

GEMSTONE TREATMENT

Color and clarity are two of the attributes that give gemstones used in jewelry value. Gemstones deficient in either color or clarity can be enhanced (1). Almost worthless material can at times be converted into valuable-appearing gemstones. An estimated two-thirds of all colored gemstones used in jewelry have been treated. Accordingly, the identification of the use of treatments and the disclosure of enhancements to the purchaser are important. Table 1 lists the materials discussed herein.

Some treatments are practiced so widely that untreated material is essentially unknown in the jewelry trade. The heating of pale Fe-containing chalcedony to produce red-brown carnelian is one of these. Another example involves turquoise where the treated material is far superior in color stability. Such treatments have traditionally not been disclosed. Almost all blue sapphire on the market has been heat treated, but it is not possible to distinguish whether it was near-colorless corundum containing Fe and Ti before treatment, or whether it had already been blue and was only treated in an attempt at marginal improvement. The irradiation of colorless topaz to produce a blue color more intense than any occurring naturally is, however, self-evident, and treatments used on diamond are always disclosed.

The stability of a particular treatment is also important. The enhancement should survive during normal wear or display conditions. Whereas all the enhancements from heat treatments are stable, some produced by irradiation are not. There are also surface coatings which wear off, oilings which dry out, etc.

It is convenient to discuss enhancements in three groups: heat treatments, irradiations, and other treatments (1). Several types of treatments are at least 3000 years old; others, such as the filling of cracks with glass, arose only in the late 1980s.

1. Heat Treatments

The most commonly seen of the gemstones that have been enhanced by heat treatment are listed in Table 2. Parameters for specifying the conditions for heat treatment of a gemstone material include the maximum temperature reached and the time for which the maximum temperature is sustained; the rate of heating to temperature, the rate of cooling down from temperature, and any holding stages while heating and cooling; the chemistry and pressure of the atmosphere; and any material in contact with the gemstone. Exact conditions for heat treatments vary widely according to the natural materials used.

1.1. The Effects of Heat

Heat can have many different effects, but to avoid cracking, gemstones are often preformed or even fully polished to eliminate any existing cracks and imperfections. Gemstones are then usually buried in an inert powder or placed into nested crucibles to avoid thermal shock. However, in some instances cracking may be desired as in amber or in the crackling of quartz by heating and dropping it into a dye solution. The stone resulting from the latter appears to be uniformly colored and may be used in imitation of another gemstone such as

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Table 1. Gemstone Materials Discussed

Material	CAS Registry Number	Molecular formula
agate	[15723-40-7]	SiO ₂
amber	[8002-67-3]	
amethyst	[14832-91-8]	SiO ₂ + Fe
aquamarine	[1327-51-1]	Be ₃ Al ₂ Si ₆ O ₁₈ + Fe
azurite	[1319-45-5]	Cu ₃ (CO ₃) ₂ (OH) ₂
beryl	[1302-52-9]	Be ₃ Al ₂ Si ₆ O ₁₈
carnelian		
chalcedony	[14639-89-5]	SiO ₂
citrine	[14832-92-9]	SiO ₂ + Fe
coral		
corundum	[1302-74-5]	Al ₂ O ₃
diamond	[7782-40-3]	C
emerald	[12415-33-7]	Be ₃ Al ₂ Si ₆ O ₁₈ + Cr ³⁺
fluorite	[7789-75-5]	CaF ₂
jade (jadeite)	[12003-54-2]	Na(Al,Fe)Si ₂ O ₆
jade (nephrite)	[12172-67-7]	Ca ₂ (Mg,Fe) ₅ Si ₈ O ₂₂ (OH) ₂
lapis lazuli	[1302-85-8]	complex and variable
malachite	[569-64-2]	Cu ₂ (CO ₃)(OH) ₂
marble	[471-34-1]	CaCO ₃
opal	[14639-88-4]	SiO ₂ ·xH ₂ O
pearl		
quartz	[14808-60-7]	SiO ₂
ruby	[12174-49-1]	Al ₂ O ₃ + Cr ³⁺
sapphire	[1317-82-4]	Al ₂ O ₃
spodumene	[1302-37-0]	LiAlSi ₂ O ₆
topaz	[1302-59-6]	Al ₂ SiO ₄ (F,OH) ₂
tortoiseshell		
tourmaline	[1317-93-7]	complex and variable
turquoise	[1319-32-0]	Al ₆ Cu(PO ₄) ₄ (OH) ₈ ·4H ₂ O
zircon	[1490-68-2]	ZrSiO ₄
zoisite	[1319-42-4]	Ca ₂ Al ₃ Si ₃ O ₁₂ (OH)

