Kirk-Othmer Encyclopedia of Chemical Technology.

GEMSTONE MATERIALS

There are three types of gemstone materials as defined by the U.S. Federal Trade Commission (1): (*1*) natural gemstones are found in nature and at most are enhanced (see Gemstones, gemstone treatment); (*2*) imitation or simulated, fake, faux, etc, material resembles the natural material in appearance only and is frequently only colored glass or even plastic; and (*3*) synthetic material is the exact duplicate of the natural material, having the same chemical composition, optical properties, etc, as the natural, but made in the laboratory (2, 3). Moreover, the word gem cannot be used for synthetic gemstone material. The synthetic equivalent of a natural material may, however, be used as an imitation of another, eg, synthetic cubic zirconia is widely used as a diamond imitation.

The first successful synthetic gemstone material was the 1885 Geneva ruby of unknown origin, misleadingly called reconstructed at the time and sold as natural (2). This was soon followed by Verneuil's flame-fusion synthetic ruby [12174-49-1] (4), which became an immediate commercial success. Early synthetics were usually the result of mineralogical studies or focused attempts to duplicate natural gemstones. More recently these materials have been by-products of technology-oriented studies, eg, the search for laser crystals (see Lasers). The historic availability of gemstone materials is summarized in Table 1. Most of these materials are made by crystal growing techniques (2, 5, 6) (see Crystallization). The U.S. patent literature has been summarized (7). There is also a large Japanese patent literature.

Synthetic gemstone materials often have multiple uses. Synthetic ruby and colorless sapphire are used for watch bearings, unscratchable watch crystals, and bar-code reader windows. Synthetic quartz oscillators are used for precision time-keeping, citizen's band radio (CB) crystals, and filters. Synthetic ruby, emerald, and garnets are used for masers and lasers (qv).

In the gemstone jewelry market, synthetics provide a less expensive alternative to natural gemstones, but of a better quality than that available in costume jewelry. In general, a synthetic should be available for no more than 10% of the cost of equivalent-quality natural gemstone to be commercially viable. Synthetics are frequently divided into three groups: (*1*) luxury synthetics, involving slow and difficult growth processes, produced in small quantities for a price-restricted market; (*2*) intermediates; and (*3*) low cost synthetics, produced on a large scale. In 1989 more than 10^9 carats (200 t) of both synthetic ruby and cubic zirconia [1314-23-4] were produced (3). Price ranges for these groups are summarized in Table 2.

Diamond simulants are usually included under synthetics, even though not all of these have been synthetic gemstone materials, such as the garnets. Many trade names have been used for each of these imitations (see Carbon–diamond, synthetic).

Distinguishing between natural and synthetic gemstone materials is one of the tasks of the trained gemologist. Whereas difficult at times, the importance derives from the huge differences in commercial value. For example, a natural flawless ruby could be valued at $20,000 a carat ($100,000/g); a synthetic pulled from the melt by the Czochralski technique goes for $2 a carat ($10/g). Gemology is taught by the Gemological Institute of America of Santa Monica, California, and New York, the Gemmological Association of Great Britain of London, and elsewhere. Textbooks are also available (8–10).

Table 1. Availability of Synthetic Gemstone Materials

Year	Synthetic gemstone	CAS Registry Number	Manufacturing technique
1885	ruby	[12174-49-1]	Geneva
1905	ruby		Verneuil
1910	sapphire	[1317-82-4]	Verneuil
1910	spinel	[1302-67-6]	Verneuil
1947	star ruby and sapphire		Verneuil
1947	rutile	[1317-80-2]	Verneuil
1950	emerald	[1302-52-9]	flux
1950	quartz	[14808-60-7]	hydrothermal
1955	strontium titanate	[12060-59-2]	Verneuil
1965	ruby		flux
1965	emerald		hydrothermal
1970	diamond	[778-40-3][a]	high pressure
1973	alexandrite	[12252-02-7]	flux
1974	opal	[14639-88-4]	complex process
1974	citrine	[14832-92-9]	hydrothermal
1975	amethyst	[14832-91-8]	hydrothermal
1976	cubic zirconia	[1314-23-4]	skull melting
1978	sapphire		flux
1980	jadeite	[12003-54-2][a]	high pressure

[a] Grown for other purposes or experimental production.

