NOTES
· AND ·
NEW TECHNIQUES

INDUCED FINGERPRINTS

By John I. Koivula

Over the past few years, numerous Verneuil-type (flame fusion) synthetic sapphires and rubies with somewhat natural-appearing induced fingerprint inclusions have surfaced in the trade. This article reports the results of a series of experiments conducted to explain the phenomenon of induced fingerprints and how they are produced in gemstones generally and in flame-fusion synthetic corundum specifically.

Since 1980, the presence of somewhat natural-looking fingerprint inclusions in flame-fusion synthetic corundums has haunted the colored stone industry. After the first report in the literature (Crowningshield, 1980) of this new treatment, a number of treatment-related articles appeared in various gemological and jewelry trade publications. These articles detailed most of the treatment processes, such as diffusion, used on rubies and sapphires, but they only briefly mentioned induced fingerprints. In two excellent articles on heat and diffusion treatment of natural and synthetic sapphires (Crowningshield and Nassau, 1981; Nassau, 1981), the mechanisms used to induce fingerprints are described as unknown. However, the statement by Nassau (1981) that "according to some unsubstantiated reports, a flux-type chemical such as sodium carbonate or borax may assist in this process" provided an important clue to the production of induced fingerprints. Another important clue is given by C. R. Beesley (1983), who stated that "an outgrowth [of heating synthetic sapphire] was to induce fractures from the surface of the material [synthetic sapphire], then force some material into the fractures which could be almost crystallized during heating. This gives the appearance of a natural fingerprint." The statement concerning the fractures induced from the surface is significant (although the comment about some material being forced into the fractures which could be *almost* crystallized during heating, is confusing at best).

To better understand and clarify the mystery surrounding "induced fingerprints," the author performed a series of "before and after" heating experiments on flame-fusion rubies that were based on observations made on natural fingerprints and those found in flux and hydrothermally grown synthetic gemstones. The results of these experiments are reported below.

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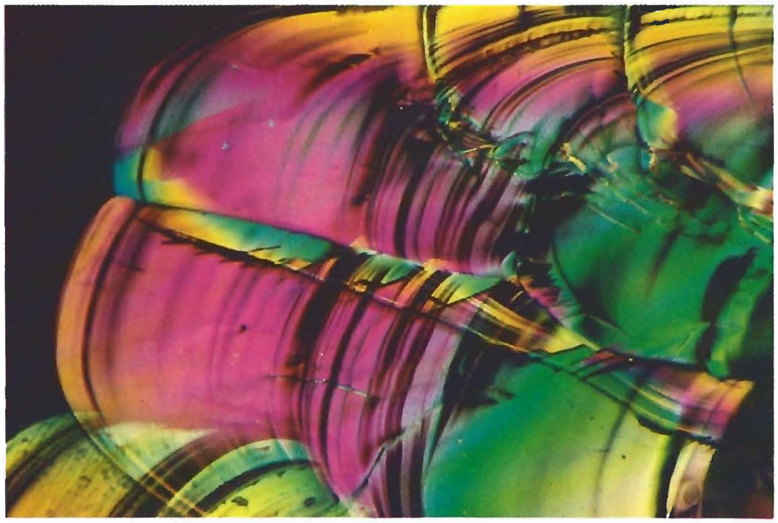


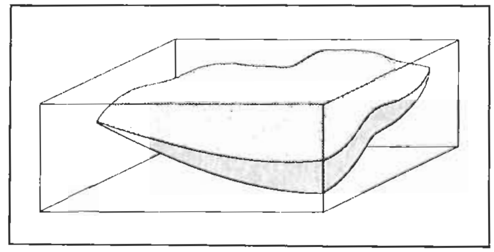


Figure 1. The fracture step in the production of fingerprint inclusions. The example shown here is a conchoidal fracture in a colorless beryl. Oblique illumination, magnified 35×. Drawing by Lisa Joko after Roedder, 1962.