ruby or emerald. A cracking process has also been used on synthetic corundum to introduce natural-appearing fingerprint inclusions (2).

Heat is used to darken amber, ivory, and jade to simulate age. Pieces of amber and tortoiseshell can be reconstructed, ie, joined under heat and moderate pressure. By careful heating in oil, milky amber can be clarified when the gas and water within small bubbles diffuse out of the stone. If heating is rapid, the attractive sun-spangle cracking shown in Figure 1 results.

The destruction of color centers (1, 3) by heating can result in bleaching or fading. Examples are brown or blue topaz, red tourmaline, smoky quartz, and some yellow sapphire. In other instances there may be a color change as when amethyst turns into yellow citrine, or when the heating of a brown topaz reveals the presence of a previously hidden Cr-derived color in a pinked topaz. These changes can usually be reversed by an irradiation treatment.

The yellow color center in some sapphire, designated Type 1 and usually from Sri Lanka, is quite unusual. This color fades gradually over the range 60–600°C. Irradiation restores and intensifies the color to a vivid orange. However, daylight restores the stable color, either from the heat-faded extreme or from the irradiated extreme (1, 4). A color change derived from hydration in chalcedony involves the irreversible change from a yellow iron oxide or hydroxide or hydrate such as goethite [1310-14-1], FeO(OH), to brown-to-red hematite [1317-60-8], Fe₂O₃.



Fig. 1. An amber bead containing sun spangles produced by a special heat treatment. (Courtesy of Gemological Institute of America.)

The most widely practiced enhancement is the heat treatment to produce a blue sapphire. This occurs from charge transfer involving about 0.01% each of Fe^{2+} and Ti^{4+} in Al_2O_3 , corundum (1, 3). Some pale corundum contains these impurities, but Fe is present mostly as Fe^{3+} . The desired redox balance can be reached by an appropriate heat treatment in a reducing atmosphere. An oxidizing atmosphere is used if the material is too dark from too much Fe^{2+} or reduced Ti. Temperatures in the 1800°C range for some hours are typically

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Table 2. Gemstones Enhanced by Heating

Material	Change ^a	Product	Use ^b
amber	clarified, sun-spangled	amber	F
amber	reconstructed, aged	amber	R
beryl	green to blue	aquamarine	W
chalcedony	pale to red-brown or red	carnelian, agate, tiger's eye, etc	W
corundum	develop, intensify, or lighten blue	blue sapphire	W
corundum	develop or intensify yellow	yellow sapphire	W
corundum (ruby)	remove off-shades	ruby	F
corundum (ruby, sapphires)	remove silk, remove or develop asterism	starting material	W
corundum	diffuse in color or asterism	ruby, sapphires	R
diamond	change color after irradiation	starting material	R
quartz	amethyst to yellow citrine	starting material	W
quartz	crackled and dyed	various colors	R
zircon	brown to colorless or blue	starting material	W
zoisite	brown to deep purple-blue	starting material	W

^aAll product colors listed are stable.

^bPrevalence of treatment occurring in product: R = rare to occasional; F = frequent; W = widespread or near – total.

