Trade Alert:
Flux grown synthetic red spinels again on the market

by Dr. Michael S. Krzemnicki, gemlab@ssef.ch

During and after the Hong Kong Jewellery Fair in September the SSEF Swiss Gemmological Institute has received several red spinels for testing, which were found to be flux synthetic spinels. These spinels apparently have been offered in Bangkok in the last few weeks. Flux synthetic spinels are not a new issue. Actually, Nassau describes in his book "Gems made by man" (1980) that flux synthetic spinels were accidentally grown already in the mid-18th century in an attempt to produce synthetic rubies. Muhlmeister et al. (1993) and Schaub (2003) and Notari & Grobon (2004) have well described the properties of similar stones more recently.

Fig 1: Flux synthetic spinel of high purity and well-saturated colour. © M.S. Krzemnicki, SSEF 2008

With the appreciation, which fine quality red spinels are seeing nowadays in the market, it is not astonishing that these synthetic spinels have shown up just now again. Different to flame-fusion synthetic spinels (e.g. by Verneuil-process, mostly light blue, yellowish-green, and colourless), the investigated flux synthetic spinels are very convincing and similar in appearance to natural spinels of best quality. Only by meticulous microscopic observation and chemical analysis, their synthetic formation may become evident. As their identification is difficult if not impossible for the normal gemstone dealer, testing of spinels by a reputed gemmological laboratory becomes an issue nowadays.

Traditional gemmological methods are no help:
When analysing these flux synthetic spinels with traditional methods, no difference is seen in RI (~ 1.717) and SG (~ 3.60) as for natural stones. The spectroscope is no help either, as both, natural and synthetic red spinels are coloured by chromium and show the same absorption lines ("organ pipes") and -bands. Between the crossed filters of a polariscope, the tested flux synthetic spinels all showed distinct anomalous extinction due to internal strain, which may also be observed in natural spinels, especially around inclusions. Under LW and SW ultraviolet, the stones generally exhibit a distinct orange-red fluorescence, sometimes slightly chalky yellowish orange along facet edges. But again, a safe detection based on these observations is not possible.

Fig. 2: Faceted and rough flux synthetic spinels. The octahedral crystals have been produced in Russia in the early 90ies. The colour of the blue crystal is due to cobalt. Although not seen this time in the market, blue flux synthetic spinels may also appear again in the trade. © M.S. Krzemnicki, SSEF 2008

Microscopic evidence:
When checking these synthetic spinels with microscope, these flux synthetic spinels all showed a high purity with only a few inclusions. In all tested spinels we found small jagged to tubular cavities filled with black to orange brown residues of flux. The presence of large gas bubbles within the flux residues is due to an exsolution of the homogeneous flux during the cooling of the synthetic spinel. Apart from flux inclusions, one spinel showed a distinct six-sided metallic flake, most probably originating from the platinum crucible in which the flux synthetic spinel had grown. Furthermore, tiny ±parallel hollow channels were found in that specimen.

Fig. 3: Jagged flux residue with air bubble. © M.S. Krzemnicki, SSEF 2008

Natural red spinels, especially the ones from Burma (Myanmar) often are quite included, showing healed fissures with plenty of small octahedral negative crystals and various crystal inclusions, notably rounded (corroded) carbonates. Often they may also contain brownish iron-hydroxide in open fissures and cavities, which should not be confused with the above described flux residues in flux synthetic spinels.

Fig. 4: Six-sided metallic platinum flake and short hollow tubes have been found in one of the flux synthetic red spinels. © H.A. Hänni, SSEF 2008

Sophisticated analyses support microscopic evidence:
The chemical composition of the analysed red flux synthetic spinels is at a first glance quite similar to the composition of natural red spinels. Different to Verneuil-synthetic spinels which show a high alumina concentration, these flux synthetic spinels have a stochiometric Mg:Al similar to natural spinels. This explains also why the flux synthetic spinels do not differ in RI and SG from their natural counterparts. Apart from these main constituents, the stones all revealed distinct chromium concentrations (0.5 – 2.5 wt% Cr2O3), combined with traces of iron, vanadium, nickel, zinc, and gallium. Platinum was found in one specimen due to the metallic flake described above (see figure 3). These elements (except platinum) may also be present in natural spinels. In accordance with Muhlmeister et al. (1993), the main distinguishing feature is the low concentration of zinc (0.01 – 0.02 wt% ZnO). Natural spinels show concentrations generally exceeding these concentrations by a factor of ten or more (Schaub 2004).

Raman spectra show a distinctly broader peak shape (at 406 cm-1 Raman shift) for these flux synthetic spinels when compared to natural spinels. Similar peak-broadening is also known for Verneuil-synthetic spinel and is an expression of internal strain locally deforming the cubic crystal structure. Apart from this, the excitation with our green laser (514 nm) resulted in strong photoluminescence peaks due to chromium. However, these emission peaks are much less structured than in natural chromium-bearing spinels (see also Notari & Grobon), offering another good possibility to distinguish these flux synthetic spinels from natural ones.