Table 2. Price Ranges of Synthetic Gemstone Materials

	Cost, \$/g[a]	
Material	Rough	Faceted[b]
Luxury		
emerald,[c] black opal, alexandrite, flux ruby,[c] sapphires[c]	50 to <500	500 to <5000
Intermediate		
star ruby[d] and sapphires,[d] white opal, amethyst	5 to <50	50 to <500
Low cost		
cubic zirconia, Verneuil ruby, sapphires, spinel, colorless and citrine quartz	0.50 to <2.50	5 to <50

[a] To convert \$/g to \$/carat, divide by 5.00.

[b] Usually at least five times the cost of the rough because cutting recovery is only 25% or even less. The cutting cost may become more important than the materials cost in small faceted stones.

[c] Flux crystal clusters sell for about the same price as faceting-quality rough.

[d] Star material has generally not been available as rough.

1. Properties

The important properties are those of importance in natural gemstones. First is hardness (qv), H. A value of 7 or greater on Mohs' scale is desirable to avoid scratches from the quartz ($H = 7$) sand present in dust. Next is color (qv) or a total lack of color, as in diamond and its simulants. A high refractive index (RI) permits the return by total internal reflection of most of the light falling onto a well-cut gemstone, giving brilliance, and a high dispersion (DISP) spreads the internally reflected light into spectral colors, resulting in fire. A large birefringence is undesirable, because that produces a doubling of facet junctions seen through the top of the stone.

Table 3. Properties of Diamond and Synthetic Gemstone Materials[a]

Material	CAS Registry Number	Composition	RI	DISP	H	Specific gravity	Year[b]	Disadvantages
spinel[c]	[1302-67-6]	$MgAl_2O_4$	1.73	0.02	8	3.64	1910	much less brilliant
sapphire, colorless	[1317-82-4]	Al_2O_3	1.77	0.018	9	4.00	1920	much less brilliant
rutile	[13463-67-7]	TiO_2	2.8	0.33	6	4.26	1947	yellowish, soft, birefringent, excessive fire
strontium titanate (tausonite)	[12060-59-2]	$SrTiO_3$	2.41	0.19	$5\frac{1}{2}$	5.13	1955	soft, excessive fire
YAG garnet	[12005-21-9]	$Y_3Al_5O_{12}$	1.83	0.028	$8\frac{1}{4}$	4.55	1968	less brilliant
GGG garnet	[12024-36-1]	$Gd_3Ga_5O_{12}$	1.97	0.045	7	7.02	1974	slightly less brilliant, heavy
cubic zirconia[c]	[1314-23-4]	$9ZrO_2 \cdot Y_2O_3$	2.16	0.060	$8\frac{1}{4}$	6.0	1976	somewhat heavy
diamond	[7782-40-3]	C	2.42	0.044	10	3.52		

[a] RI = refractive index; DISP = dispersion; H = hardness on Mohs′ scale.

[b] Represents the start of widespread use.

[c] Composition property values can vary from batch to batch.

Diamond is supreme among natural gemstones in H, RI, and DISP. Table 3 shows the steady improvement in the sequence of diamond imitations, the aim being to produce a colorless, adequately hard material having closely matching optical properties. The introduction of synthetic cubic zirconia in 1976 brought about a sufficiently close match.

Several gemstone species occur in various colors, depending on the presence of impurities or irradiation-induced color centers. Examples are the beryl, corundum, and quartz families. Quartz has poor optical properties ($RI = 1.55$, $DISP = 0.013$), but becomes of gemological interest when it exhibits attractive colors. Any material can have its color modified by the addition of various impurities: synthetic ruby, sapphires, and spinel are produced commercially in over 100 colors (2). Synthetic cubic zirconia has been made in essentially all colors of the spectrum (11), but only the colorless diamond imitation is produced commercially in any quantity.