Table 3. Rays and Particles Commonly Used for the Irradiation of Gemstones

Irradiation type	Average energy, eV	Coloration uniformity	Induced radioactivity	Localized heating
x-rays	1×10^4	poor	none	none
γ -rays from Co-60 or Cs-137	1×10^6	good	none	none
neutron beam	1×10^6	good	strong	none
electron beam	1×10^6	poor	none	strong
electron beam	2×10^7	good	some	strong

required. Depending on conditions, colors ranging from almost colorless through yellow, green, blue, to black can be obtained. If enough Ti is present, a star effect can be seen by reflection from microscopic needles of rutile [1317-80-2], TiO₂.

When synthetic Al₂O₃ containing about 0.2% TiO₂ is crystallized from the melt, the titanium, mostly as Ti₂O₃, remains in solid solution when cooled relatively rapidly, or it can crystallize as TiO₂ needles and form asteriated material on annealing at about 1200°C for some hours. Natural rubies and sapphires may contain the necessary titanium, but an anneal may be needed to develop the star. If the TiO₂ needles are too coarse or irregular, forming silk, a high temperature may be used to dissolve the needles. This is then followed by rapid cooling for clear material or a lower temperature anneal for asterism. If corundum contains Fe but no Ti, a high temperature heating produces a deep yellow-to-orange color.

If enough Ti to form a star or enough Fe and Ti for a blue color are not present in corundum, it is possible to diffuse these into the surface of a gemstone by heating for many hours close to the melting point in contact with an Al₂O₃ powder containing the missing impurities.

2. Irradiation Treatments

The process of irradiation involves the exposure of a specimen to one of a variety of radiations (1). A summary is given in Table 3.

Table 4. Gemstones Enhanced by Irradiation

Material	Change or product	Comments ^a	Use ^b
corundum	colorless to yellow	S,U,R	R
diamond	near colorless to black, blue, green, yellow, or red	S,R	F
pearl	darken to black	S	F
quartz	colorless to smoky	S,R	W
quartz	amethyst to amethyst–citrine	S,R	F
spodumene	pink kunzite to deep green	U,R	R
topaz	colorless or pale to blue	S,R	W
topaz	colorless or pale to brown	S,U,R	R
tourmaline	colorless or pale to red or multicolor	S,R	F

^aS = stable; U = unstable, may fade; R = can be reversed by another treatment.

^bPrevalence of treatment occurring in product: R = rare or occasional; F = frequent; W = widespread to near – total.

There are three significant possible effects when radiation interacts with matter (5, 6). First, the radiation can interact with the nucleus and induce radioactivity as in the case of neutrons. Second, displacement of atoms can occur. This has happened in a number of uranium- and thorium-containing minerals over geological periods. The outstanding example is zircon, which can contain over 10% Th and 2% U. The internal bombardment from these materials and their decay products over geological periods produces low or metamict zircon, where the disorder gives an amorphous state having a low density.

The third possible effect, a displacement of the outermost electrons in atoms, is of the most interest herein. This displacement can lead to the formation of color centers or to valence state changes. Many gemstones have been colored by irradiation over geological periods from radioactive elements in the earth's crust and from cosmic radiation. Blue and brown topaz, smoky, rose, and amethyst quartz, and purple fluorite are a few examples. All topaz is not blue or brown, because some of these materials have lost color either from relatively recent geological heating or by other means so that there has not been enough time for the color to reform. Additionally, in some localities the rock or soil is particularly free of radioactivity from geochemical depletion and acts as shielding.

With the exception of diamond coloring and the turning of topaz blue, the source of the irradiation is immaterial. Gamma rays are the preferred source because of uniformity of coloration and the absence of heating and induced radioactivity. The most commonly seen gemstones enhanced by irradiation are summarized in Table 4.

2.1. Color Centers

Characteristics of a color center (1, 3, 7) include production by irradiation and destruction by heating. Exposure to light or even merely time in the dark may be sufficient to destroy these centers. Color arises from light absorption either from an electron missing from a normally occupied position, ie, a hole color center, or from an extra electron, ie, an electron color center. If the electron is a valence electron of a transition element, the term color center is not usually used.