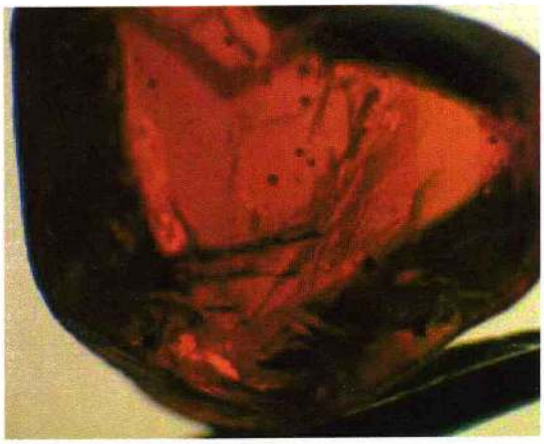

Fig. 5: Natural spinel from Myanmar with yellow and colourless crystal inclusions, small negative crystals and some fissures. © M.S. Krzemnicki, SSEF 2008

Even the crystals look like natural:
Flux synthetic spinels grow as well-shaped octahedral crystals. They often show triangular surface features similar to those commonly found on natural spinel crystals. Also slightly different in close inspection, these beautiful crystals may easily fool rough gemstone buyers when mixed into natural spinel crystals.
Only careful observation with the loupe or microscope, and eventually sophisticated testing at a reputed gemstone laboratory such as the SSEF Swiss Gemmological Institute may tell the difference between natural and synthetic.

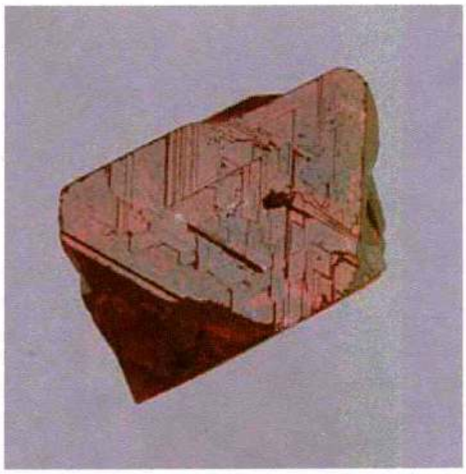
Fig. 6: Flux synthetic red spinel octahedron with triangular growth features on its surface. © H.A. Hänni, SSEF 2008

References:

Notari F., Grobon C. (2003) Spectrométrie de fluorescence du chrome dans les spinelles, Revue de Gemmologie AFG, N° 147, pp. 24-30.

Muhlmeister S., Koivula J.I., Kammerling R.C., Smith C.P., Fritsch E., Shigley J.E. (1993) Flux-grown synthetic red and blue spinels from Russia. Gems & Gemology, Vol. 29, No. 2, pp. 81-98

Schaub P. (2004) Spektrometrische Untersuchungen am Al-Spinellen, (unpublished diploma thesis), Mineralogical Institute of the University of Basel, Switzerland

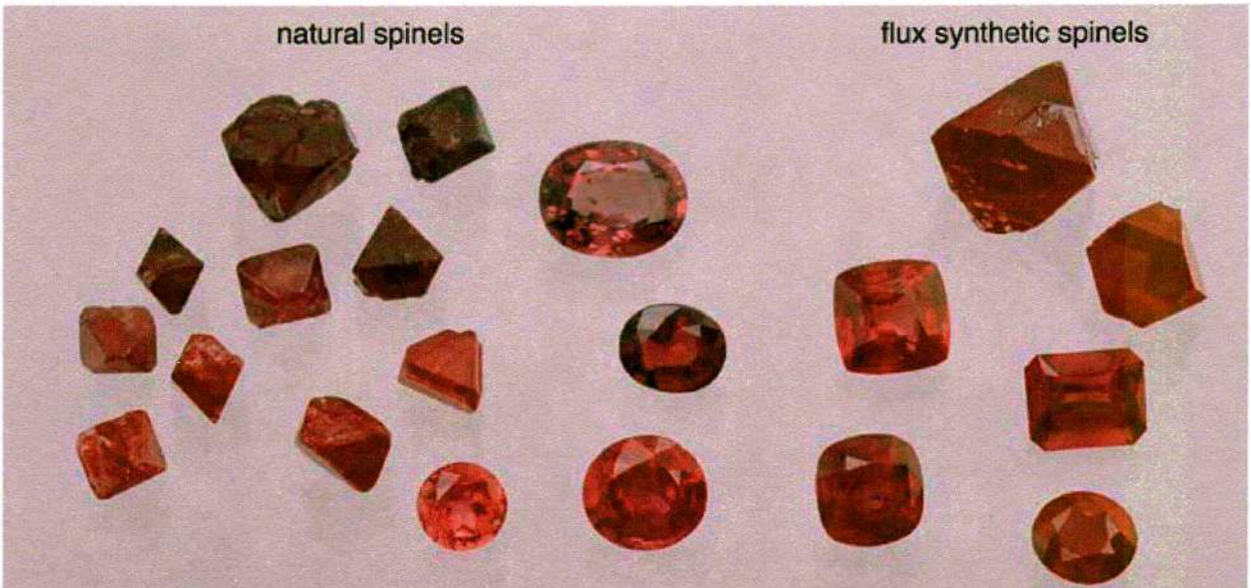

Fig. 7: Natural and flux synthetic red spinels (6 samples on the right) on display. Be aware that these synthetic spinels are growing as octahedra very similar to natural spinels. Only careful observation and sophisticated analyses will tell the difference © M.S. Krzemnicki, SSEF 2008

SSEF
SWISS GEMMOLOGICAL INSTITUTE

The science of gemstone testing®

Falknerstrasse 9
CH-4001 Basel / Switzerland

tel. +41 61 262 06 40
fax.+41 61 262 06 41

e-mail: gemlab@ssef.ch
homepage: www.ssef.ch