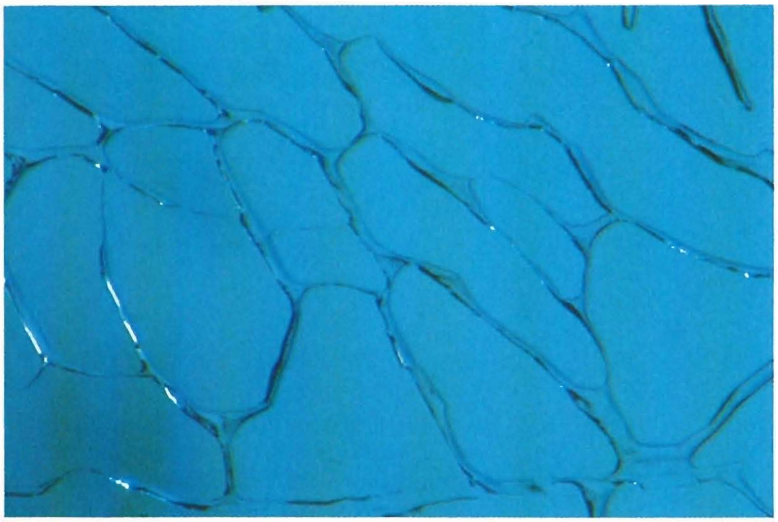
HOW DOES A FINGERPRINT INCLUSION FORM?

The most important clue to understanding induced fingerprints lies in the very name we have given them. In both natural and synthetic gemstones, fingerprint inclusions have been induced, that is, stimulated by internal and external forces acting on the crystallized host material. The mechanism behind natural fingerprints is well documented in the literature (see, for example, Eppler, 1959, 1966; and Roedder, 1962, 1982). This knowledge of how the process occurs in nature is helpful to our understanding of the synthetic production of similar inclusions.

When a crystal is fractured (figure 1), the fracture instantly becomes a vacuum, drawing in whatever surrounds it to alleviate the negative pressure. Capillarity provides further impetus in drawing fluids into the break. If the crystal is even slightly soluble in the fluids surrounding it, repair of the fracture will begin immediately. The more kinetic energy, in the form of heat, supplied during the healing process, the faster the fracture will heal. Heat is (1) generated by sources outside the crystal, such as igneous or metamorphic activity or the heat generated by pressure commonly associated with burial at depth in the earth; and (2) released by the crystal itself, as it seeks to regain crystallographic equilibrium (upset by the increase in potential energy of the fracture zone created by the additional surface area exposed) and return to a lower energy state.

Provided the fractured crystal is in a repair environment, as it starts to heal itself individual atoms or groups of atoms will leave an area of high energy on the fracture surface and redeposit on a surface of lower energy, releasing their heat of crystallization in the process. Molecule by molecule this process continues. Material is dissolved from both the convex and flat portions of the fracture walls and redeposited in the concave areas, gradually trapping small volumes of the repair fluid. Trapping occurs between recrystallized walls, pillars, and columns that have formed, like the adjoining stalagmite and stalactite pillars in a cavern, between the opposing surfaces of the fracture. These fluid islands are often interconnected by a series of fine tubes termed *communication tubes.* So many of these communication tubes may be interconnected that a fishnet-like pattern results. This intermediate stage in the healing process is shown in figure 2.

If healing continues, the communication tubes will gradually thin out in certain areas, gain volume in others, and eventually separate into numerous smaller fluid-filled cavities all lined up along the original path of the pre-existing communication tube. This process is called necking down. Necking down continues throughout the entire original fracture zone, as numerous smaller fluid inclusions are formed from a few larger ones. Ultimately we are left with a uniformly arranged grouping of small fluid-filled voids that occupy the same space as the original fracture. The resulting healed fracture has now taken on the appearance of a "fingerprint," composed of numerous dots or islands. Each of the dots that so geometrically make up the total fingerprint is in fact a separate and distinct fluid inclusion, as portrayed in figure 3. The process of forming a fingerprint inclusion in any crystalline material, synthetic or natural, should be the same. The environment of growth



