

A Lead-Glass-Filled Corundum Doublet

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In September 2015, a 1.75 ct pink sapphire was submitted to the Gem and Jewelry Institute of Thailand's Gem Testing Laboratory (GIT-GTL). The stone showed internal features (e.g. flash effects and trapped gas bubbles) that are commonly seen in lead-glass-filled rubies and sapphires. In addition, the sample turned out to be an assembled stone, consisting of a pink sapphire crown and a near-colourless sapphire pavilion. These two portions were joined along a lead-glass-filled contact layer slightly below the girdle that locally contained areas of corundum fragments. We concluded that this stone was a lead-glass-filled corundum doublet.

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Introduction

Lead-glass-filled corundum was first encountered over a decade ago, and has continued to appear in the gem market in various forms (see, e.g., Kitawaki, 2004; Smith et al., 2005; McClure et al., 2006; Milisenda et al., 2006; SSEF, 2009; Henn et al., 2014; Leelawatanasuk et al., 2015; Ounorn and Leelawatanasuk, 2015; Panjekar, 2015). Nowadays such treated stones are widely available, mostly in the low-end jewellery market. The starting material typically consists of low-quality corundum, although a variety of colours and transparencies have been treated by this method (see references above). In September 2015, the GIT-GTL received an unusual lead-glass-filled sapphire that was characterized for this report.

Sample and Methods

The 1.75 ct sample consisted of a pear-shaped modified brilliant measuring $8.32 \times 7.21 \times 3.81$ mm (Figure 1). We used standard gemmological instruments to measure the stone's properties, and its internal features were observed with both

a gemmological microscope and an immersion microscope using methylene iodide. In addition, the gem was viewed with a DiamondView instrument. We used a Thermo Nicolet 6700 Fourier-transform infrared (FTIR) spectrometer

Figure 1: This 1.75 ct pear-shaped stone proved to consist of a sapphire doublet containing fissures and cavities filled with lead glass. Photo by S. Saengbuangamlam.



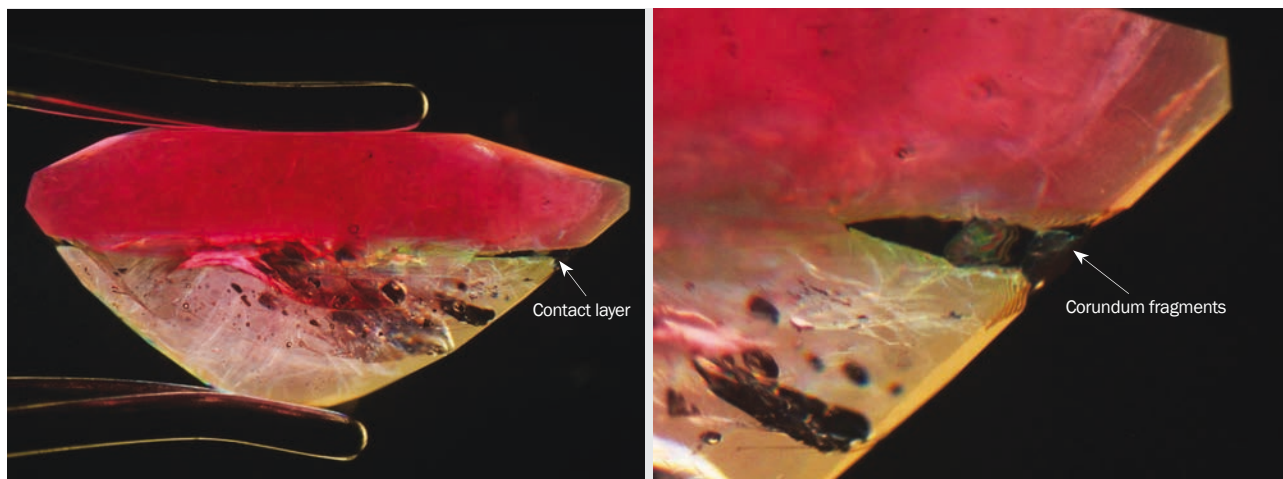


Figure 2: The doublet consists of a pink sapphire crown and a near-colourless sapphire pavilion that are joined along a contact layer located slightly below the girdle (left, image width 6.5 mm). Viewed with higher magnification, the contact layer is seen to locally consist of dark (isotropic) glassy material containing some anisotropic angular fragments of corundum (right, image width 2.2 mm). Photomicrographs by S. Promwongnan, in immersion with cross-polarized light.

to obtain IR transmittance spectra in the range of 4000–400 cm^{-1} . X-radiography was performed with a Softex SFX-100 instrument. Chemical analysis was carried out by energy-dispersive X-ray fluorescence (EDXRF) spectroscopy using an Eagle III system.