2. Manufacture

The most frequently used techniques for the commercial manufacture of synthetic gemstone materials are summarized in Table 4. More details on these can be found in various texts (2, 5–7). An overview including the various manufacturers is also available (3). Only rarely used for synthetics are such alternative growth techniques as the Bridgman technique of solidification in a crucible and the float zone technique, both involving growth from the melt (2, 5–7)

The easiest and most rapid crystal growth techniques employ crystallization from the melt. Some materials are incongruently melting, ie, they decompose below their melting point, in which case solution techniques must be used, as for emerald. The same applies to those instances where there is a strong tendency for glass formation from the melt, as in the quartz family. Diamond is a special case in that it is only thermodynamically stable at high pressure. Whereas low pressure techniques have been successful for producing very thin single-crystal films or for bulk polycrystalline material, the growth of bulk single-crystal synthetic diamond has not been achieved (12).

Table 4. Techniques for Commercial Gemstone Material Synthesis

Technique	Material
	Crystal growth from the melt
Verneuil (flame fusion)	ruby, sapphires, and stars; spinel; rutile; strontium titanate
Czochralski (pulling)	ruby and sapphire; alexandrite; garnets; YAG and GGG
float zone	ruby and sapphire; alexandrite
skull melting	cubic zirconia
	Crystal growth from solution
flux	alexandrite; emerald; ruby and sapphire; spinel
hydrothermal	colorless, amethyst, citrine, and smoky quartz; emerald; ruby and sapphire
high pressure	diamond;[a] jadeite[a]
	Other techniques
complex chemical[b]	opal

[a] Grown for other purposes or experimental production.

[b] See text for a description of this technique.

2.1. Crystal Growth from the Melt

2.1.1. The Verneuil Technique.

The Verneuil technique is also known as flame-fusion. A very pure feed powder is first made by chemical decomposition. For the growth of corundum, ammonium alum [7785-25-0], $NH_4Al/(SO_4)_2\cdot 12H_2O$, is recrystallized from water containing added color-causing transition-metal impurities, such as Cr for ruby, again in the form of an alum. Decomposition at about 1200°C forms Al_2O_3 powder that is very fine and free-flowing. This feed powder is now sprinkled through a downward-pointing hydrogen-oxygen torch, as shown in Figure 1. The particles melt on impinging, forming a molten cap about 20 μm thick on the upper surface of the growing crystal boule, which is slowly lowered. Because of the small melt volume, even small irregularities in the feed rate and flame temperature produce irregular solidification, resulting in curved color striations and occasional incompletely melted grains or gas bubbles. The growth rate is one to several cm/h and boules over 4 cm in diameter and 40 cm in length have been grown.

For the Verneuil growth of rutile and strontium titanate it is necessary to maintain strongly oxidizing conditions to prevent excessive reduction of Ti^{4+} to Ti^{3+}. This is achieved by adding a third outer tube carrying extra oxygen to the Verneuil torch (Fig. 1) in the tricone modification. Annealing in O_2 at about 1100°C is subsequently used to achieve full oxidation.

2.2. The Czochralski Technique

Pulling from the melt is known as the Czochralski technique. Purified material is held just above the melting point in a crucible, usually of Pt or Ir, most often powered by radio-frequency induction heating coupled into the wall of the crucible. The temperature is controlled by a thermocouple or a radiation pyrometer. A rotating seed crystal is touched to the melt surface and is slowly withdrawn as the molten material solidifies onto the seed. Temperature control is used to widen the crystal to the desired diameter. A typical rotation rate is 30 rpm and a typical withdrawal rate, 1–3 cm/h. Very large, eg, kilogram-sized crystals can be grown.

