CIBJO Ruby Day, Munich, February 2010

Heating and diffusion processes on rubies:

characteristics, detection and declaration

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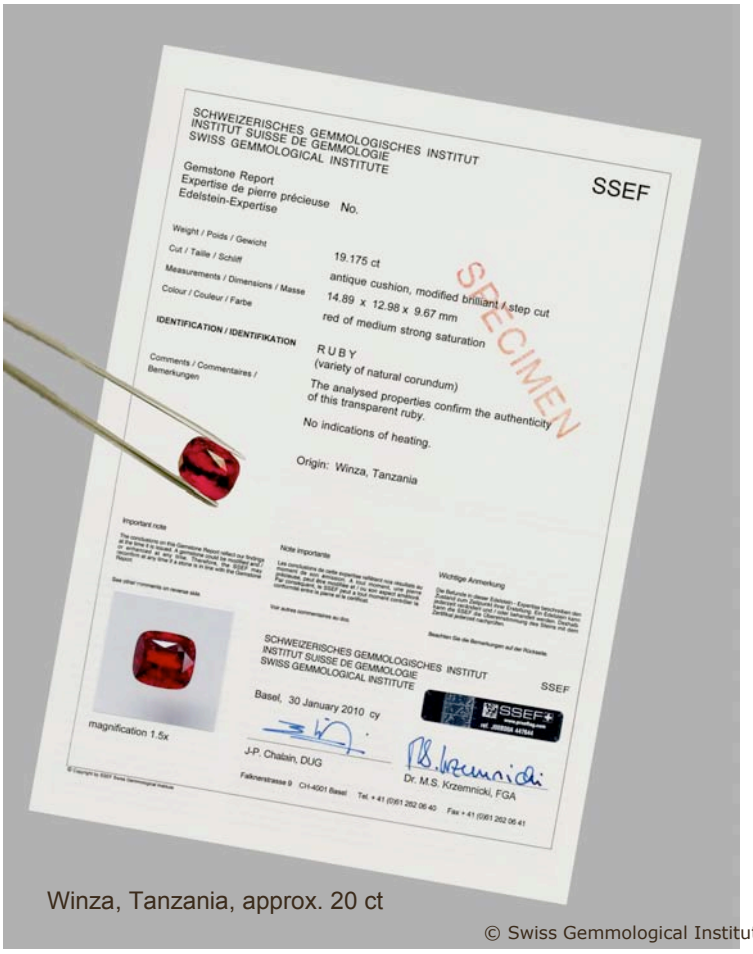


