Identification of fissure-treated gemstones

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Introduction

The subject of enhancing the appearance of various gemstones in which fractures have a detrimental effect on their beauty or saleability has been addressed previously by a number of authors (e.g. Ringsrud, R., 1983; Kane, R., 1984; Scarratt, K., and Harding, R.R., 1984; Martin, D.D., 1987; Hänni, H.A., 1988; Koivula, J.I., *et al.*, 1989; Themelis, T., 1990; Shida, J., 1991; Kammerling, *et al.*, 1991; Hänni, H.A., 1992).

Fissures in gemstones, originate from mechanical tension, pressure or temperature stress. They originally contain vacuum, gas or a fluid. If such fractures reach the surface of the stone, the narrow voids of the fractures are filled with air. All of the above media possess refractive indices considerably lower than that of the gemstone itself. The fractures are therefore capable of reflecting light. This is an undesirable capability that would be considerably reduced if they were to be filled with a substance of a similar refractive index to that of the gemstone.

The objective of this short review is to bring the topic of artificially introduced filling substances, which may be used in gemstone enhancement procedures to the attention of the reader. It seems to the author that developments in this field have either not been given sufficient exposure, or have gone unnoticed by certain elements of the trade during the last few years. It is also important to be able to differentiate clearly between natural deposits on fracture planes (e.g. iron hydroxide) and organic artificial matter, commonly but very often incorrectly called 'oil'.

Why a treatment can be effective

Light entering a faceted gemstone only re-emerges after multiple internal reflections between several facets. Along its path the light is absorbed selectively and in a manner that the transmitted spectral colours result as the perceived colour of the gemstone. The shorter the distance the light travels, the weaker the absorption and the lighter the colour. Should the light path be interrupted by a fracture, because it is reflected at this point, the length of light path is shorter than it might be if the crack were not present and the colour thus weaker. Also the reflecting fractures are conspicuously lighter to the observer (or darker if the fracture plane is observed from behind the 'mirror' of the reflecting plane).

If a filler replaces the air previously in the fracture, and the filler substance has a refractive index higher than air and near to that of the treated stone, it inhibits a reflection of light rays from most incoming directions. The light may then travel across the fracture plane and is not deviated or reflected. The fracture no longer acts as an obstacle in the light path.

On many occasions fractures, pores or grain boundaries of polycrystalline stones are used to introduce colour into the material. Recently jadeite has been treated with acid, then heated to widen the grain boundaries and increase the porosity. The stones are now easier to treat with an artificial resin. The result is called B-Jade and in the Far East leads to a superabundance of supposed Imperial jade.

Another example is represented by an originally colourless, heavily fractured corundum which is cut into beads for necklaces (Figure 1). The beads are treated with a ruby-red stain, which may be seen on fractures and twin planes. The material makes a very convincing imitation of ruby (Schmetzer *et al.*, 1992).

How treatments are performed

For decades, if not centuries, fillings of oils and resins with a viscosity low enough to penetrate into fractures have been used to seemingly improve the quality of the stones. The lower the viscosity of the filling medium, the more complete is the penetration into the fissure. Sometimes vacuum techniques are used to improve the result. The range of fillers encompasses numerous substances: apart from those such as vegetable and mineral oils with volatile components (Figure 2), there are the more durable fillers like fats and resins. Compounds such as synthetic epoxy resins are frequently used today (Themelis, 1990); these possess the advantage of a

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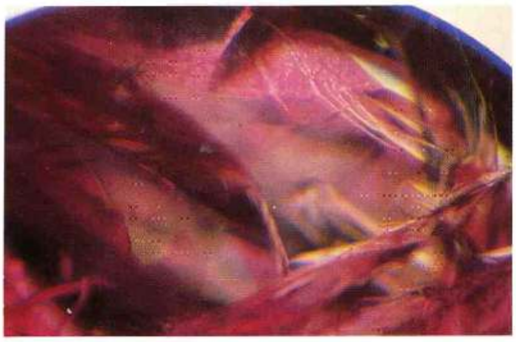

Fig. 1. Fractures stained red do not make a ruby! this picture shows part of one button shaped bead in a necklace. The material was originally near-colourless corundum. Magnification 25x.