2.3. Skull Melting

Skull melting, or the cold crucible technique, is used for high melting point materials. Zirconia [1314-23-4], ZrO_2, has such a high (about 2750°C) melting point, that no crucible material is available that does not react with the melt. In skull melting (2, 5, 13) a container of closely spaced water-cooled copper fingers, as shown in

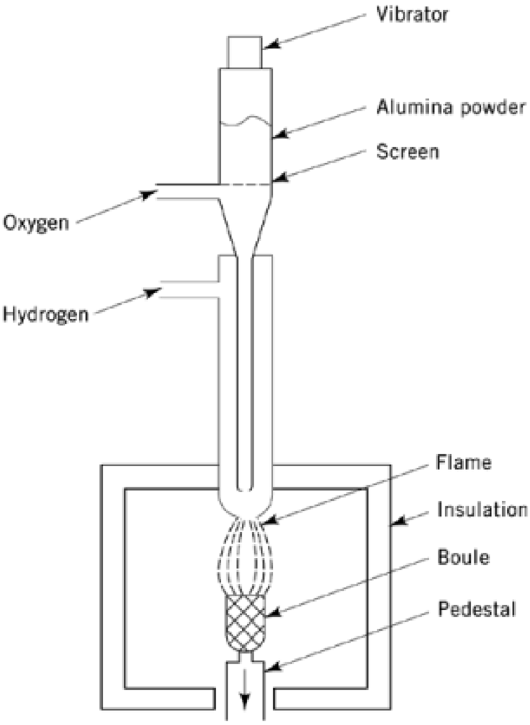

Fig. 1. The Verneuil technique, or flame-fusion growth, as used for synthetic ruby and sapphire.

Figure 2, is filled with powder of the desired composition, usually about 90% ZrO_2 and 10% yttria [1314-36-9], Y_2O_3. A piece of Zr metal is added. Radio-frequency power from the coil readily passes through the narrow gaps between the fingers and melts the metal, which in turn melts the adjacent oxides, which then couple directly to the coil; the metal oxidizes in the air. All the contents of the container melt except for a relatively thin crust adjacent to the cold copper fingers, so that the melt is only in contact with a cold crucible of its own composition. On slowly lowering the container from out of the coil, random nucleation begins at the bottom and produces irregularly shaped crystals, which can be over 10 cm across.

2.4. Crystal Growth from Solution

2.4.1. Flux Growth

This procedure is called growth from the fluxed melt in the United Kingdom. A flux in this context is a high melting inorganic solvent containing substances such as PbO [1317-36-8], PbF_2 [7783-46-2], B_2O_3 [1303-86-2], etc. For example, for the growth of ruby, 3.6 kg Al_2O_3 and 30 g Cr_2O_3 [1308-38-9] are mixed with 22 kg of PbF_2 and melted in a 20-cm diameter and high, heavy-walled platinum crucible at about 1300°C and mixed by a period of rotation via the supporting pedestal as in Figure 3. A controlled, slow cooling to about 1000°C over a several-week period exceeds the solubility of ruby in the flux, and crystals self-nucleate and grow to several centimeters across on the slightly cooler crucible bottom. The unwanted flux may be drained off through the hollow pedestal. This is flux growth by slow cooling.

Flux reaction growth is one of several modifications used for the growth of emerald. Here one of several fluxes, possibly composed of Li_2O [12057-24-8] and MoO_3 [1313-27-5], is used with the constituents of emerald,

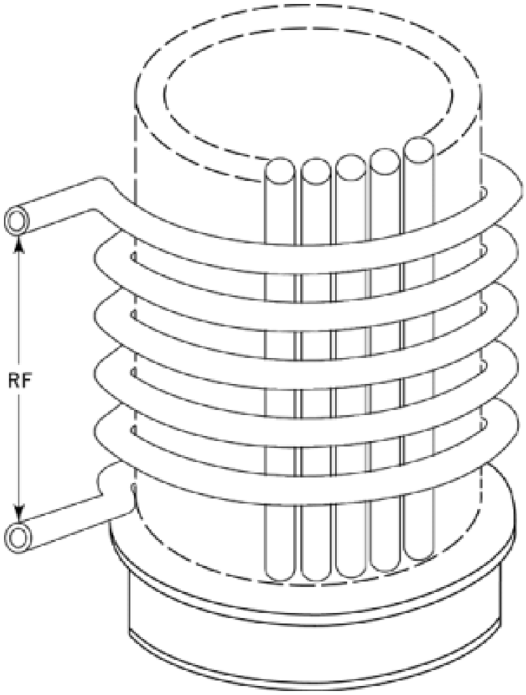

Fig. 2. Schematic drawing of one form of skull-melting apparatus; only some of the fingers are shown (2). RF=radio frequency.