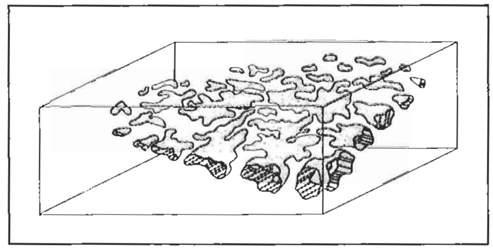


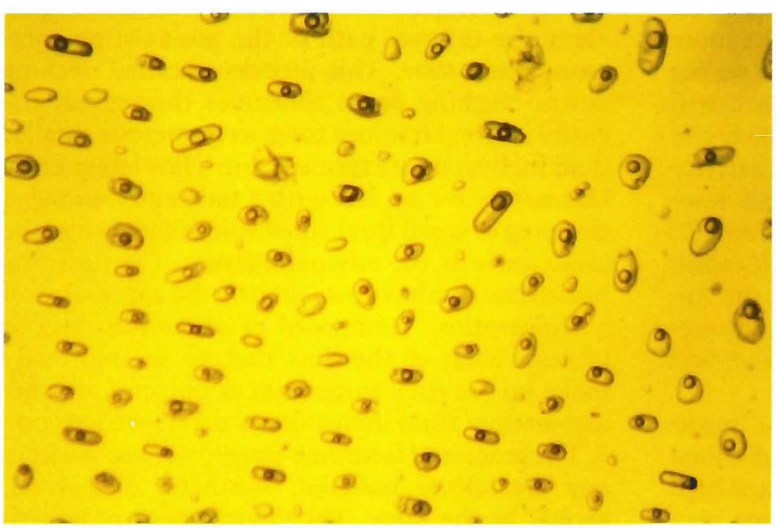
Figure 2. This aquamarine illustrates the intermediate, communication-tube-forming stage in the healing of fractures. Transmitted and oblique illumination, magnified 60×. Drawing by Lisa Joko after Roedder, 1962.

and the fluids used to transport the atoms required to heal a fracture may be different, but the step-by-step process, as shown in figures 1 through 3, does not change.

THE THEORY BEHIND THE FORMATION OF INDUCED FINGERPRINTS IN FLAME-FUSION SYNTHETICS

All gemologists have studied fingerprint inclusions in natural gemstones, as well as in flux and hydrothermally grown synthetics. But gems synthetically grown from a melt by the Verneuil flame-fusion method or by the Czochralski crystal-pulling process are not products of environments that produce crystals with healed fractures. Yet synthetic corundums cut from Verneuil and Czochralski melt crystals containing induced fingerprints have been mistakenly purchased as the more expensive flux synthetic rubies, and even as natural rubies and sapphires. Quite often parcels of corundums will be found to contain some of these synthetics with induced fingerprints. How are fingerprint inclusions placed in such synthetic crystals?

Corundum is corundum regardless of the environment in which it is grown. If we were to take a gem cut from a synthetic melt crystal and thermally shock it to produce fractures, then place it in a synthetic growth environment, such as a flux-growth bath, surrounded by a fluid in which the corundum is at least partially soluble, the induced fractures should, over a period of time, heal themselves, turning into induced fingerprint inclusions. The following experiments were carried out to test this theory.



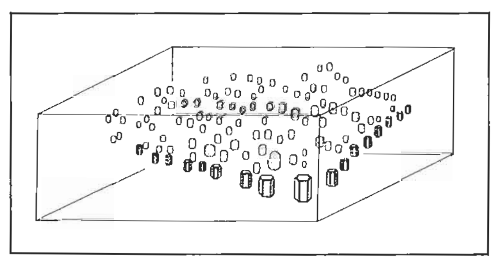


Figure 3. A golden beryl demonstrates the third and final step in fingerprint formation. Now the communication tubes have necked down to form individual fluid inclusion islands. Transmitted light, magnified 100×. Drawing by Lisa Joko after Roedder, 1962.

PRODUCTION OF INDUCED FINGERPRINTS

Verneuil material (figure 4) was selected for testing because it was more readily available than the Czochralski pulled synthetic. A flat, windowlike configuration was chosen for the experimental synthetic rubies because of the ease with which they could be studied under the microscope. Three subjects were heated and then quench-crackled in cold water, resulting in badly fractured slabs. One of these is illustrated in figure 5.

Each of the three fractured synthetic rubies was then designated for a separate experiment as follows: (1) fingerprints induced through flux healing (the most important of the three experiments),