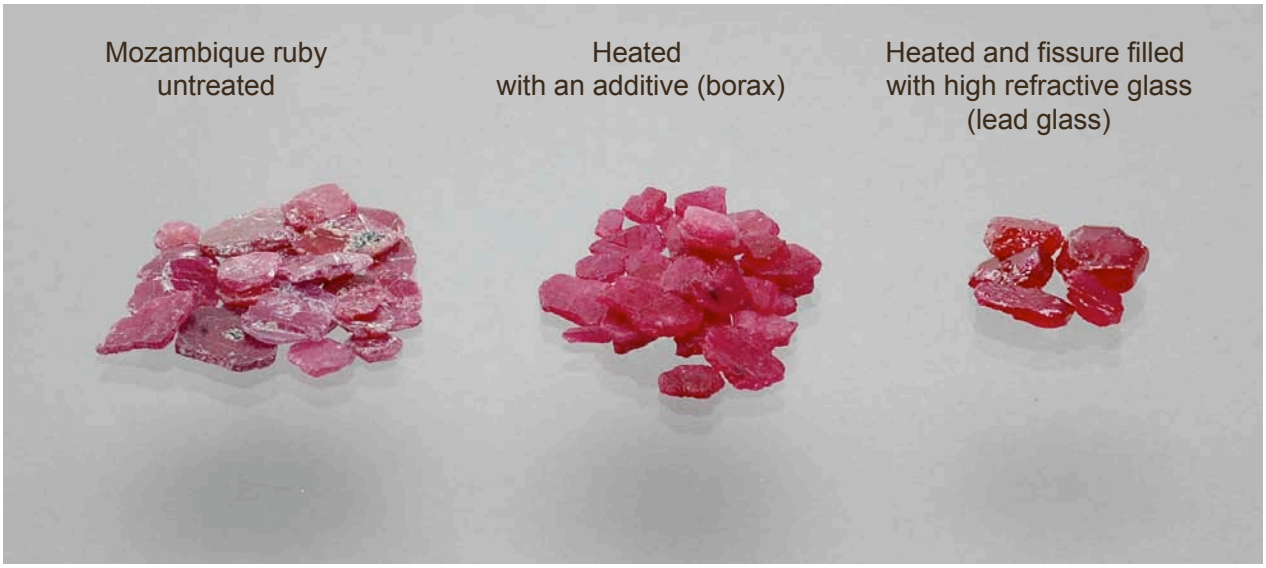
Winza, Tanzania, approx. 20 ct

The beauty of untreated Rubies

Mozambique, approx. 8 ct

Burma (Myanmar)

Ruby Treatment



most of them need a treatment to look better !

Ruby treatment

Heating

Low temperature heating (<1000 °C), e.g. Mong Hsu rubies and pink sapphires
effect: colour shift, blue colour component is reduced

Heating > 1000 °C, e.g. for geuda corundum, rubies from Thailand
effect: colour modification, reducing visibility of inclusions

Heating with flux

Heating with high refractive glass flux, e.g. lead glass
effect: significant enhancement of transparency and colour (and stability)

Heating with borax flux, e.g. rubies from Mong Hsu (Burma)
effect: significant enhancement of transparency and colour

Heating with diffusion

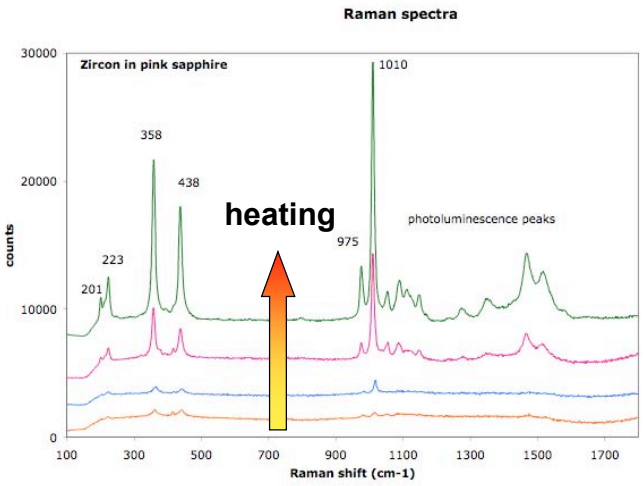
Heating with chromium diffusion (shallow)
effect: creation of shallow colour zone

Heating with beryllium diffusion (lattice, bulk), e.g. corundum from Songea
effect: significant colour modification

Low Temperature heating (below 1000° C)

By the heating at low temperature at oxidising conditions, the blue colour component is reduced, thus a better red to pink colour is achieved!

Detection of low-Temperature heating



Raman spectroscopy on zircon inclusions
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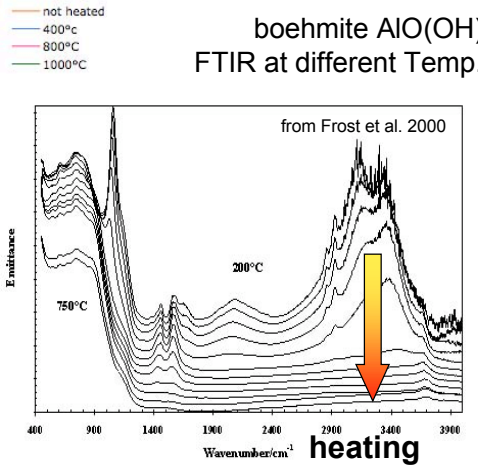
Very difficult with microscope!
But spectroscopic detection possible!