Results

Basic Properties

The gemmological properties of this stone were consistent with natural corundum. It had RIs of 1.770–1.761 (measured on the table facet) and a hydrostatic SG of 3.98. The polariscope gave a doubly refractive, uniaxial negative reaction. The stone fluoresced moderate red to long-wave UV radiation, with somewhat weaker luminescence to short-wave UV.

Microscopic Features

When the sample was examined using an immersion microscope between crossed polarizers, it was obvious that it was actually a composite stone (Figure 2, left). It consisted of a doublet formed of two distinctly different pieces: pink sapphire for the crown and near-colourless sapphire for the pavilion. These two pieces were joined together along a contact layer located slightly below the stone's girdle. Higher magnification (Figure 2, right) revealed that the contact layer was filled with glassy material that locally contained randomly oriented fragments of corundum.

Further examination with a standard gemmological microscope at high magnification showed internal features indicative of lead-glass-filled corundum. A blue flash effect associated with filled fractures/fissures was the most prominent characteristic, and was seen in both the pink and near-colourless portions (Figure 3a). Several trapped gas bubbles also were clearly seen in the filled fissures (Figure 3b). Additional inclusions consisted of minute particulates (Figure 3c) and planar 'fingerprints' (Figure 3d), which suggested that the corundum pieces forming both parts of this stone were of natural origin.

In the DiamondView, the relatively low lustre of the glass-filled cavities could be seen easily with reflected light using the instrument's sample chamber illuminator (Figure 4a). When exposed to the DiamondView's deep-UV excitation, the glassy material was inert and appeared as dark areas along the contact layer and in fissures and cavities, in contrast to the strong red fluorescence of the host corundum (Figure 4b,c). In places, the contact layer contained some bright-luminescing angular fragments of corundum embedded in the dark-appearing glassy material.

Mid-Infrared Spectroscopy

The mid-IR spectrum of the sample showed broad absorption features at approximately 3500, 2597 and 2256 cm^{-1} that are commonly present in lead-glass-filled corundum (Figure 5).

