

Breakthrough in Orange – the potential of GemLIBS in gemstone testing

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Beryllium diffusion-treated sapphires entered the gemstone trade in 2002. By this new treatment, low-quality corundum is modified into a stone of attractive colour, usually orange to rose colour. LA ICPMS analyses have shown that a concentration of 5 ppm Be diffused into the corundum lattice is already resulting in a distinct yellow to orange coloration of the stone. Natural corundum of similar colour does not contain Be and is rather rare and expensive. The trade was concerned about the

Fig. 0 Dr. Michael Krzemnicki of SSEF presents the SSEF GemLIBS at the International Jewellery Tokyo fair in January



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correct disclosure of such Be-diffusion treated stones, as testing based on traditional gemmological methods (for example microscopy, UV-VIS spectroscopy, FTIR spectroscopy, ED-XRF) was no longer providing all data necessary for a complete characterisation of such treated material.

In July 2003, SSEF first evaluated the possibilities of laser induced breakdown spectroscopy LIBS for gemstone analysis. LIBS had not been applied so far in gemmology. First research results showed that LIBS has a high potential to detect corundum that has been treated by the beryllium diffusion process.

THE METHOD: LIBS IN THE SSEF LABORATORY

By the end of April 2004, SSEF Swiss Gemmological Institute was the first gemmological laboratory having installed a LIBS instrument for gemstone testing. The instrument was specially designed for the institute's purposes in cooperation with Ocean Optics. The SSEF GemLIBS system (see figure 2) is based on

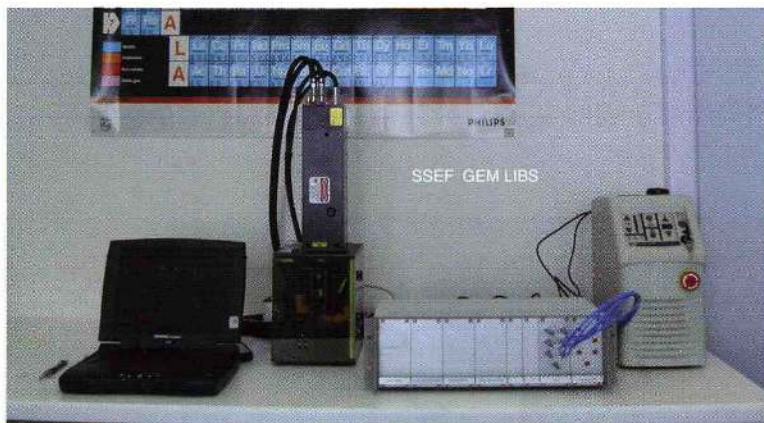


Fig. 1 The SSEF GemLIBS system consists of a number of portable units, including (from right to left) power supply, spectrometers, laser tube on sample chamber and computer display

a 1064nm Nd-YAG laser and a series of seven high-resolution fibre optic spectrometers, which enable recording of emission spectra in the range between 200 - 970 nm. A special sample holder enables precise targeting of the confocal laser beam to minimise any damage on the gemstone due to laser ablation. The gemstone is analysed on the girdle to minimise the visibility of the laser ablation (diameter of laser spot < 0.1 mm). When the sample is flushed with a stream of Argon gas the Be signal is enhanced by 5 times.

RESULTS AND DISCUSSION OF GEMSTONE TESTING WITH SSEF GEMLIBS

First results on the specially designed SSEF GemLIBS system have provided positive results to safely detect Be-diffusion treated sapphires. Beryllium could be detected in all the investigated diffusion treated samples (containing 3 - 50 ppm Be). Based on extensive testing, the institute was able to develop an analytical routine for serial testing of gemstones, especially for their Be content. Since June 2004, SSEF offers Be detection in sapphires as a reliable and affordable testing service for the gemstone trade. Several hundred stones have been successfully tested so far at SSEF.

The analytical procedure is characterised by:

- easy sample preparation
- minimal damage due to reduced laser beam energy
- precise positioning of the laser beam on the sample
- continuous emission spectrum (200-970 nm)
- simultaneous detection of many different chemical elements