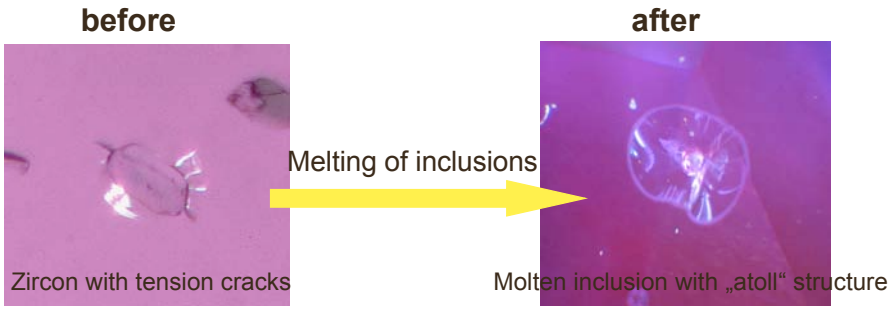


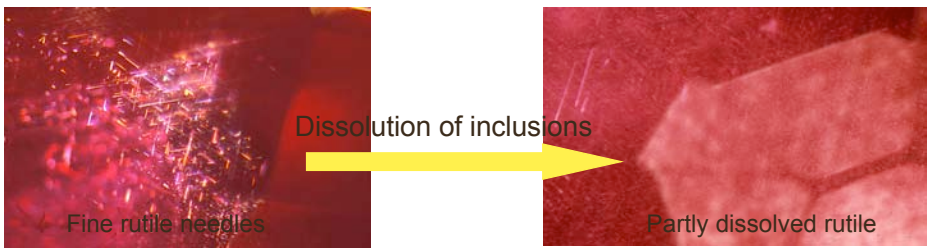
boehmite AlO(OH)
FTIR at different Temp.

Heating of ruby above 1000° C

At higher temperatures, it is possible to significantly enhance or modify the colour of corundum (for rubies, usually the bluish colour component is reduced to get a brighter red colour!). Furthermore, it is possible to reduce visibility of inclusions.

Detection of heating above 1000° C



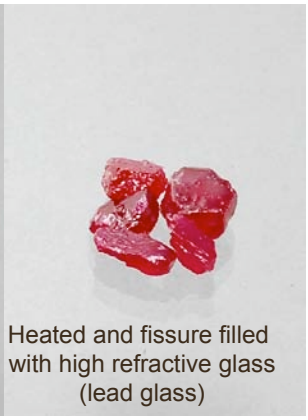


Generally possible with the microscope!

Heating with high refractive glass flux

In the last few years large quantities of rubies heated at low temperatures with a high refractive glass flux (e.g. containing lead, bismuth...) have entered the market.

Mozambique ruby untreated



Heated and fissure filled with high refractive glass (lead glass)

from Andilamena, Madagascar

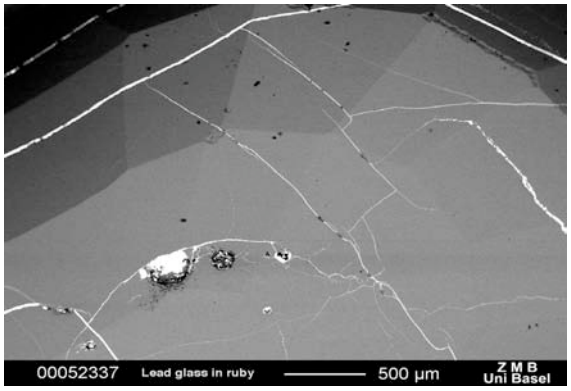
Rutile needles may be still present (sometimes rubies show even star effect !)

Detection of heating with high refractive glass flux

Blue and orange „flash effect“ (interferences) along lead-glass filled fissures. Also with distinct flattened air bubbles along fissures. At these low temperatures, no „healing“ of fissures occurs!