ie, SiO_2, Al_2O_3, BeO [12269-78-2], and Cr_2O_3, diffusing toward thin seed plates in the central growth region, as shown in Figure 4.

2.5. Hydrothermal Growth

Hydrothermal growth is used in the form of solution transport for the growth of synthetic quartz. Crushed natural quartz is placed into the lower part of a high pressure steel vessel, called a bomb, and thin seed plates are located in the upper region, as seen in Figure 5. The vessel is filled, for example, to 80% capacity with a 4% NaOH [1310-73-2] solution; the NaOH acts as a mineralizer to increase the solubility of SiO_2. When the closed vessel is heated to 390°C bottom and 330°C top, the resulting pressure is 170 MPa (25,000 psi) and a single fluid fills the vessel. It dissolves nutrient at the bottom and flows by convection, controlled by a baffle, to the upper region, where growth occurs at the lower temperature. A typical 4 cm × 152 cm × 1 mm thick seed plate grows to a 5-cm thickness in about 3 weeks in a 3-m long production vessel.

For emerald, a hydrothermal reaction arrangement is used, analogous to that of Figure 4. Here the mineralizer is strongly acid, containing 8*N* NH_4Cl [12125-02-9] and HCl [7647-01-1] to give pH between 0.2 and 4.5.

2.6. High Pressure Growth of Diamond

Ni dissolves graphite [7782-42-5] or diamond at about 7 GPa (1×10^6 psi) and 1800°C and crystallizes diamond in a temperature gradient onto a cooler seed plate (2, 5–7, 14). To reach such high pressures, a massive support apparatus using two pistons and a belt made of tungsten carbide [12070-13-2] bonded with cobalt [7440-48-4] and supported by shrunk-on steel rings is used in a uniaxial hydraulic press. In an alternative geometry, four

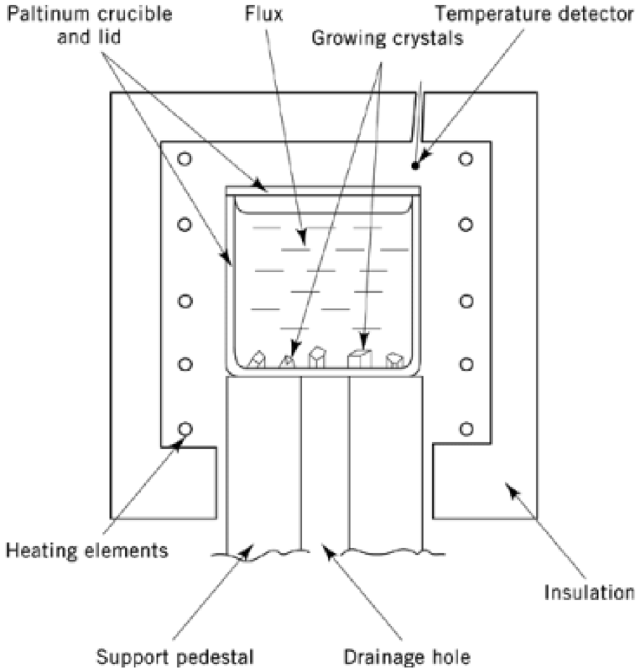

Fig. 3. Crucible inside a furnace as used for growth from the flux (2).

pistons are arranged to form a tetrahedral space. Pyrophillite [12269-78-2] is used as an insulator and as a nonextruding gasket. Metals other than Ni and some alloys can also be used as solvents.