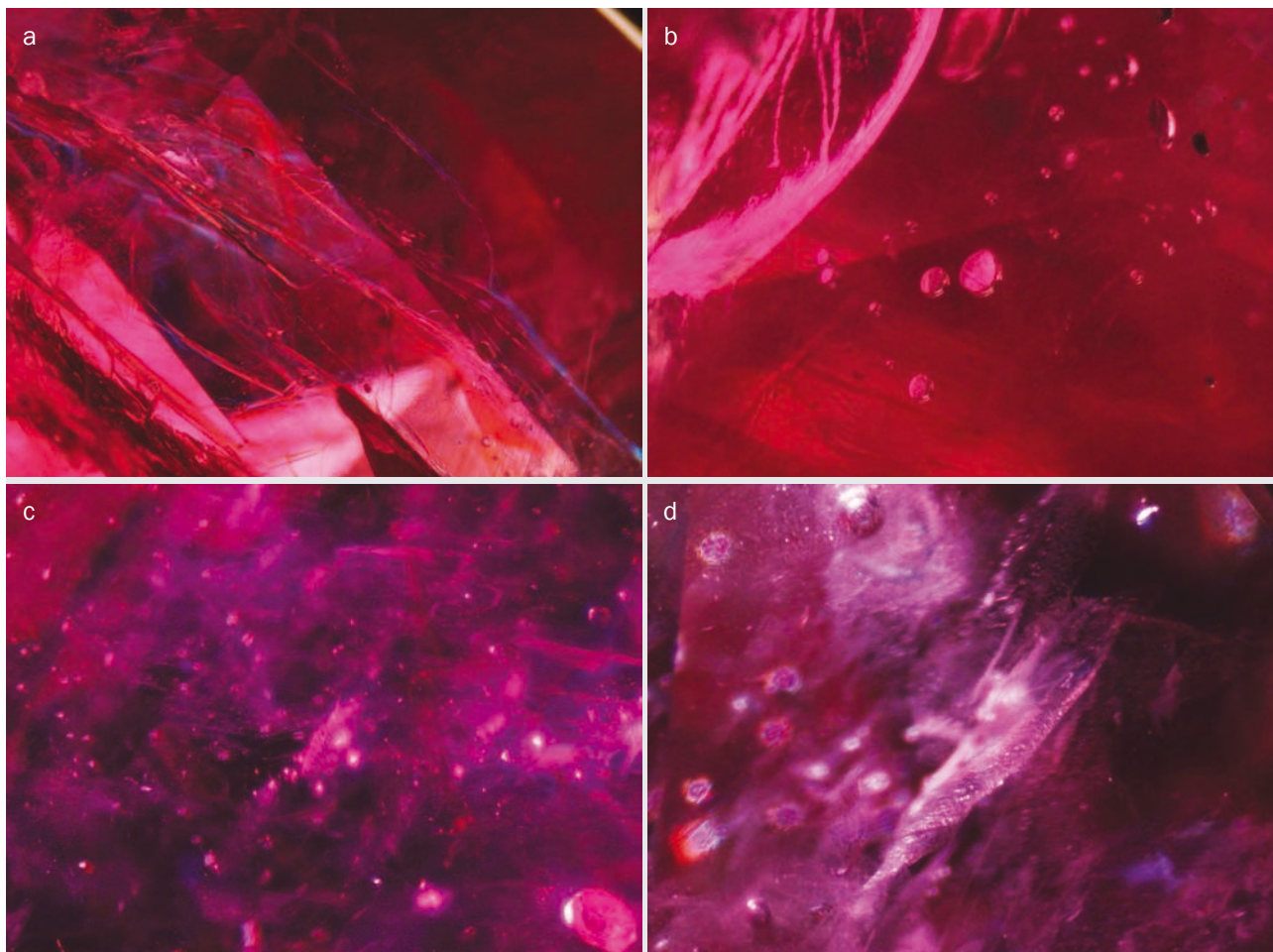


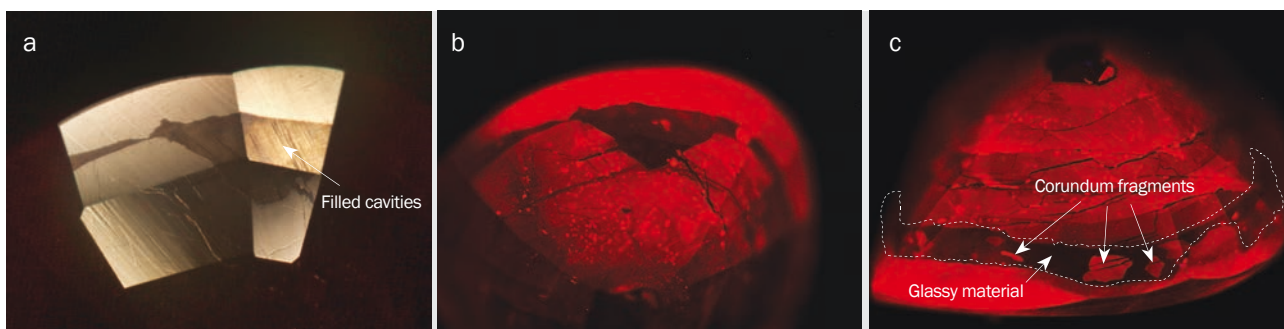
Figure 3: Internal features seen in the lead-glass-filled corundum doublet consist of (a) several filled fractures showing blue flash effects, (b) gas bubbles trapped inside the filler, (c) minute particulates and (d) planar fingerprints. Photomicrographs by S. Promwongnan; image widths 2.1, 1.5, 1.6 and 1.1 mm for photos a–d, respectively.

X-radiography

The X-ray image of the stone (Figure 6, left) revealed that the contact layer consisted of a high-density (dark-appearing) material. Furthermore, high-density material also was present along

many fractures inside the host corundum and in cavities on the stone's surface. These results are consistent with the presence of a lead-containing glass filler in fissures and cavities, as well as within the contact layer of this doublet.

Figure 4: These DiamondView images of the 1.75 ct lead-glass-filled corundum doublet show the sample in reflected light from the sample chamber illuminator (a), and exposed to ultra-short-wave UV radiation (b and c). The glass filler has a low lustre in reflected light, and is inert to UV radiation—in contrast to the strong red fluorescence of the host corundum. Note also the presence of some angular fragments of corundum in the glassy material along the contact layer between the crown and pavilion (particularly in image c). Photos by S. Promwongnan.