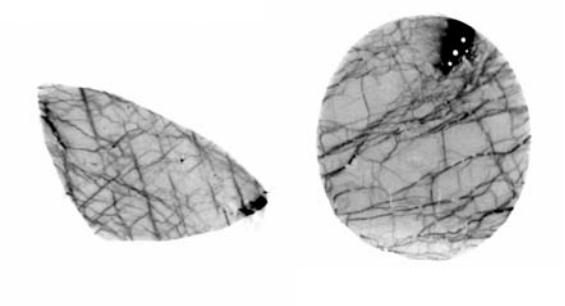
In most cases easy with the microscope!

Detection of heating with high refractive glass flux



Fissures filled with glass (lead = heavy element) appear bright on the SEM-BSE micrograph.

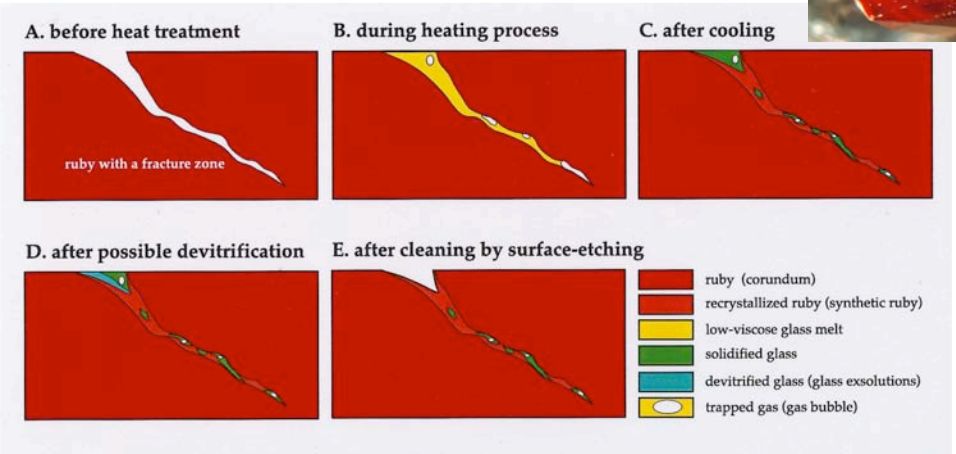
But also with chemical analysis radiography and X-ray micro tomography



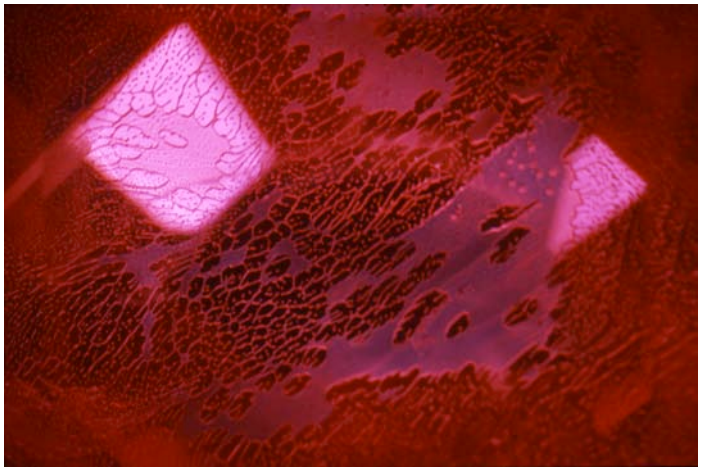
Tomographical cross sections showing X-ray absorbing glass filled fissures in dark.

Heating with borax flux

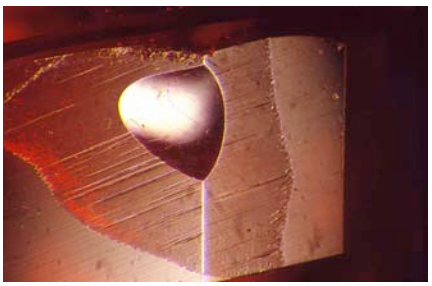
The borax flux is supporting the treatment induced “healing” of fissures by dissolution and re-crystallisation of ruby during the heating and cooling process. Residues of the borax flux are solidifying as glass droplets which are trapped within the rubies. The colour and the transparency are significantly enhanced!