Any material which can form a color center contains two types of precursors as shown in Figure 2a. The hole center precursor is an atom, ion, molecule, impurity, or other defect which contains two paired electrons, one of which can be ejected by irradiation, leaving behind a hole center (Fig. 2b). The electron center precursor is an atom, ion, etc, which can produce an electron center by trapping the electron ejected from the hole center precursor. A hole and an electron center are thus formed simultaneously. Either or both can be the color center.

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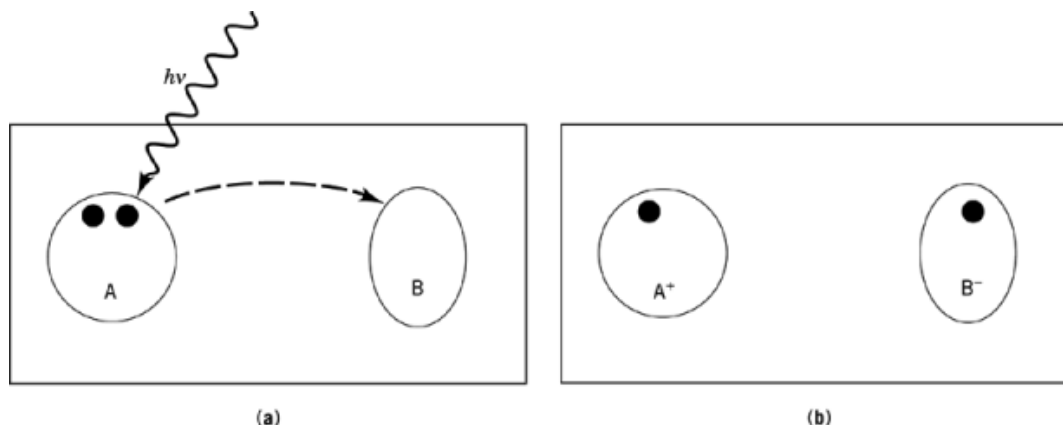


Fig. 2. (a) Irradiation of a material containing A, a hole precursor having an electron pair, and B, an electron precursor, to form (b) a color center having A^+ , a hole center, and B^- , an electron center (1, 3).

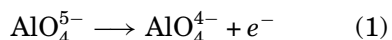
Almost all materials have hole center precursors. If there is no electron center precursor, however, the displaced electron returns to its original place and the material remains unchanged.

When light or heat liberates the trapped electron from the electron center, the hole center can be restored to the original state and color is lost, ie, fading or bleaching occurs. If the trap is shallow, room temperature alone can supply enough energy (about 0.1 eV) to release the trapped electron. For a deeper trap, the 1 to 3 eV of visible light is needed to produce fading as in blue Maxixe beryl, unstable yellow sapphire, and unstable brown topaz. A deep trap holds the electron so strongly that the coloration is stable. Smoky quartz, amethyst, blue topaz, stable yellow sapphire, and stable brown topaz are examples. Heating to as high as 600°C may then be required for bleaching.

In some materials the same color center can exist in both fading and nonfading forms, which are derived from the presence of different electron traps. Examples are yellow sapphire and brown topaz. When any colorless sapphire is irradiated to become yellow, the color usually fades on exposure to light. At the same time nonfading yellow sapphire that contains a color center, but no Fe, occurs naturally. Although both fading and nonfading color centers lose color on heating, irradiation restores both, and light exposure removes only the unstable color. The same pattern occurs in brown topaz. Some amethyst fades very slowly.

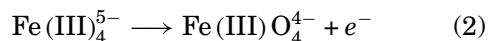
2.1.1. Color Changes in Quartz

All quartz contains small amounts of substitutional Al, typically 0.01%, as well as similar amounts of interstitial hydrogen or alkali metal ions. When an Al^{3+} replaces a Si^{4+} , one positive charge is missing and electroneutrality is maintained by H^+ or Na^+ , etc. Irradiation ejects one of a pair of electrons on an O adjacent to an Al:



The electron can be trapped, for example by an interstitial H^+ , which is converted to an H atom. The AlO_4^{4-} is the hole color center which absorbs light and gives the color to smoky quartz. Bleaching is the result of thermal energy releasing the trapped electron, which then produces the reverse of reaction 1.