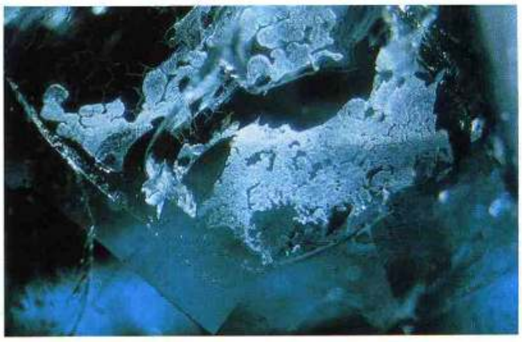

Fig. 2. Emerald with oil-treated fracture. Extensive 'lakes of air' form their lobular patterns near the fracture opening when the oil dries out. Magnification 20x.

more stable adhesion to the stone. This leads to a longer lasting improvement in appearance which is not possible with volatile compounds like oil and paraffin used traditionally. The latter are released quite easily due to their solubility in soaps, detergent and solvents.

The most stable fracture filler material is glass, which is introduced into fractures in its molten state. For the moment, diamond and corundum are the only gemstone species treated by this method. Only these species resist the required high temperature without being seriously damaged (Kane, 1984; Hänni, 1986). All other stones like quartz, tourmaline, beryls, jade, lapis lazuli etc are normally treated with natural or artificial organic compounds.

Emeralds

Before a filler is applied to an emerald, a cleaning process should be used to remove all dirt and residual filler material from any former treatments. This may be done by prolonged immersion in solvents and/or acids, possibly with the assistance of ultrasonics or steam. The use of a vacuum may extract previously inserted 'oil'. The procedure of cleaning may, depending on the intended filler to be used, take many hours or even days (Themelis, 1991).

For a good penetration into the fractures, a low viscosity filler is essential. This objective is reached with oil, paraffin (Figure 3) or resin by slightly heating these and using a vacuum to assist in the treatment. Special additives are sometimes used to lower the viscosity as far as to 260 centistokes.

A well known product name is sometimes used for a group of epoxy resins with similar characteristics: Opticon. Most emeralds in the trade today are treated in the rough or cut state with 'Opticon' or a similar product. Other epoxy resins such as Araldite NU 471, Dobeckot 505, Novogen P40 etc have similar effects as fillers. Their refractive indices are in the range 1.5 to 1.6.

Fig. 3. Resin treated fracture in an emerald, with spectral colour flashes and dendrite-shaped gas bodies. Opticon treated fractures may look like this. Magnification 20x.

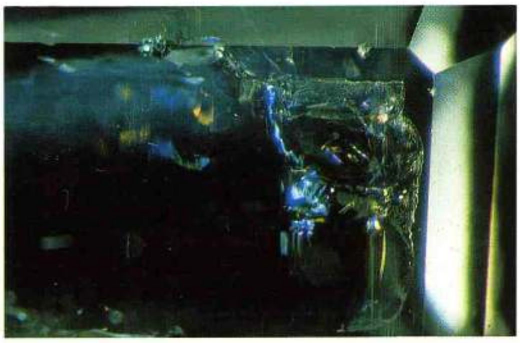

Fig. 4. Ruby with a fracture, probably treated with wax or paraffin. A typical dendrite-like pattern is visible.

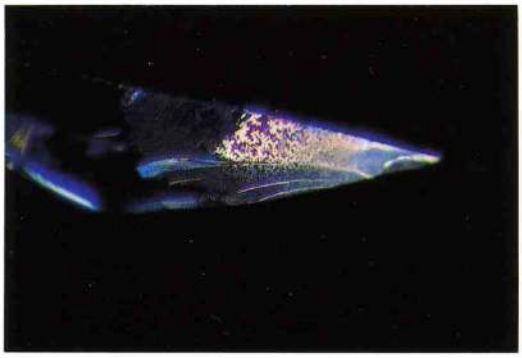

Fig. 5. Sapphire with a filling of artificial resin, showing dendrite-like patterns of gas bodies. In UV light the filling has a whitish fluorescence. Magnification 40x.

Epoxy resins can be used with or without a hardener. If the resin is used without a hardener, it remains soft and usually entirely fills the fracture. Gas bubbles and other structures like viscous fingering are rare in the fissure planes. When the artificial resin is mixed with a hardener, polymerisation takes place. A shrinking effect causes fine fissures and pseudo-dendritic patterns to occur in the filler plane, making it easier to detect.

