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# Fluxes and the Heat Treatment of Ruby and Sapphire

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THE DISCOVERY OF EXTENSIVE RUBY deposits in the Mong Hsu area of Myanmar (Burma) has provided the marketplace with a much-needed source of fine ruby in smaller sizes (see, e.g., Hlaing, 1991; Henn and Bank, 1993; Kammerling et al., 1994). Much of the ruby from this location is characterized by blue or blue-black cores which generally are removed by heat treatment (Peretti et al., 1995). The heat treatment is straightforward: Temperatures of 900°C or above in an air atmosphere are usually sufficient to accomplish the desired change in color. When substantial quantities of Mong Hsu ruby began arriving at gemological laboratories worldwide, new features were recognized that were soon identified as residual flux inclusions (Peretti, 1993; Milisenda and Henn, 1994). These

*Figure 1. This 28 ct ruby from the John Saul mine in Kenya shows two glassy blebs that resulted from heat treatment without flux. They probably represent the melting of micaceous inclusions, and could easily be mistaken for residual flux.*



observations were mentioned in grading reports (Hänni et al., 1998), and have initiated a serious debate on the use of flux in heat treatment and, in particular, what the presence of large amounts of flux means in terms of the quality and durability of the original ruby.

What is a flux? A flux is simply a material that is usually a solid at room temperature, but at high temperatures it becomes molten and is a solvent for minerals and other inorganic materials. There are many types of fluxes in use, such as oxides, borates, silicates, molybdates, and fluorides, as well as various combinations of these materials. For example, fluorides are often used to reduce the viscosity of other fluxes. Fluxes based on borax (sodium tetraborate decahydrate) are widely used in Thailand for the heat treatment of ruby and sapphire (Abraham, 1982; Peretti et al., 1995).

It has been stated that a flux is necessary to prevent thermal-shock fracture of ruby and sapphire during heat treatment (Robinson, 1995). Nothing could be further from the truth. Natural ruby and sapphire are highly resistant to thermal shock fracture; much more so, in fact, than the ceramic crucibles in which the stones are placed, or the refractory materials of the furnace. Fluxes are used solely to enhance the appearance of lesser-quality material.

In principle, a flux can have many functions. It can slightly dissolve the surface of ruby or sapphire, giving it a somewhat polished appearance. It can fill fractures that are open to the surface. It can match the average index of refraction of corundum, thus masking fractures and other cavities. It can dissolve inclusions or staining in fractures that reach the surface. It can dissolve the amorphous layers that result from

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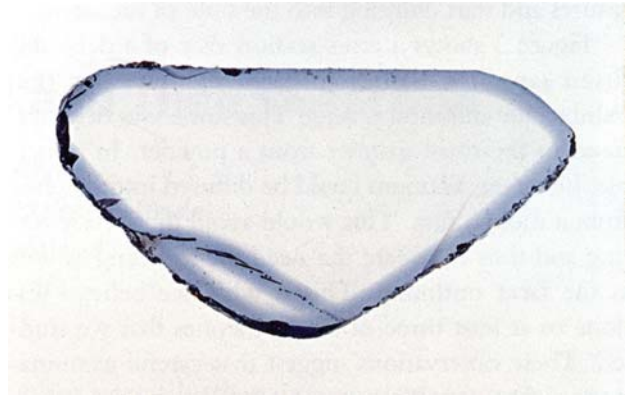
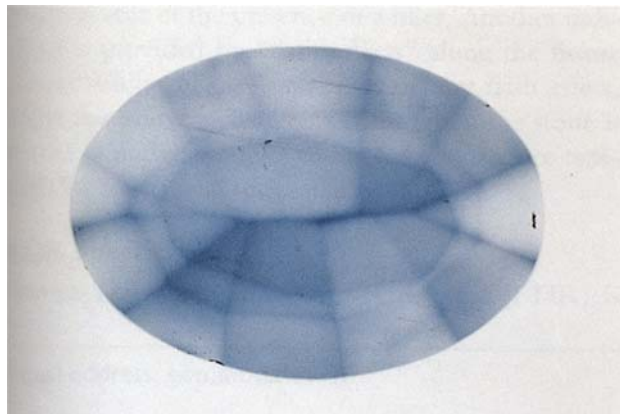
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aluminum hydroxide decomposition along parting planes. Finally, a flux can combine chemically with inclusions that melt out of a ruby or sapphire.