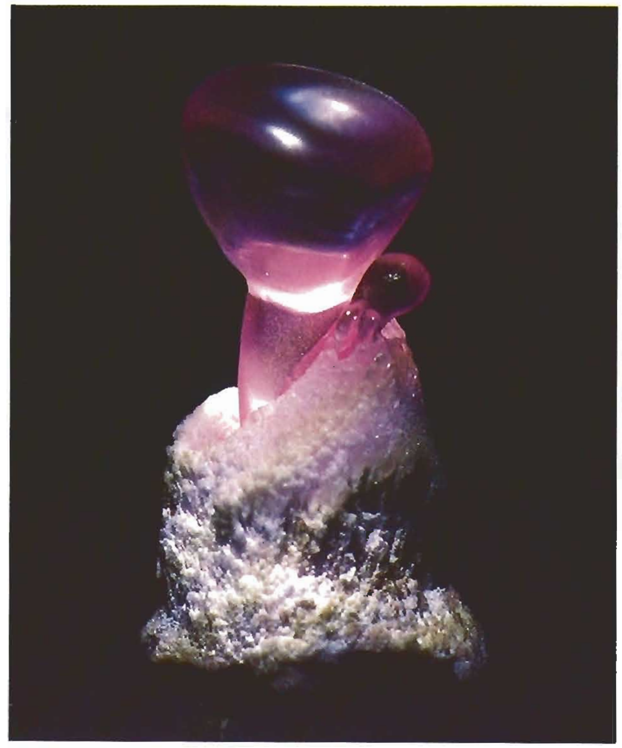


Figure 4. A flame-fusion synthetic ruby boule similar to the material used in the experiments. Photo by Mike Havstad.

(2) fingerprints induced by secondary fusion, and (3) induced chemical dendrites.

Fingerprints Induced Through Flux Healing. After carefully documenting the appearance of the fractured synthetic rubies (again, see figure 5), the author mailed one of the stones to Thomas H. Chatham, president of Chatham Created Gems, in San Francisco. Mr. Chatham had volunteered to place this test subject in a flux-growth environment in an attempt to heal the fractures. A platinum wire affixed to the synthetic ruby slab provided a convenient means of transporting the test subject into and out of the growth chamber. The regrowth time was 42 days (Chatham, personal communication, 1983). The result is shown in figure 6. Note the remnants of the platinum wire at the base of the crystal.

To prepare the regrown mass for study, the author first sawed off the two ends and then had them polished so the thickness of the overgrowth could be observed (figure 7). Already it was apparent that fingerprints were present throughout the overgrown Verneuil subject. Next the crystal was

A SIMPLE EXPERIMENT FOR INDUCING FINGERPRINT INCLUSIONS

There is a simple experiment that anyone can do to test the mechanism of fingerprint formation and actually observe the step-by-step repair process first hand. All that is needed is water, a good supply of a highly water-soluble salt such as alum or sodium chloride (common table salt), at least one transparent single crystal of the chosen salt weighing 2 ct or more that you have studied carefully under the microscope before beginning the experiment, and a source of heat such as a kitchen stove. First, prepare a boiling supersaturated solution of the salt in water. Pour only the liquid portion off, leaving the excess undissolved salt behind. While keeping the solution very hot, supercool your test crystal(s) using a freezer, an alcohol and dry ice solution, or, if you have access to a cryogenically liquefied gas such as liquid nitrogen, use that.

Once the crystal(s) are very cold and the solution is hot, plunge the crystal(s) into the solution. This will cause the crystal(s) first to fracture in the growth-nutrient-rich salt solution and then to begin healing virtually immediately. Now allow the solution to cool gradually. At first some dissolving of the crystal(s) surfaces may be noticed, but quickly the process will reverse itself and the crystal(s) will begin growing. Remove the crystal(s) from the solution periodically and study them under the microscope. Fingerprint inclusions will be observed forming where the fractures once were.

Figure 5. A quench-crackled synthetic ruby window used for flux healing (14.2 × 12.0 × 3.0 mm). Photo by Mike Havstad.

Figure 6. Verneuil synthetic ruby slab after flux regrowth for 42 days (20.7 × 16.8 × 7.6 mm). Note the platinum wire at the base. Photo by Mike Havstad.

completely faceted and the induced fingerprints were studied. Although it was no longer possible to locate the exact configuration of fractures shown in figure 5, any number of induced fingerprints were available for photomicrography. One of these is shown in figure 8 together with the curved striae characteristic of the synthetic material. The author also examined two other large Verneuil boule sections with flux ruby overgrowths prepared by Chatham Created Gems (figure 9). These were found to contain numerous flux regrowth induced fingerprints as well.

Inducing Fingerprints by Secondary Fusion. In the second experiment, to see if heat alone could induce pseudo-healing, one of the prefractured synthetic rubies was placed on a charcoal block and a jeweler's torch was used to melt and recrystallize it several times. Although the overall appearance of the melted mass was not an attractive sight, and on cooling numerous additional unwanted fractures appeared, a few somewhat fingerprint-like inclusions were observed.

