Marble-hosted Ruby from Vietnam

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ABSTRACT

In the 1980s the first discovery of ruby was made in the Luc Yen district of Vietnam (Yen Bai province). Then in the 1990s, occurrences of ruby were found in Quy Chau (Nghe An province). This article briefly reviews the geology and gemmological properties of marble-hosted rubies from Luc Yen and Quy Chau. The most notable features of these rubies are the high content of chromium compared to iron and titanium, and the widespread presence of mineral inclusions. Fluid inclusions provide evidence of CO_2*-*H_2S*-COS-*S_8*-AlO(OH)-bearing fluids with native sulphur and diaspore daughter minerals.*

SOMMAIRE

Dans les années 80, les premiers rubis furent découverts dans le district de Luc Yen au VietNam (Province de Yen Bai). Puis, dans les années 90, du rubis fût également découvert à Quy Chau (Province de Nghe An). Cet article décrit brièvement la géologie ainsi que les propriétés gemmologiques de rubis issus de marbres des mines de Luc Yen et de Quy Chan. L'aspect le plus remarquable de ces rubis se trouve être leur forte concentration en chrome par rapport à la présence de fer et de titane et les nombreuses inclusions minérales. L'analyse des inclusions liquides démontre la présence de molécules de CO_2*-*H_2S*-COS-*S_8*-AlO(OH) contenant en inclusions solides du soufre et du diaspore.*

INTRODUCTION

Rubies and sapphires have been found and mined in many places in Vietnam. Rubies are now being exploited in the northern part of the country in the Luc Yen mining district and in the Quy Chau deposits. In 1983, the first discovery of ruby was made in the An Phu area (Luc Yen district), 270 kilometres north of Hanoi (Figure 1), by a geologist during field mapping. The corundum occurrence was investigated in detail and the exploitation started in 1987. In March 1988, Vinagemco (Vietnam Gemstones Company) was set up by the Vietnam government to control the gemstone activity. That same year, Vinagemco and BH Mining Co. from Thailand established a joint venture to mine the Khọan Thong placer. From November 1989 to March 1990, the company produced about 244 kg of gem quality corundum, most of which was cut and traded in Bangkok.

From 1990 to 1994, thousands of independent miners swarmed over the area turning up new gem-quality ruby occurrences, such as Minh Tien,

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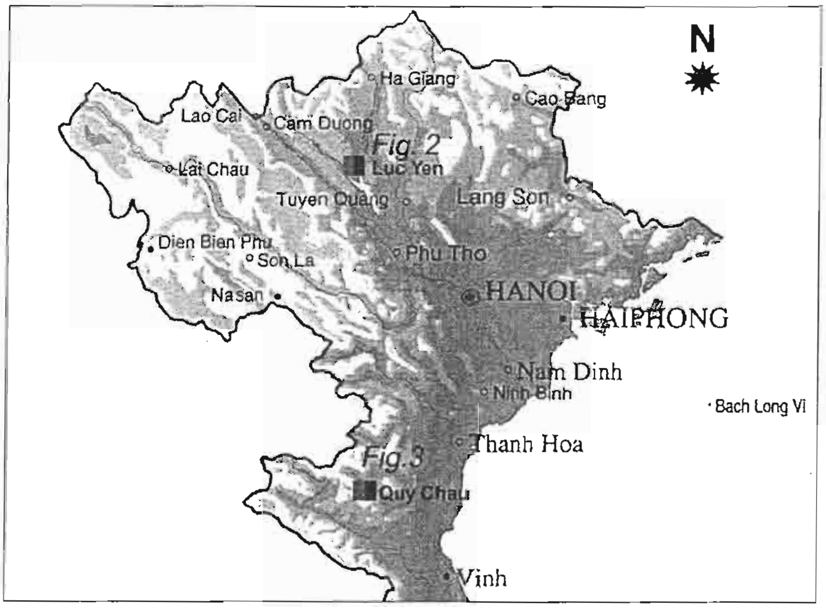

Figure 1. Map of North Vietnam showing the location of the Luc Yen and Quy Chau ruby deposits.

Nuoc Ngap, Hin Om, Khau Nghiem, Vang Sao, May Thuong and May Ha. Sapphires and other minerals were also washed and sorted from placers. Ruby crystals have also been extracted from the marbles, and marketed in the town of Luc Yen.

The Quy Chau mining district is located about 120 km northwest of Vinh City (Nghe An Province), about 300 km south of Hanoi (Figure 1). In 1988, ruby was discovered. From 1989 to 1990, 10,000 miners came to the region from all over Vietnam. The illegal mining activities were beyond the control of the Nghe An People's Committee, and hundreds of people died in July 1990 from a cave-in at the Ho Ty mine.