Corundum

Although some historical filling substances are encountered today (most probably wax or paraffin) the majority of fractures in ruby and sapphire are treated with a boro-silicate melt. Before treatment, a process of cleaning is performed to remove all dirt and residual filler material from any former treatments. Also natural deposits like iron hydroxide or clay minerals must be dissolved. A cleaning treatment is normally carried out by using hydrofluoric acid, which attacks all silicate minerals that may still

Fig. 7. Artificially healed fracture in a ruby. Under the flux effect of the glass the fracture plane has recrystallized. In newly formed crystalline cells, the glass has been trapped, devitrified and formed bunches of white radiating crystals. Magnification 40x.

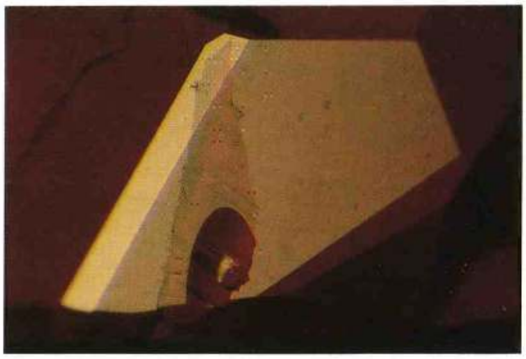

Fig. 6. A facet of a heat and glass treated ruby. A cavity (and all open fractures) have been filled with glass, identifiable by its lower lustre compared with corundum. Within the glass part an air bubble is cut by the surface. Magnification 20x.

adhere on rough stones. Sulphuric or hydrochloric acid may also be used to remove foreign material on and in the open fractures of corundum.

The treatment requires the presence of boron or lithium to lower the melting point of the silica-compound forming the melt. The melt, besides filling the fractures, also acts as a solvent or flux respectively and enables some recrystallization of the fracture planes.

Treatments with glassy substances today appear to be performed faster and/or at lower temperatures than some time ago, since the included rutile 'silk' may not be affected during the glass infilling treatment.

How treatments are identified

Some of the filler materials contain air or gas bodies, forming individual bubbles or dendrite-like patterns. Such patterns, when present, prove the existence of two different phases on a fissure plane. Many of the organic fillings show a bright fluoresc-

Fig. 8. Treated cleavage fractures in a brilliant-cut diamond. Yellow and violet interference colours together with gas bodies are characteristic for the Yehuda treatment.

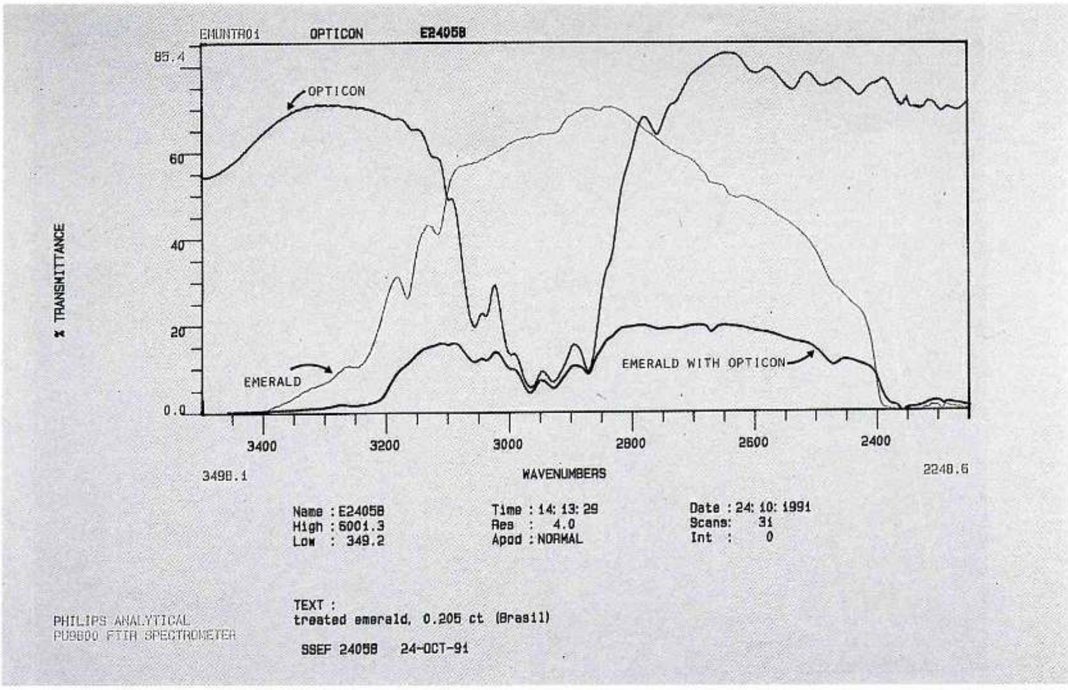

Diagram 1. Infrared spectra of untreated emerald, Opticon (artificial epoxy resin), and emerald treated with artificial epoxy resin. FTIR-Spectrum recorded by E. Jegge, SSEF laboratory, Zurich.

ence under ultraviolet light which is visible either with the unaided eye or a loupe.