Induced Chemical Dendrites. The third and last experiment resulted from observations, by the author, of a flame-fusion ruby that had fractures decorated by a crystalline chemical with a melting point just over 100°C. This low melting point made it possible to melt and recrystallize the contents of the fractures until a desirable, somewhat

Figure 7. Cross-sectional view through the end of the experimental synthetic ruby illustrated in figure 6. Note the outline of the flux ruby layer over the flame-fusion core. Some fingerprints are visible even at this low magnification. Oblique and transmitted light, magnified 5×.

Figure 8. Induced flux fingerprint produced through experimentation in a Verneuil flame-fusion synthetic ruby together with the characteristic curved striae. Shadowing, magnified 45×.

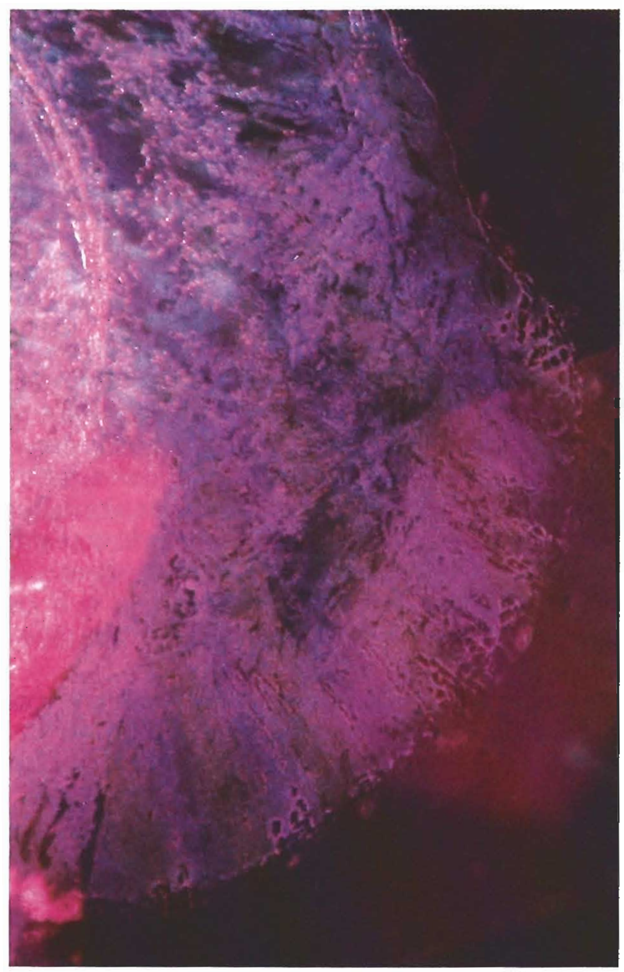
Figure 9. Two Verneuil flame-fusion ruby boule sections with a thin flux ruby overgrowth. Both of these samples contained numerous induced fingerprints. The largest sample is approximately 5 cm long. Photo by Mike Havstad.

natural-appearing dendritic form was obtained.

The low melting point of this unknown chemical suggested that it might be organic in nature. Although the author had access to two organic crystalline compounds with similarly low melting points, acetanilide (melting point, 114°C; boiling point, 304°C) and resorcinol (melting point, 111°C; boiling point, 178°C) acetanilide was chosen for the experiment because of its greater melting point to boiling point spread and, therefore, less crucial temperature control.

The prefractured synthetic ruby was heated over a Bunsen burner to create a vacuum in the fractures through rarefaction. The heated ruby was then quenched in enough premelted acetanilide, contained in a test tube, to completely submerse the ruby. The acetanilide was allowed to crystallize and then was remelted and poured off. The ruby was cleaned and then studied under the microscope. One of the resulting patterns decorating the fractures is reproduced in figure 10.

Figure 10. Acetanilide chemical stain decorating a fracture plane in a Verneuil synthetic ruby. Dark-field and oblique illumination, magnified 80×.



WHO PRODUCES THESE SYNTHETICS WITH INDUCED FINGERPRINTS?

On the basis of the author's observations, it is likely that those synthetic rubies and sapphires that appear in the trade with induced fingerprint inclusions, such as the one shown in figure 11, are probably not healed in a well-controlled flux-growth furnace. Rather, they are probably the sometimes accidental result of clever but often crude heat treatments carried out, both in Thailand and Sri Lanka, on synthetic rubies and various colors of synthetic sapphires in the attempt to dissipate the curved color and growth zoning.