The amethyst color center in quartz is exactly like the smoky, except that Fe^{3+} replaces Al^{3+} .



The Fe(III)O^{4-}_4 hole color center gives the purple color. On being heated, the trapped electron is released and the reverse of reaction 2 occurs producing Fe(III)O^{5-}_4 , which provides the pale yellow color of citrine.

2.1.2. Color Changes in Topaz

Essentially all colorless topaz can be turned either brown or blue by irradiation. There are two brown color centers formed at quite low irradiation doses. One is stable to light, the other fades. A stable blue color center (1, 8, 9) is formed also at higher irradiation doses and is revealed when heating at about 300°C removes the browns. Different shades of blue are produced by different irradiation procedures. Gamma rays give a medium blue color but lengthy exposure is needed. Electrons in the 10- to 20-MeV range produce a satisfactory blue within a reasonable time, as do neutrons, all at similar dose levels. The best color is obtained by a double treatment, using first neutrons, then electrons.

2.2. Color Center-Like Color Changes

Green aquamarine contains Fe in two different sites. A blue color, which is not affected by heat, is produced by Fe^{2+} in interstitial sites. Most specimens also contain some Fe^{3+} substituting for Al^{3+} , which by itself produces the yellow color of golden beryl or, together with the blue Fe^{2+} , gives green aquamarine. On heating, the substitutional Fe^{3+} changes to Fe^{2+} from an electron released from a trap. This substitutional Fe^{2+} contributes no significant color. Heat thus bleaches yellow beryl to colorless and converts green aquamarine to blue aquamarine. Irradiation restores the yellow, frequently to a deeper intensity than that originally exhibited.

The irradiation darkening to produce black pearls is merely a charring of the organic conchiolin.

2.3. Radioactive Gemstones

Zircon can contain radioactive elements, but the amount in jewelry-grade material is insignificant. Some of the treatments of Table 3 may leave irradiated material radioactive. Such gemstones have been released on rare occasions without the required cooling-off period (10).

3. Other Treatments

Other treatments fall into the three groups (1) summarized in Table 5. There are chemical treatments and impregnations which penetrate below the surface of the gemstone. Then there are surface coatings of various types. Finally, there are composite stones.

3.1. Chemical Treatments and Impregnations

3.1.1. Bleaching

Diluted hydrogen peroxide [7722-84-1], H_2O_2 , and sunlight are employed to bleach essentially all pearls. The organic conchiolin is thus lightened. Similar processes can be applied to other organic gem materials such as coral, producing gold from black, and to lighten ivory that has become dark with age. Brown tiger's-eye is frequently bleached to give the desired honey color.

3.1.2. Colorless Impregnations and Crack Filling

There may be several different aims in applying a colorless wax, oil, polymer, or glass to a gem material. First, cracks may be hidden, as in oiling an emerald. Ordinary lubricating oil has been used, but this may seep out of the cracks when the stones are warm, or the oil may dry up. Canada balsam [8007-47-4] and polymers have also been used; a vacuum and gentle heating improve penetration (11, 12). A material such as turquoise may

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Table 5. Gemstones Enhanced by Other Processes