The presence of oil can be indicated by bathing the stone in a solvent such as acetone or hexane for a short period. The soluble oil is diluted by the solvent. Under a microscope the drying out of the solvent which has replaced some of the oil is easily visible. Lobular air dendrites develop at the fracture openings. Such a reaction does not appear with resin treated fractures, since resins are less soluble.

Artificial resin fillings are often apparent by their orangy or violet colour flashes (Figures 4 & 5). A further proof for a foreign organic filling is possible by means of the hot needle. If the needle is put next to the fracture opening, the filling turns to a liquid state and moves slightly. The examination should be observed through the microscope and performed very carefully. The 'observation' of a foreign filling in a fracture by microscopic inspection may be quite easy, but the 'identification' of a particular filler may be difficult. Organic fillers, can be detected by infrared spectroscopy (see Diagram 1). The infrared spectrometer appears to be the most reliable tool for an identification, but owing to the numerous varieties of organic compounds, and the difficulties due to the superimposition of the stone's spectra over the filler spectra, this may prove to be a handicap for an exact identification of a particular filler. Also, if stones were subjected subsequently to different treatments without proper cleaning, the IR spectrum may be difficult to interpret.

Glassy substances, at the surface of a stone may be recognized by their different lustre compared with that of corundum (Figure 6, Hänni, 1986). If glassy fillings are to be identified within the stone, Raman spectroscopy may be the most useful method. In many cases, voids filled with artificial glass are whitish. The glass has served as a flux, recrystallized the fracture plane and sealed itself into a newly formed geometric void. In some instances devitrification starts, resulting in the appearance of radiating fibrous crystals that emerge from trapped gas bubbles (Figure 7).

Heavy liquids possess a high refractive index. This property is utilized for the Yehuda fracture treatment of diamond (Figure 8). Also with this fracture treatment, the filler shows vivid colour flashes, and the fracture planes contain gas bubbles and 'lakes' (see Koivula *et al.*, 1989). The heavy elements are identifiable by X-ray spectroscopy (Scanning Electron Microscope SEM or Energy Dispersive X-ray Fluorescence Analysis EDS-XFA). Glassy fillings in corundum show the presence of silica and Yehuda treated diamonds indicate the presence of lead and occasionally bismuth.

Outlook

Although the above mentioned treatments are not new, it appears that many trade members have little knowledge of some of them or do not wish to complicate their business by acknowledging their presence on the market. The use of colourless oiling is accepted as a common enhancement practice. This rather historic treatment method is being increasingly replaced by more stable treatments that utilize modern filler substances. Whilst the newer treatment methods in no way represent single cases, it is unfortunate that only a small number of stone dealers, jewellers or goldsmiths have the time, the knowledge, the motivation or the equipment to check or confirm these treatments. So it will probably take a long time for trade opinions about this kind of treated stone to change and make an impact on the market place.

At present time the CIBJO rules require only a disclosure of fracture treatments performed with substances other than colourless oil. This means that almost all emeralds, an important number of rubies and many sapphires should be labelled or sold respectively as treated gemstones. The exact identification of the numerous more or less viscous oils (vegetal, animal or synthetic), fats, paraffins, natural and artificial resins is rarely simple and often impossible. The analyses might be expensive and finally come only to the result that the stone is treated and has a foreign organic substance in its fissures. Because of the multitude of organic compounds which could serve as fillers it may be more appropriate not to draw an arbitrary line between 'permitted' treatments and those which are not tolerated without declaration.

This would mean a change in the CIBJO rules that govern this problem. To the author it seems advisable to handle all fracture treatments in the same way, either to disclose all fracture filled stones or to consider fracture filling as a common practice (whatever substance is used) and omit any comment to individual stones. However, the author considers that disclosure of the glass filling of any gemstone should be mandatory.

In any case the final consumer should be informed of the commonly used practices by his gemstone specialist to prevent damaging action by people with unfair goods.

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