During heat treatment in these countries, it is

Figure 11. Borax (?) induced fingerprint in a flame-fusion synthetic sapphire. Dark-field and oblique illumination, magnified 65×.

common practice to use borax or a borax-based solution, purportedly on the outside of the crucible holding the stones (Abraham, 1982). Any skilled bench jeweler or gemologist knows that corundum becomes soluble in borax at elevated temperatures. That is to say, borax acts as a flux to the corundum; it is an agent capable of promoting quick healing in any fractures—whether placed accidentally or on purpose—that may be present. The mechanism of fracture repair at work in these cases is the same three-step process described earlier for natural stones.

IDENTIFYING GEMS WITH INDUCED FINGERPRINTS

For some time gemologists have known that unless one is very skilled in the study of inclusions, it is no longer possible to say a gemstone is natural merely because it contains fingerprint inclusions. Now, however, with the presence of induced fingerprints in Verneuil and Czochralski synthetic corundum, the question is not only whether the gem is natural or synthetic, but if it is synthetic, is it a more costly flux-grown synthetic or is it an upgraded flame-fusion or pulled synthetic with induced fingerprints?

There is no question that recognizing induced fingerprint inclusions can be a problem. When fingerprint inclusions that reach the surface are the only immediately observable internal characteristics, be suspicious. Check the gem in question for color zoning both by diffused transmitted light and by immersion in methylene iodide. The presence of curved color zoning together with fingerprint inclusions would tell you that the fingerprints have been induced into a synthetic stone. The presence of curved striae in conjunction with fingerprint inclusions in synthetic rubies and some synthetic sapphires is a sure sign that a flame-fusion gem has been doctored. Gas bubbles are another clue to the less expensive synthetics.

With the above clues, treated synthetic rubies and sapphires grown by the Verneuil flame-fusion process are easily spotted. However, gems cut from Czochralski pulled crystals rarely have any recognizable inclusions: quite often they are essentially flawless. Therefore, gems with fingerprint inclusions that are otherwise flawless should be treated with the highest suspicion. Straight or sharply angular growth and color zoning and recognizable included crystals are important clues that a gem is not a melt-grown Verneuil or Czochralski synthetic.

CONCLUSION: WITH THOUGHTS TO THE FUTURE

Of the three experiments conducted, experiment 1, regrowth in a flux environment, performed at the Chatham laboratories in San Francisco, was by far the most successful.

Although the introduction of organic chemical dendrites into pre-existing fractures proved both successful and interesting, the dendrites achieved in experiment 3 in no way resembled or could be mistaken for fingerprint inclusions. Problems could result, however, for gemologists who, in the past, have considered dendrites a sign of natural origin. The attempt to produce fingerprints through secondary fusion met with very little success. Although some inclusions were produced that slightly resembled fingerprints, they were few and far between, and were always accompanied by numerous unrepaired fractures, areas containing large gas bubbles, and zones of translucent cloudy material. Or, in a few words, they were useless as gemstones. However, flux regrowth was, as expected by the author, a complete success. All of the fractures showed fingerprint-healing patterns. Once the regrowth layer was removed, the remaining material, containing the induced fingerprints, could easily have been cut into gemstones.

Possible future applications for this and similar techniques are interesting, to say the least. If the technology that exists to repair fractures today is further refined, we might someday encounter synthetically repaired natural gem materials.

In his excellent book *Gems Made By Man,* Kurt Nassau shows before and after photographs of a Japan-law twin of quartz that was broken and subsequently successfully repaired synthetically by Giorgio Spezia . . . the year was 1908. In more recent work (Shelton and Orville, 1980), synthetic fingerprint inclusions were produced hydrothermally in natural quartz. Imagine in the future if we had the ability to hydrothermally repair a ruby, emerald, or sapphire that had been accidentally fractured during setting, repair, or cutting. Surely a fingerprint inclusion is infinitely more desirable—and infinitely more durable—than a fracture.

In spite of the future potential for good, the logical application today of induced fingerprints is to upgrade less expensive Verneuil flame-fusion and Czochralski pulled synthetic corundums so that they may be sold to the unsuspecting trade either as more costly flux-grown synthetics or even as natural gems.

Because of this, induced fingerprints, whether intentionally or accidentally produced, represent a type of treatment that must be disclosed. A treatment of this type should concern not only the gemologist and the jeweler, but also those involved in the flux growth of synthetic gems as well, because the presence of such a treated material on the market could seriously undermine the sale of their products. It is only through mutual cooperation between gemologists and crystal growers that such a problem can be dealt with.

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