Thousands of carats of high-quality rubies and pink sapphires were sold to Thai traders, in spite of the monthly auctions of ruby held by the provincial government. In one of the auctions in 1996, a 56-carat ruby was sold for US$562,000. That same year, management of the deposit was taken over by the Nghe An Gem and Gold Company. Projects carried out in this area have resulted in the production of hundreds of kilograms of rubies and pink sapphires. These were auctioned September 15, 2001. Since July 2002, the Vietnam National Gem and Gold Corporation has organised gem sales weekly at the head office in Hanoi.

MATERIALS AND METHODS

The samples used in this study were rough and polished samples of ruby that were taken from placers and the marble. Gemmological investigation were carried out at the Centre for Gem and Gold Research and Identification (VGGC) in order to record their refractive indices, birefringence, optics, pleochroism, and optical absorption spectra, all with standard gemmological instruments. Specific gravity was determined on polished stones and rough crystals by the hydrostatic weighing method. Characteristics of solid inclusions were examined using a gemmological microscope. Thin section studies were performed at Hanoi University of Science.

All Raman spectra of inclusions were obtained with a Jobin-Yvon Horiba Labram instrument, using the 488-nm Argon laser to avoid ruby fluorescence.

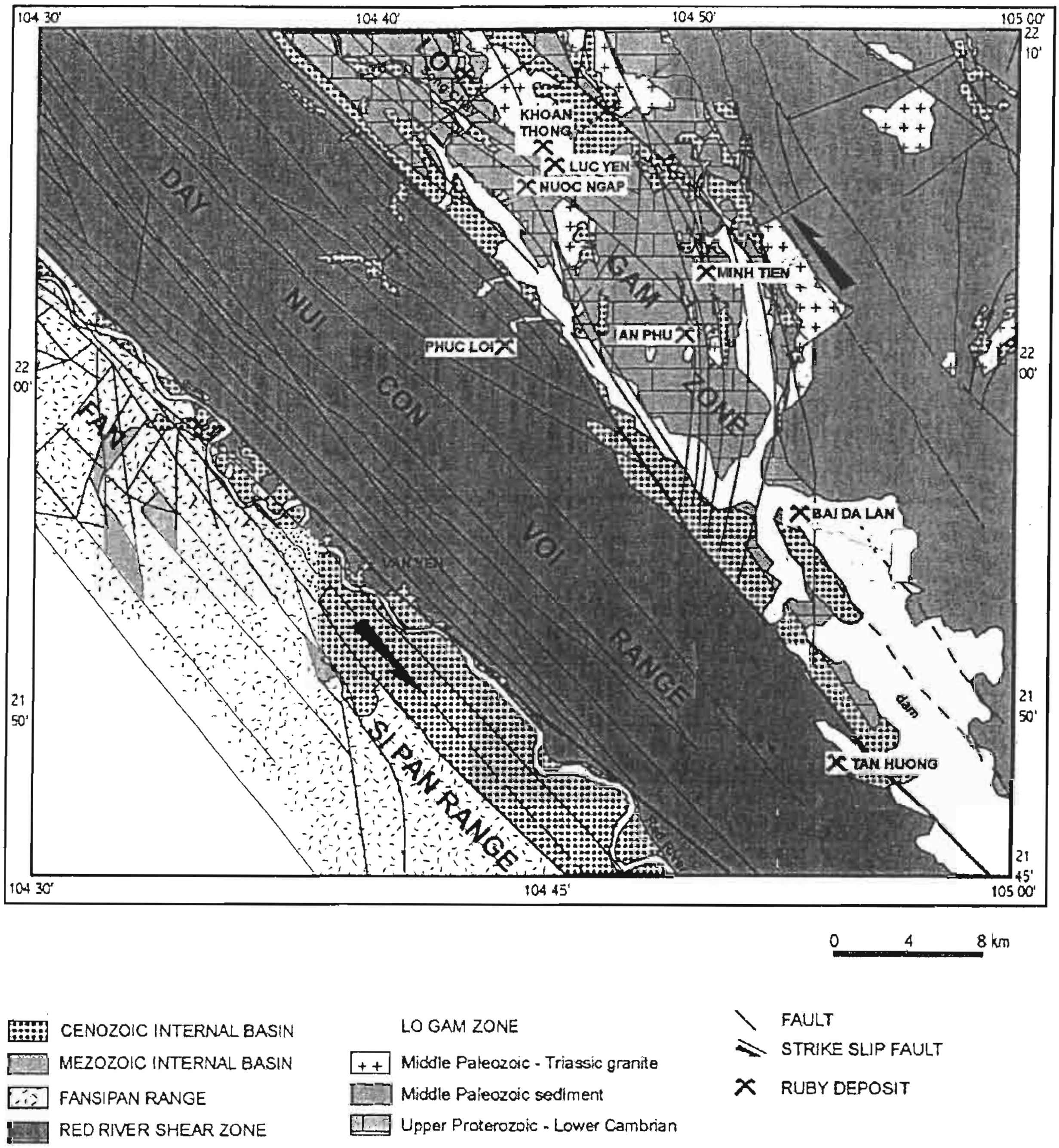

Figure 2. Geological map showing the major tectonic domains of the Red River shear zone and the location of the main corundum occurrences and deposits.