Detection of heating with borax flux



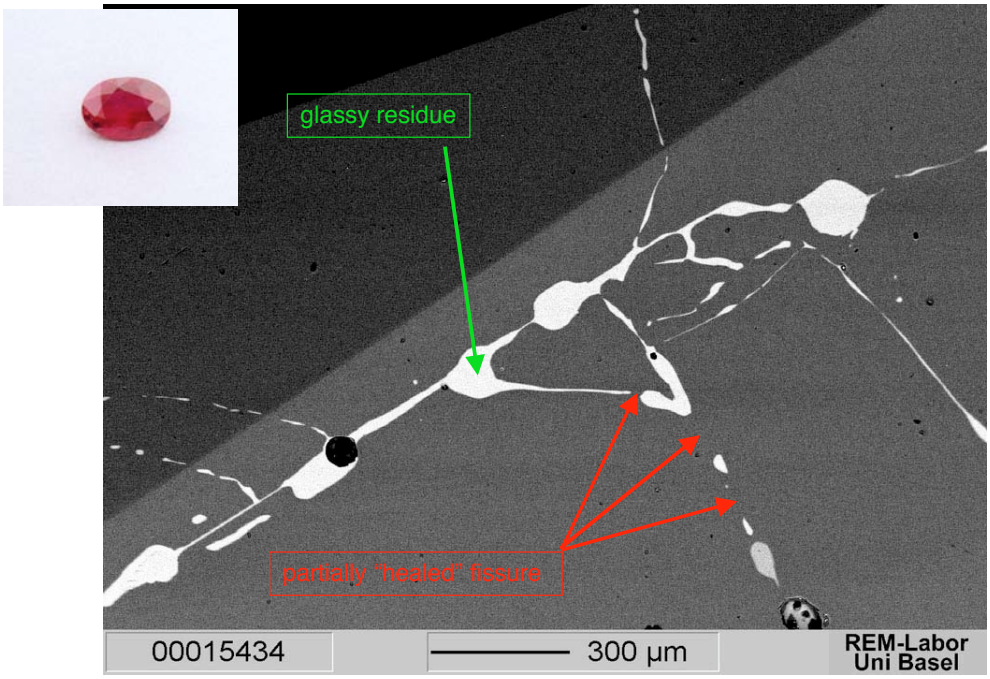
Residues of borax flux (glass) in thin films, curved tubes, and networks, along former fissure planes.



During heat treatment with the flux a glassy melt spreads over the surface, and may stay in cavities and fissures even after re-polishing.

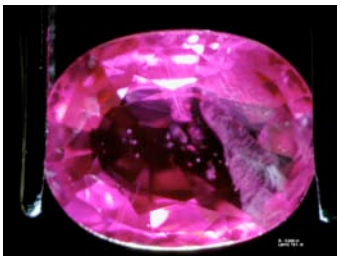
with the microscope!