Process	Material ^a	Use ^b
	<i>Impregnations</i>	
bleaching	chalcedony, coral, ivory, pearl, petrified wood, tiger's-eye, etc	W
impregnation, colorless oil/wax/plastic	agate, chalcedony, fluorite, jade, lapis lazuli, malachite, marble, opal, turquoise, etc	W
impregnation, colored oil/wax/plastic	same	F
crack filling, colorless plastic	emerald	R
crack filling, colorless oil	emerald	W
crack filling, colorless oil	ruby, sapphire	R
crack filling, colored oil	beryl, emerald, ruby, quartz	R
crack filling, colorless glass	diamond, ruby, sapphire	R
dyeing	agate, chalcedony, marble, onyx, etc	W
	amber, carnelian, coral, ivory, jade, malachite, opal, pearl, turquoise, etc	F
	<i>Surface modifications</i>	
surface color coating	amber, carnelian, diamond, pearl, etc	R
foil back, mirror back, star back	used on any gemstone	R
lasering	diamond inclusions	F
	<i>Composite gemstones</i>	
doublets, triplets	opal	F
doublets, triplets, artifact-included, gel-filled	amber, beryl, emerald, ruby, sapphire, etc	R
synthetic overgrowth	emerald on beryl	R

^aMost of the enhanced products listed are unstable.

^bPrevalence of treatment occurring in product: R = rare or occasional, F = frequent, W = widespread to near – total.

be stabilized using paraffin wax [8002-74-2] or polymers to prevent perfume and perspiration from entering the porous surface and producing a color change. At the same time, low grade turquoise which is white because of light scattering from its porosity may show an improvement from an impregnation which fills the pores. This same process may convert worthless chalky opal into a brilliantly colored form. A more recent development is the use of glass (qv) to fill cracks and even holes in ruby (13), using a high melting glass, and in diamond (14), using low melting glasses containing oxides and halides of Pb, Bi, etc.

3.1.3. Colored Impregnations

Colored oil to fill cracks is used on gemstones primarily to improve color, most frequently on emerald. Colored oil is also used to simulate other stones, most frequently quartz. Depending on the dye used, the colors may fade, in addition to the problems associated with colorless oiling.

3.1.4. Dyeing Porous Chalcedony

Many gemstone materials of the cryptocrystalline quartz family collectively known as chalcedony are porous and resistant to heat and acids. Thus many dyeing techniques are applied. The use of stable inorganic precipitates or decomposition products has been perfected (1). The oldest process, going back to Roman times, is the honey- or sugar-acid technique used to produce deep yellow, brown, or black. After cleaning, the material is soaked in honey or a sugar (qv) solution. Heat then gives yellow to brown colors. Alternatively, boiling in concentrated sulfuric acid [7664-93-9], H₂SO₄, carbonizes the sugars, giving a permanent black; essentially all black onyx is made this way. Many colors are produced by forming insoluble pigments within the pores, eg, Fe₄(III)(Fe(II)(CN)₆)₃, Prussian blue [14038-43-8] or Turnbull's blue [25869-98-1]. Metallic silver [7440-22-4] is precipitated for black. Heating to decompose water-soluble substances is used to produce chromic oxide [1308-38-9], Cr₂O₃, for green, iron oxides for yellow, brown, red, etc.

3.1.5. *Dyeing Materials Other than Chalcedony*

Most other gemstone substances cannot tolerate the acids or the temperatures used on chalcedony. Natural or synthetic organic dyes, even ordinary inks (qv), fabrics dyes, etc, are frequently used. Fading is, however, a problem (see Dyes and dye intermediates). Pearls are frequently dyed pink using an organic dye such as eosin [15086-94-9]. Exposure of pearls to silver salts followed by light exposure, or using a dyed pearl seed for culturing, give black. The honey- or sugar-acid process has also been used on porous opal to give a black background to the flashes of color. The older process, smoking, of opal packs the stones with oil or manure in paper, followed by heating.

3.2. Surface Modifications

A wide variety of surface modifications have been used on gemstones. These include treatment with wax, paints, varnishes, interference filters, foil backs, mirror backs, inscribings, selective decorations, and synthetic overgrowth. A clear varnish or wax coating is frequently applied to poorly finished tumbled stones, cabochons, or carved objects to improve the appearance or even to avoid completely the polishing step. Foil backs, shiny or colored metal sheets or metallic paints, behind a gemstone reflect light. These modifications were widely used in the days before faceting for total internal reflection. Most frequently such modifications are seen on glass or even plastic stones for costume and stage jewelry. Metal foil that has been embossed or inscribed with scratches is used behind a clear cabochon to simulate a star; the scratches also may be made directly on the back of the stone itself.