3. Materials

3.1. Alexandrite

Alexandrite [12252-02-7] is chrysoberyl [1304-50-3], $BeAl_2O_4$, when pure. The Cr-containing alexandrite form has a psychooptic color change, appearing blue-to-green when viewed in daylight or fluorescent-tube lighting, and red in the light from a candle or an incandescent light bulb. It is grown as a luxury synthetic from the flux and also more recently by the Czochralski technique.

3.2. Beryl

Beryl [1302-52-9], $Be_3Al_2Si_6O_{18}$, is called aquamarine [1327-51-1] when pale green or blue from inclusion of Fe emerald [12415-33-7] when dark green from Cr or at times V, and morganite or red beryl when pink or red, respectively, from Mn. Only the synthetic emerald is in commercial production, although the other colors can also be grown. Both the flux and hydrothermal techniques are used to grow this luxury synthetic.

3.3. Corundum

Crystalline Al_2O_3, corundum [1302-74-5], is called ruby [12174-49-1] when colored red by about 1% Cr, and sapphire [1317-82-4] for other colors, particularly when blue from charge transfer (2, 11) between about 0.01%

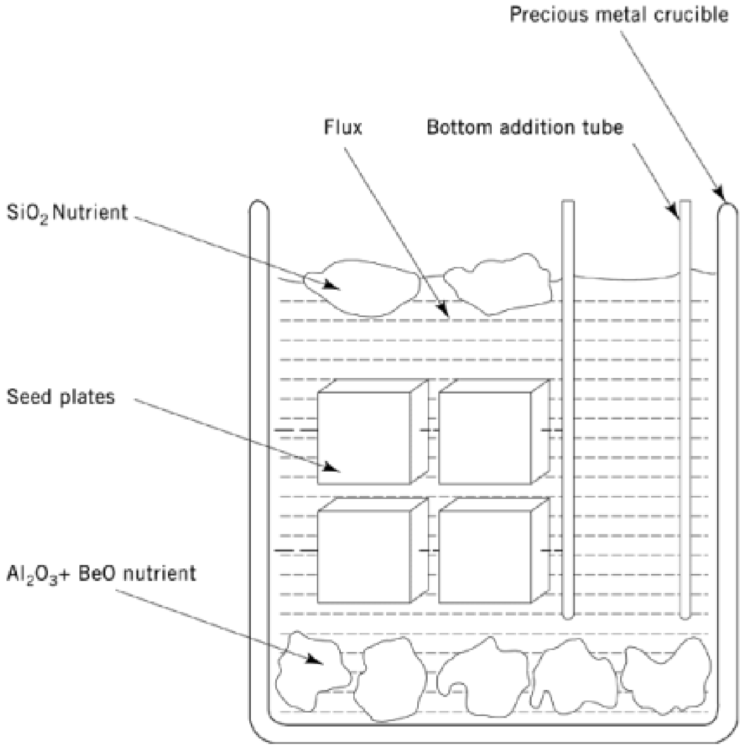

Fig. 4. Schematic diagram of one of the arrangements for the flux-reaction growth of synthetic emerald (2).

each of Fe^{2+} and Ti^{4+}. A wide range of other colors can be grown, including colorless (pure), yellow (containing Ni), orange ($Ni + Cr$), green (Co), alexandrite-like green/purple (V), and so on. Synthetic ruby is grown (*1*) for high optical-quality laser use by the Czochralski technique; (*2*) together with many colors of sapphire as a low cost synthetic by the Verneuil technique; and (*3*) together with blue sapphire as luxury synthetics by the flux technique. The hydrothermal technique has also been used. The colorless material has been used as a poor diamond imitation (Table 3), called Brillite, Diamondite, Thrilliant, etc. Synthetic ruby and variously colored sapphires are used in class rings as imitations of various gemstones, as well as for other jewelry uses.

The addition to Al_2O_3 of up to 1% TiO_2 [1317-80-2] results in the growth of clear crystals containing a solid solution. On annealing at about 1200°C, the exsolution of TiO_2 needles results in the formation of a star effect, as shown in Figure 6.

