a mixture of known materials can be modeled by a linear combination of the spectra from the pure reference materials.

In this study, reference spectra from diamonds with well-defined nitrogen aggregates were used: Type IaA, Type IaB, and Type Ib. Since the actual concentrations of nitrogen in the spectra were not known, an arbitrary value of 100 ppm was assigned; the actual values reported here are relative to this arbitrary value and are used for illustrative purposes only. The results of this example CLS analysis are shown in Figure 3. Although the calculated standard error terms indicate that all three nitrogen types are present, adding more standards to the method might resolve any questions about the presence of Type Ib aggregates.

Assisting in the detection of synthetic diamonds

Besides the platelet peak, several other peaks in the spectrum can help to identify treated stones. The amount of nitrogen in treated diamond is typically very low, so it can be difficult to confirm the presence of particular peaks. In this example, a simple CLS method was developed to confirm the presence, and measure the intensity, of peaks at 3107 cm^{-1}, 1344 cm^{-1}, and 1332 cm^{-1}. A key advantage to the CLS approach is that a standard error term is reported with the analysis. While it is not a rigorous statistical confidence level, if the peak intensity is three to five times the reported error term, the presence of a peak has been confirmed. An example for a sample containing very low levels of nitrogen is shown in Figure 4.

Conclusions

Numerous gemological laboratories employ FTIR on a daily basis to characterize diamonds and other gemstones. In this application note, a Nicolet Summit X FTIR Spectrometer was used in combination with a Collector II Diffuse Reflectance Accessory and multivariate statistical analysis techniques to provide rapid, reliable information for the classification of gemstones. By automating these methods, the results are consistent and supported by a quality (confidence) metric. In many cases, FTIR can be used by an operator to rapidly screen out the samples that clearly pass or fail a test, allowing the outliers to be examined by an expert.

Figure 3. Quantitative analysis of nitrogen aggregates in diamond by CLS.

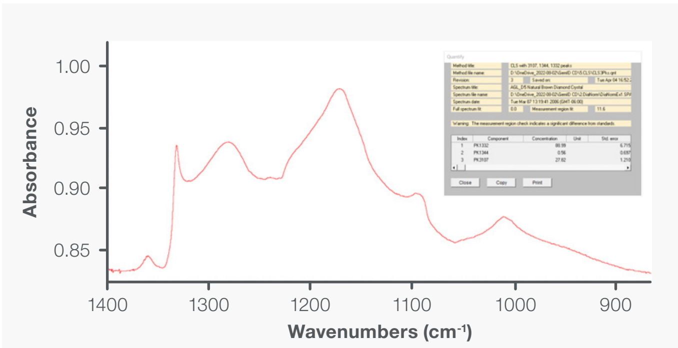

Figure 4. Nitrogen content evaluation based on CLS method.

Learn more at **thermofisher.com/summit**