Detection of heating with borax flux

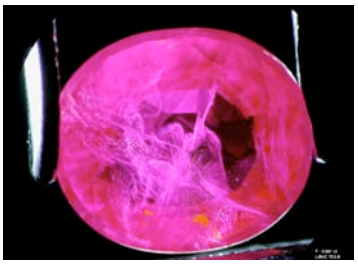


SEM-BSE micrograph

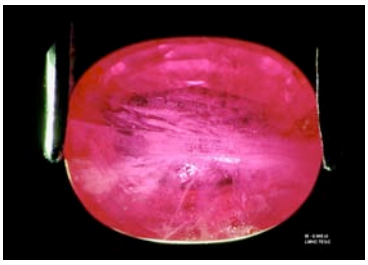
Quantification of residues



minor



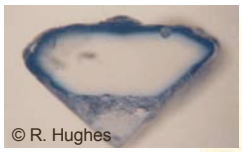
moderate

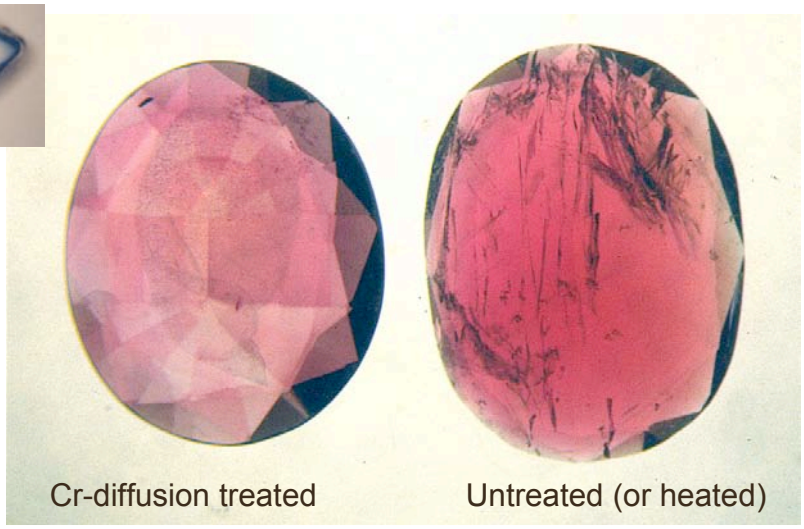


significant

See LMHC Infosheet 1

Heating with chromium diffusion (shallow)





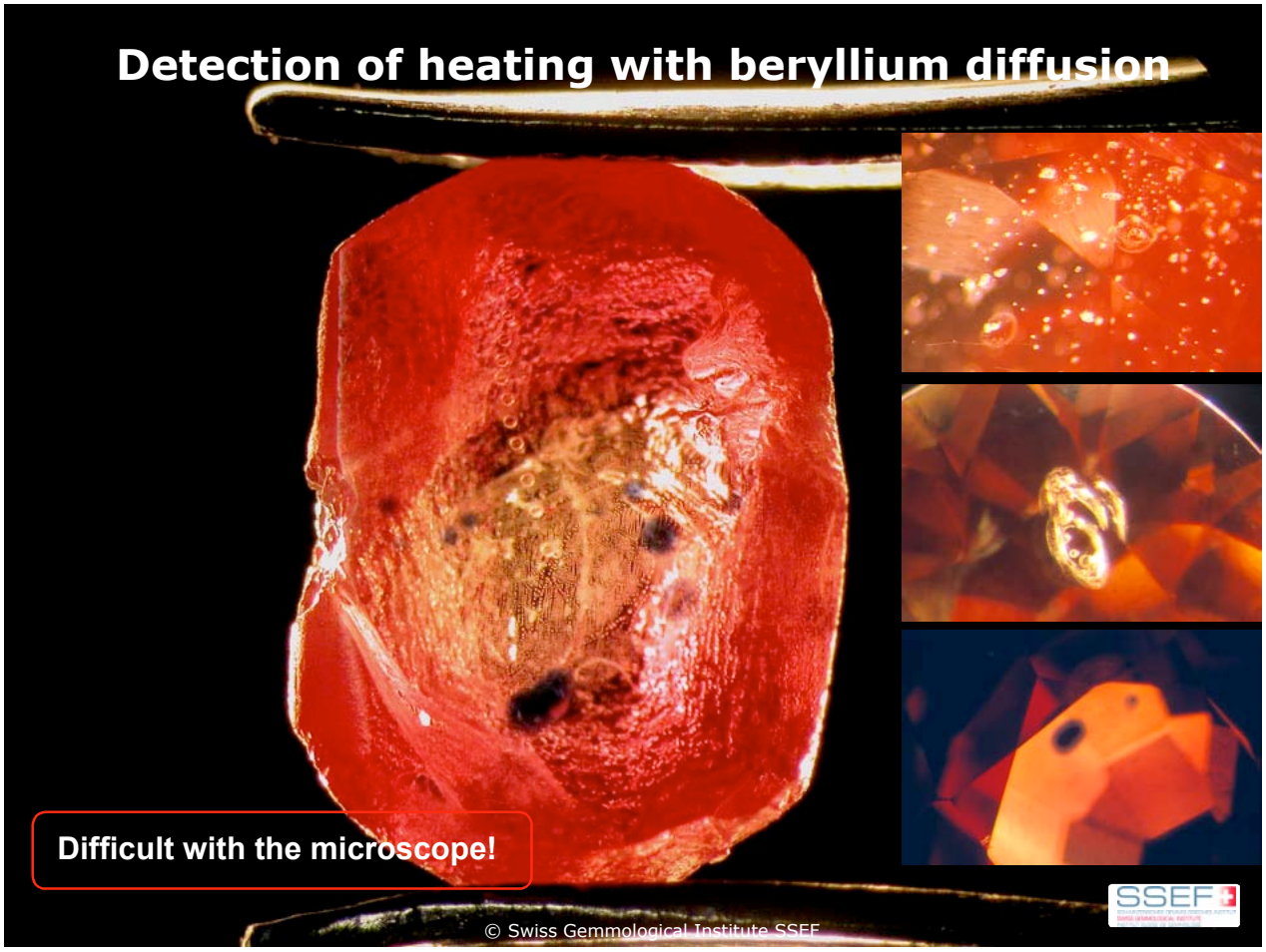
Chromium from an external source is entering the corundum lattice during a prolonged heat treatment at high temperature. The result is a very shallow red zone (blue with titanium) along the surface. By repolishing, the colour is lost!