Drilling diamonds using a focused laser beam to burn out dark inclusions or make the inclusions accessible to a chemical treatment is a frequent enhancement. In a potential deception, a cubic zirconia diamond imitation was laser drilled to make it more convincing (15).

3.3. Composite Gemstones

Many types of composite or assembled gemstones have been made (1). Some are shown in Figure 3. In the United States a doublet has two pieces combined using a colorless cement. If three pieces are used, or if two pieces are assembled using a colored cement, the gemstone is a triplet. The use of composite stones has declined rapidly with the rise of inexpensive synthetics. Frequently seen are opal doublets, where precious opal is backed by a black material. In opal triplets a thin slice of precious opal is cemented between a black backing and a clear cover, usually of quartz. Additionally, insects and even fish have been inserted into amber.

A thin layer of dark green beryl had been grown by a hydrothermal technique over the surface of a pale beryl to imitate emerald. It has been suggested that such stones should be called synthetic emerald-beryl doublets (16). The ability to grow thin, but not thick, single-crystal diamond on the surface of natural diamond (17) leads to the possibility of growing such a thin film colored blue with boron; this has been done experimentally (18).

4. Identification of Treated Gems

A trained gemologist, taught by the Gemological Institute of America of Santa Monica, Calif., and New York, the Gemmological Association of Great Britain of London, or elsewhere, is needed for identification of treated gems. This topic is also discussed in textbooks (19–21). In some materials the induced change is the exact equivalent of a process that also occurs naturally, so that such treatments cannot be identified.

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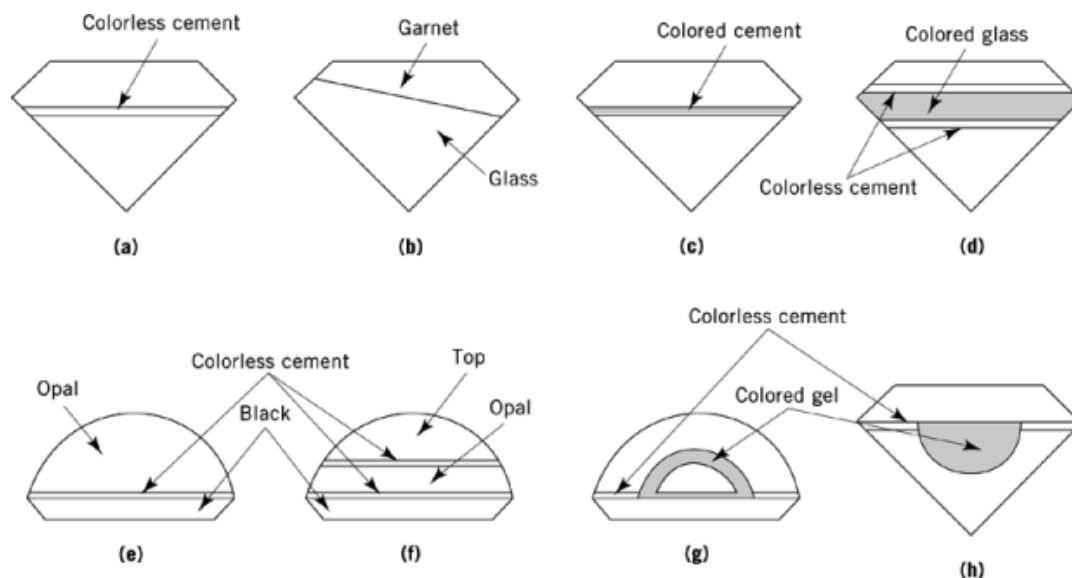


Fig. 3. Forms of composite gemstones: (a) doublet; (b) fused garnet-top doublet; (c) and (d) triplets; (e) opal doublet; (f) opal triplet; (g) and (h) gel-filled triplets (1).

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