Upon cooling, a flux can redeposit dissolved corundum (Hänni, 1997–1998; Hughes and Galibert, 1998), resulting in very limited re-growth, or it can simply solidify as a corundum-containing material. It cannot, however, completely re-grow corundum in a fissure, because the flux usually contains less than 20% dissolved corundum (Nelson and Remeika, 1964; Linares, 1965). The solidified flux can be a glass, a polycrystal, a single crystal, or all of these phases, depending on the final composition, viscosity, cooling rate, volume, and substrate. In addition, each of these phases can have a different chemical composition.

Although today fluxes are used routinely to heat treat nearly all types of ruby and sapphire, determining that a flux has been used is *not* straightforward. First, many fluxes are based on borates, and boron is not detected by the X-ray fluorescence (XRF) spectrometers routinely used in gemological laboratories. Second, mineral inclusions may melt out of the corundum during heat treatment, and these melt droplets may appear very similar to flux deposits. The two glassy blebs on the surface of the ruby in figure 1 could easily be misidentified as flux. These glassy materials are composed of the oxides of silicon, aluminum, and magnesium, sometimes with calcium,

*Figure 2. Color concentrations along facet junctions are clearly visible in this deep-diffused sapphire that has been immersed in methylene iodide. This uneven coloration—which is considered characteristic of diffusion treatment—results primarily from the recutting and repolishing of the stone, not the diffusion treatment.*



*Figure 3. This slice of a faceted, deep-diffused sapphire shows the diffusion profile extending out from a “fingerprint”-like inclusion (bottom left) that reaches the stone’s surface. The deep diffusion of this sample was performed by heating in a Ti-containing powder, which is the usual practice. However, a Ti-containing flux theoretically could produce the same result without the surface damage caused by powder sintering.*

potassium, or sodium. They apparently result from the melting of various micaceous mineral inclusions. Given the wide compositional range of fluxes, the variety of inclusions that can melt and/or dissolve in a flux, and the variety of morphologies that result from the cooling of flux materials, the identification of a flux as a filling in fractures or cavities can be very challenging.

In what may be yet another application of molten fluxes, we recently examined five fine sapphires of 3–5 ct each that we believed to have been deep diffused; yet they did not exhibit the color concentrations at the facet junctions that are typical of this treatment (Kane et al., 1990; see, e.g., figure 2). Note that this facet outlining is an artifact of recutting and repolishing the sapphire, which was necessitated by the pitting of the surface that was caused by powder sintering (Carr and Nisevich, 1975) during deep diffusion, and not the deep diffusion process itself. Prior to recutting and repolishing, the outlining of the facet junctions in deep-diffused gemstones generally is not visible even with immersion. We identified three of these stones as being deep diffused on the basis of the diffusion profiles seen on surface-reaching “fingerprint” inclusions when they were viewed edge-on. Because titanium dioxide—the material typically used for diffusion treatment—has a very high surface-diffusion rate on sapphire, it will propagate into any open

fissures and start diffusing into the bulk of the stone.

Figure 3 shows a cross-section slice of a deep-diffused sapphire with a fingerprint inclusion that exhibits the diffusion profile. This stone was deep diffused in the usual manner from a powder. In principle, however, titanium could be diffused into sapphire from a molten flux. This would avoid the surface pitting and thus eliminate the need for recutting as well as the facet outlining. This is what we believe was done to at least three of the five stones that we studied. These observations suggest that careful examination of fine sapphires is most prudent, even when there is no evidence of color concentrations at the facet junctions.

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