Detection with the microscope and chemically

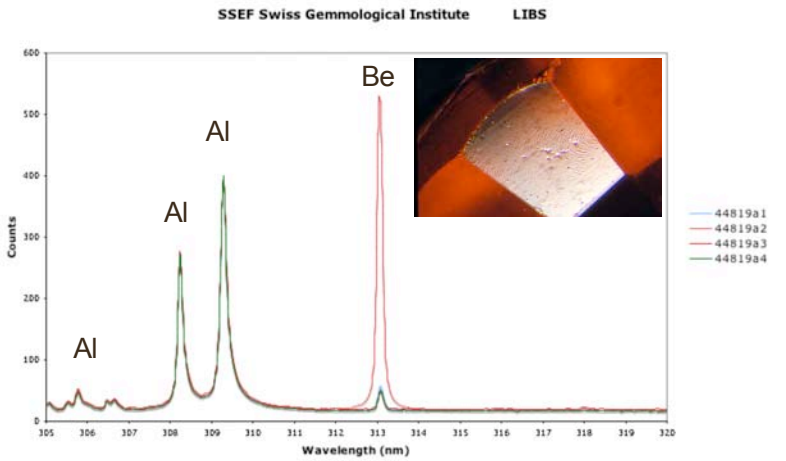
Heating with beryllium diffusion (lattice, bulk)

Beryllium diffusion treated rubies from Songea (Tanzania)

Beryllium from an external source is deeply penetrating the corundum lattice during a prolonged heat treatment at high temperature. The result is usually a significant colour modification often showing a yellow/orange rim (but not necessarily!).
By repolishing, the colour is usually not lost.



Detection of heating with beryllium diffusion



LIBS spectra of beryllium diffusion treated orange sapphire.
The high beryllium peak is measured in the glassy residue at the surface!
The corundum shows smaller peaks, indicating approx. 30 ppm Be (low traces!), but which is already enough for a colour modification.

Safe detection only with LIBS, LA-ICP-MS and SIMS

Disclosure

Internationally harmonized wording for the confidence of the customers !

The Swiss Gemmological Institute SSEF uses a full disclosure policy following **CIBJO** and **LMHC** (Laboratory Manual Harmonization Committee with the members CISGEM, GAAJ, GIA, Gübelin GGL, GIT, SSEF) recommendations:

Unheated ruby: - No indications of heating

Heated ruby: - Indications of heating

Heated with flux: - Lead glass filled fissures and cavities
Extent: significant (minor, moderate)
The introduction of lead glass into fissures involves heating
This treatment usually applies on low quality stones.

- Indications of heating with minor/moderate/significant residues in healed fissures

Heating with diffusion: - Indications of heating
Colour modified by shallow/lattice diffusion of chromium/ beryllium from an external source

Thank you
for your attention