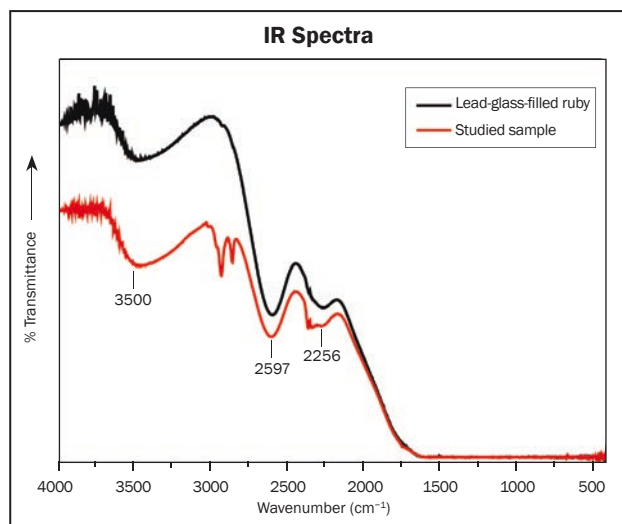


Figure 5: FTIR transmittance spectra are shown here for the tested corundum doublet as compared with a lead-glass-filled ruby reference sample. Both spectra display similar broad bands at approximately 3500, 2597 and 2256 cm^{-1} that are commonly seen in lead-glass filled materials. The bands at approximately 3000–2900 cm^{-1} in the doublet may be due to sample contamination (finger oils, etc.).

Chemical Analysis

EDXRF spectroscopy of the crown and pavilion sides of the corundum doublet showed traces of Ti, V, Cr, and Fe (and Ga in the crown); as expected, the Cr content of the pink sapphire forming the crown side was much higher than that of the near-colourless pavilion (Table I). Also detected were significant amounts of Pb and Si in both portions of the stone, which confirmed that it was lead-glass filled. Much greater quantities of these elements were detected in the pavilion than

Table I: Trace-element contents by EDXRF of the corundum doublet.

Area	TiO ₂	V ₂ O ₅	Cr ₂ O ₃	Fe ₂ O ₃	Ga ₂ O ₃	SiO ₂	PbO ₂
Crown	0.03	0.02	0.57	0.19	0.01	0.36	0.39
Pavilion	0.02	0.02	0.11	0.19	nd*	1.63	7.11

* Abbreviation: nd = not detected.

in the crown, apparently due to a larger amount of glass filling encountered by the beam in that area (i.e. within fractures and/or in the contact layer of the doublet).

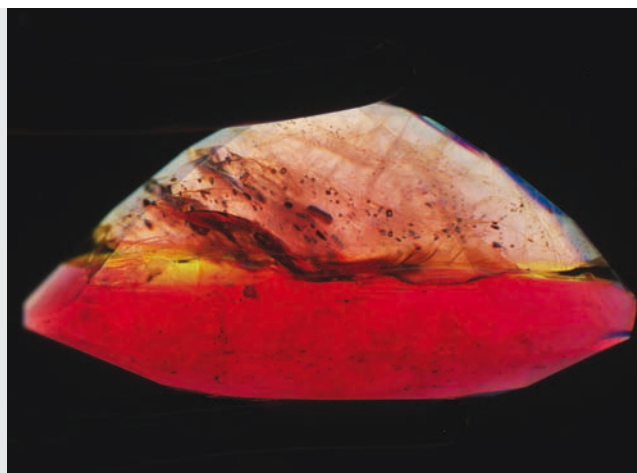
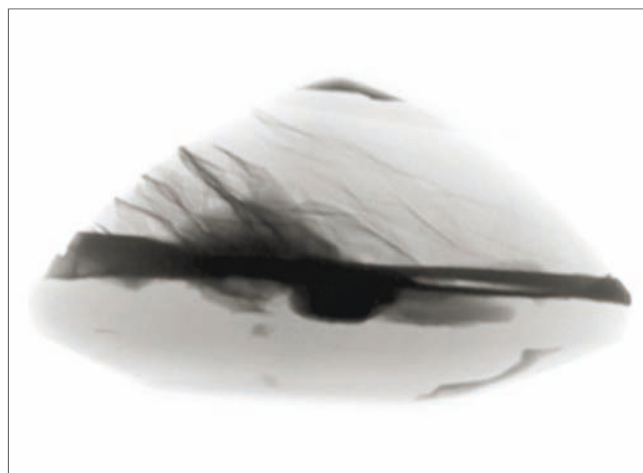
Discussion and Conclusions

Based on the above evidence, it is clear that this stone was a lead-glass-filled doublet composed of pink and near-colourless sapphire. The presence of small, angular corundum fragments within the glass-filled contact layer suggests that these two pieces could have been accidentally or intentionally brought together during the lead-glass treatment process. Bonding together of their flat surfaces enabled this composite material to be cut into an attractive but deceiving gemstone. Thus, this sample should be referred to as a lead-glass-filled corundum doublet.

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Figure 6: An X-ray image (left) of the lead-glass-filled corundum doublet is shown with a corresponding view taken in immersion with cross-polarized light (right). The X-rays reveal a high-density material that appears as dark areas in fractures and fissures, and along the contact layer between the crown and pavilion. Photos by S. Promwongnan; image width 6.4 mm.





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