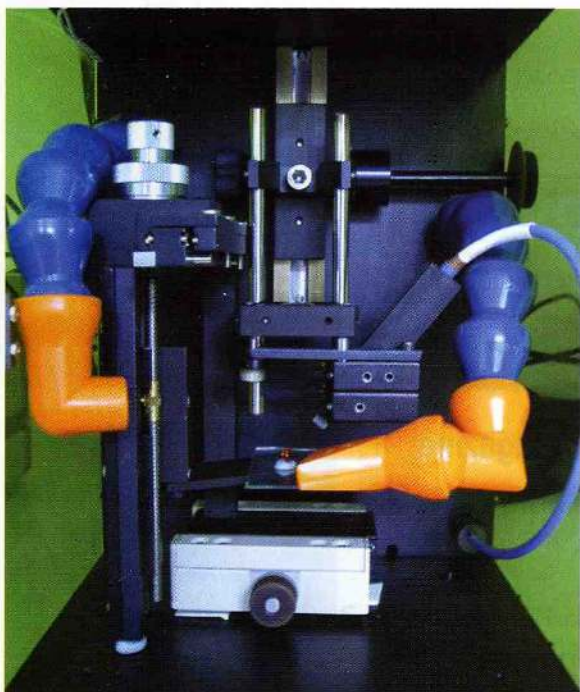


Fig. 2 Sample chamber with an orange sapphire mounted ready for analysis

Gemstones

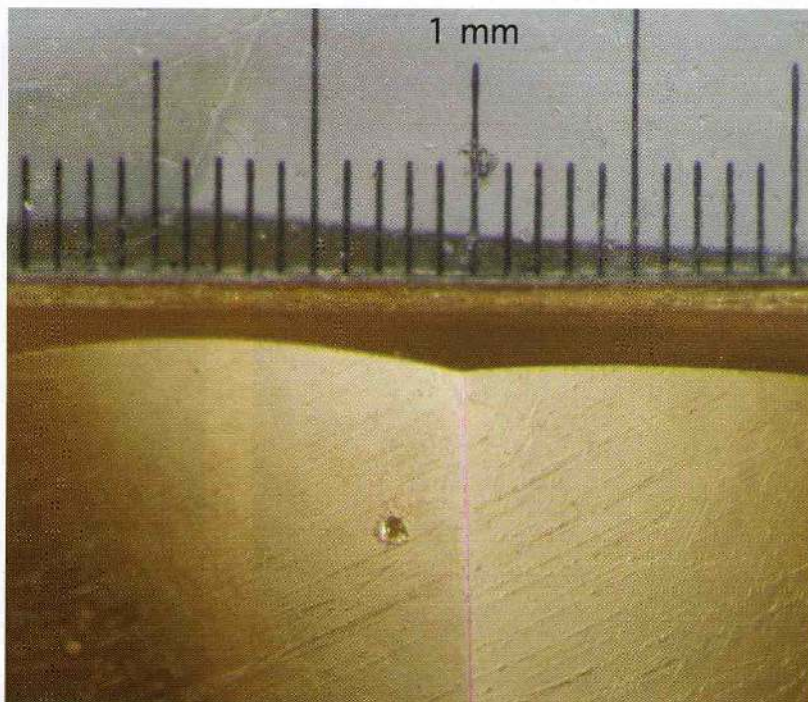


Figure 3 LIBS laser spots on a gemstone. The diameter of the spots is <math><0,1\text{ mm}</math> (<math><100\text{ microns}</math>)

- limit of detection for Be below 3 ppm
- much cheaper and easier than LA ICP MS or SIMS analysis

Further applications of the LIBS method are explored and will be published later. SSEF is selling the GemLIBS system with a know-how package and delivers offers on request. This summary is slightly modified from a poster presentation at the international LIBS Conference in October 2004 in Malaga, Spain, presented by Dr. Michael Krzemnicki of SSEF. A detailed article about the method is published in the Winter Issue of *Gems & Gemology* 2004.

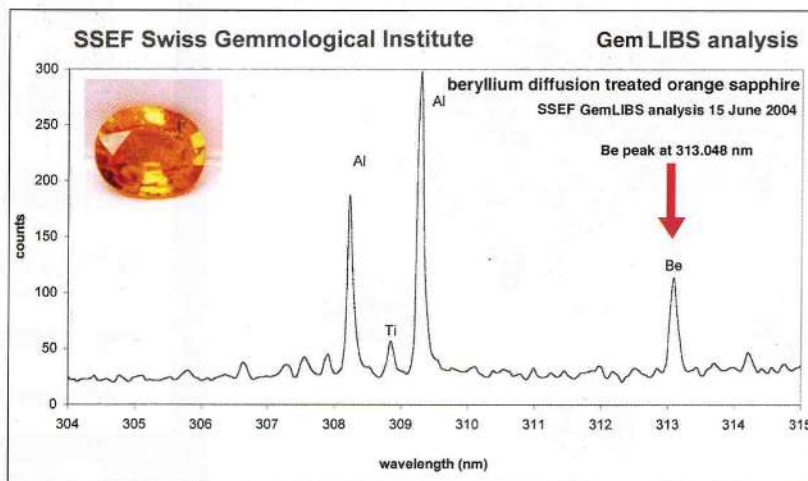
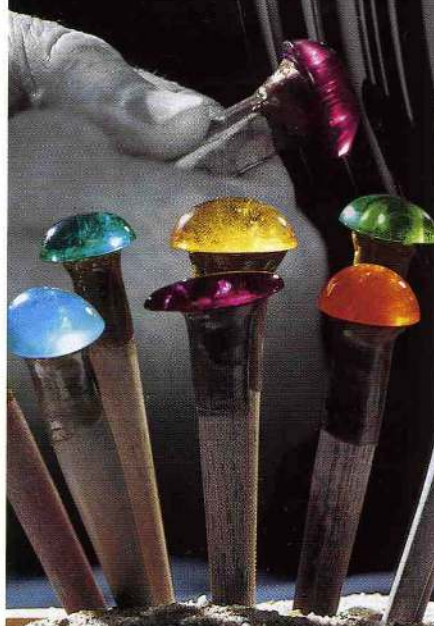


Figure 4: LIBS spectrum of Be diffusion treated sapphire. The Be detection is based on the Be peak centred at 313,1 nm. This peak corresponds to a concentration of 5 ppm, H.A. Hanni, SSEF 2004

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