3.4. Cubic Zirconia

As of this writing, cubic zirconia [1314-23-4], ZrO_2, is the best diamond imitation available (Table 3). It is marketed under such names as CZ, Cerene, Cubic zirconium, Diamonair II, Diamonique III, Fianite, etc, and grown by the skull melting technique (2, 5, 13). Pure ZrO_2 is monoclinic, changing through tetragonal to cubic at 2300°C. The addition of 10% yttria [1314-36-9] or other oxides such as calcia [1305-78-8] stabilizes the cubic form at room temperature. This material can also be made in almost any color. The pink color given by Co changes to a deep sapphire-like blue if the stabilizer concentration is raised to about 30%. The addition of any yellow-causing impurity can then produce an emerald-like green. Several techniques, including the

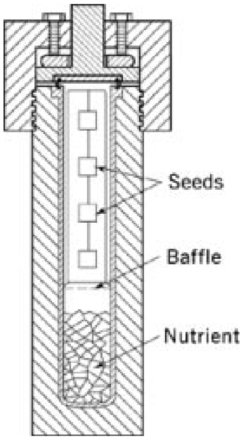

Fig. 5. A small silver-lined hydrothermal growth vessel, 35-cm long, as used for synthetic quartz.(Courtesy of Bell Laboratories.)

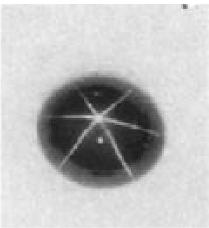

Fig. 6. Synthetic star ruby made by Linde (2).

thermal tester designed specifically for this purpose which recognizes the uniquely high thermal conductivity of diamond, provide ready distinctions between cubic zirconia and diamond (2, 8–10).

3.5. Diamond

The synthesis by a high pressure process of single-crystal diamond [7782-40-3] large enough for gemstone use was revealed by the General Electric Co. in 1971 (14). Commercial production was instituted (15) at DeBeers, Sumitomo, and others to produce gem-quality synthetic diamond crystals weighing over 11 carats (2.2 gm). The yellow color (containing N) is grown much more easily than colorless (pure) and blue (B). None of these is likely to be viable for use in jewelry in the near future. The low pressure deposition techniques used for growing diamond films (12) are not able to produce bulk single-crystal material.

3.6. Garnets

Both YAG, yttrium aluminum garnet [12005-21-9], $Y_3Al_5O_{12}$, and GGG, gadolinium gallium garnet [12024-36-1], $Gd_3Ga_5O_{12}$, have the garnet structure and were used at one time as diamond imitations (Table 3) under trade names such as Diamonaire, Diamonique, Diamonite, Kimberly, Triamond, and YAlG for YAG and Diamonique II, Galliant, and Triple G for GGG. These have been supplanted by cubic zirconia.

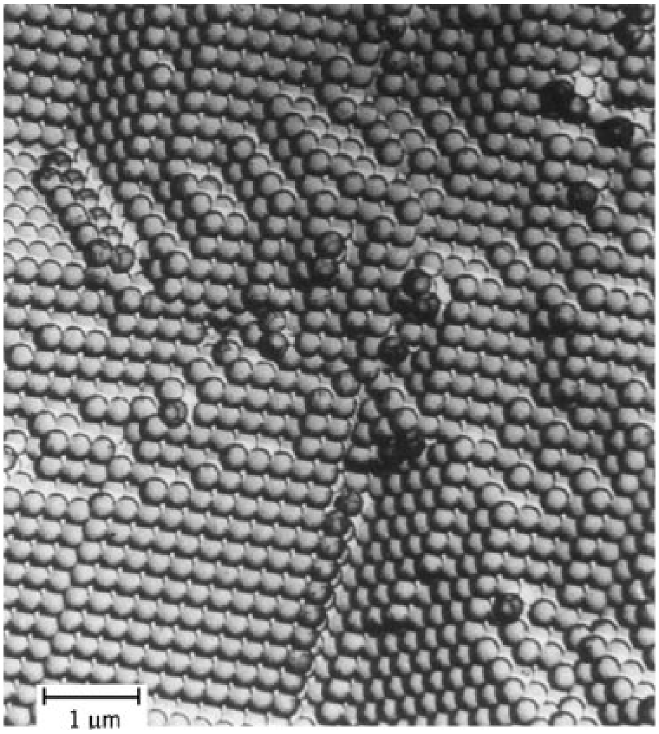

Fig. 7. Electron microscope view of Gilson synthetic opal; each sphere is $\frac{1}{4}$ μm in diameter.(Courtesy of Ets. Ceramiques Pierre Gilson.)

3.7. Opal

Opal [14639-88-4] is the only commercial synthetic gemstone material that is not a single crystal. As shown in Figure 7, it consists of a three-dimensional diffraction grating of geometrically aligned spheres of $SiO_2{\cdot}xH_2O$, where x is usually $<10\%$. The spheres have uniform size between 0.2 and 0.7 μm and are cemented together with a similar composition having a different x. A chemical precipitation process is used to make the spheres, followed by a lengthy settling process and some type of compaction step (2, 7).

3.8. Quartz

When colorless, quartz [14808-60-7] is also known as rock crystal. When irradiated, it becomes smoky from a color center associated with a ubiquitous Al impurity at about the 0.01% level. The name citrine [14832-92-9] is used when quartz is colored by Fe, and irradiation of this can produce the purple-colored amethyst [14832-91-8] under certain circumstances (2). Although not significantly lower priced than the natural materials, synthetic citrine and amethyst are used in jewelry because of the ability to provide matched sets of stones from large, up to 7-kg, hydrothermally grown crystals.

3.9. Rutile

Rutile [1317-80-2], a form of TiO_2, was at one time used as a rather poor diamond imitation under trade names such as Kenya stone, Rutania, Titanic, and Ultimate. Small amounts of Al_2O_3 or Ga_2O_3 lighten the yellow color.

Related is strontium titanate [12060-59-2], $SrTiO_3$, now more properly called synthetic tausonite. This latter was a great improvement over TiO_2 as a diamond imitation under trade names such as Brilliante, Diagem, Diamontina, Fabulite, and Wellington, and is still sometimes so used. The properties of both these materials, grown by the Verneuil technique, are listed in Table 3.

3.10. Spinel

Although the composition of natural spinel [1302-67-6] is $MgAl_2O_4$, crystal growth is much eased by growing Al_2O_3-rich material in the solid solution region. Colorless (pure), blue (Co), and other colored synthetic spinels made by the Verneuil process are widely seen in class rings and in other jewelry uses, where the blue is often mislabeled as synthetic sapphire. Flux growth has also been used for stoichiometric spinel. Colorless synthetic spinel was once used as a poor diamond imitation (Table 3) under trade names such as alumag, radient, and strongite.

3.11. Other Synthetic Materials

Many other natural gemstone materials have been duplicated in the laboratory on an experimental basis, often only in small sizes. Examples include tourmaline [1317-93-7], topaz [1302-59-6], and zircon [1490-68-2]. Of some potential is synthetic jadeite [12003-54-2], one of the two forms of jade. This crystallizes under medium pressure in polycrystalline form from an $NaAlSi_2O_6$ glass (qv) and can be colored green by Fe or lavender by Mn (16). Many gemstone-like materials have been grown for technological purposes and such material is sometimes faceted.

3.12. Discredited Synthetics

There are several materials that have in the past been considered to be synthetics, but were found on closer examination not to deserve such a designation, being merely imitations. Examples include imitation coral, lapis lazuli, and turquoise, all made by ceramic processes. This same point has been raised (17) with respect to synthetic opal, which does contain some substances not present in natural opal and somewhat less water. However, the composition of natural opal is quite variable and is usually intermixed with significant amounts of rock-derived materials; hence the synthetic designation is usually retained.

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