Chemical analysis of the stones and their inclusions was carried out using a Hitachi 2500 scanning electron microscope (SEM) equipped with an energy-dispersive spectrometer (EDS). For microthermometric analysis, double-polished wafers of rubies (200–300 μm thick) were used. The different types of fluid inclusions were defined under a microscope with magnification between 100–400X. Microthermometry studies were carried out using a Reynolds stage from CRPG / CNRS (Nancy, France). Solid phases contained in fluid-inclusion cavities were analysed on a Fuji Stereoscan 250 SEM, (accelerating voltage 20 kV) at University Henri Poincaré Nancy 1.

GEOLOGY OF RUBY DEPOSITS

Luc Yen mining district: The ruby deposits of Luc Yen (Figure 2) are set in moderate to high temperature recrystallized marble units of Upper Proterozoic-Lower Cambrian age in the eastern side of the Red River shear zone (the Lo Gam tectonic zone) (Pham Van, 1996). Primary ruby occurs as

(a) disseminated crystals within marbles (Figure 3) with phlogopite, dravite, margarite, pyrite, rutile, spinel, edenite and graphite (Bai Da Lan, An Phu, Minh Tien, Nuoc Ngap, Luc Yen and Khoan Thong mines);

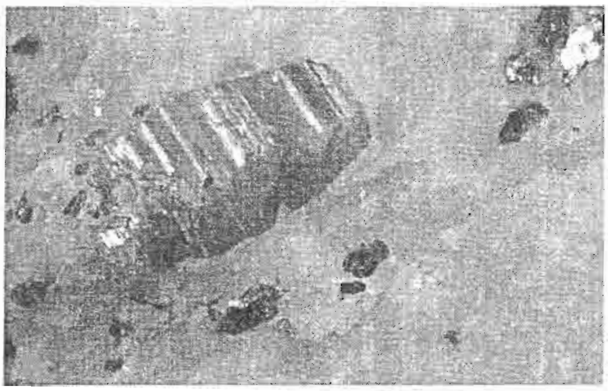

Figure 3: Ruby crystals (1.5 cm long), phlogopite, and graphite in marble from Luc Yen. Photo by G.Giuliani.

(b) veinlets associated with calcite, dravite, pyrite, margarite and phlogopite (An Phu mine);

(c) fissures with graphite, pyrite, phlogopite and margarite (Bai Da Lan mine), Minh Tien region.

Secondary deposits consist of gravel concentrations in karst pockets and in alluvial fans in the Luc Yen valleys. The gem-bearing valleys are often narrow, small depressions ranging from 0.5 to 0.7 km^2, but most commonly 2–3 km^2. Rubies are pink, purple to red. Blue and colourless sapphires coexist with rubies, as well as with grey to brown, bipyramidal sapphires and trapiche rubies. Associated gem minerals include red, pink and pale blue spinel, gem quality multicolour tourmaline, and garnet. The great variety and high quality of the gem material recovered in the placers supply the gemstone market in the centre of Luc Yen town (opened daily for dealers since 1987).

Quy Chau mining district: This area, located 200 km south of the Red River shear zone, occupies the Bu Khang dome (Figure 4). It consists in a broad antiform of Paleozoic and Mesozoic sedimentary and meta-sedimentary rocks overlying a core of mica schists, granitoids, paragneisses and orthogneisses. The northeastern part of the dome is limited by the major extensional Cenozoic shear zone of Quy Chau, where the corundum deposits are located.

Rubies have been mined since 1987 in the placer deposits of Doi Ty, Doi San, Mo Coi and Quy Hop. The corundum in the Quy Chau area occurs principally as

(a) very rare and uneconomic rubies disseminated in marbles and associated with pyrite and graphite,

(b) in placers, which form the economic deposit. In the Doi San and Doi Ty areas, granitic intrusions resulted in the injection of pegmatites and the formation of calcium-magnesium-rich skarns in the surrounding marbles, amphibolites, gneiss and mica schists. Rubies were observed neither in the skarn nor in the pegmatite. Gem material consists of ruby, with smaller amounts of blue to violet and orange sapphire. Ruby tends to be slightly purplish red.

GEMMOLOGICAL PROPERTIES

Optical characteristics

Visual appearance: Ruby crystals in marble from the Luc Yen deposit show more varied forms than those from the Quy Chau deposit. The commonest forms of ruby crystals in Luc Yen are hexagonal bipyramid (Figure 5), and hexagonal prism with the development of the rhombohedral faces creating "spindle", and "drum-barrel" shapes. In contrast, the commonest crystal forms of ruby crystals at Quy Chau are short hexagonal prisms, and barrel shapes including basal pinacoid faces, prisms, and rhombohedral faces.

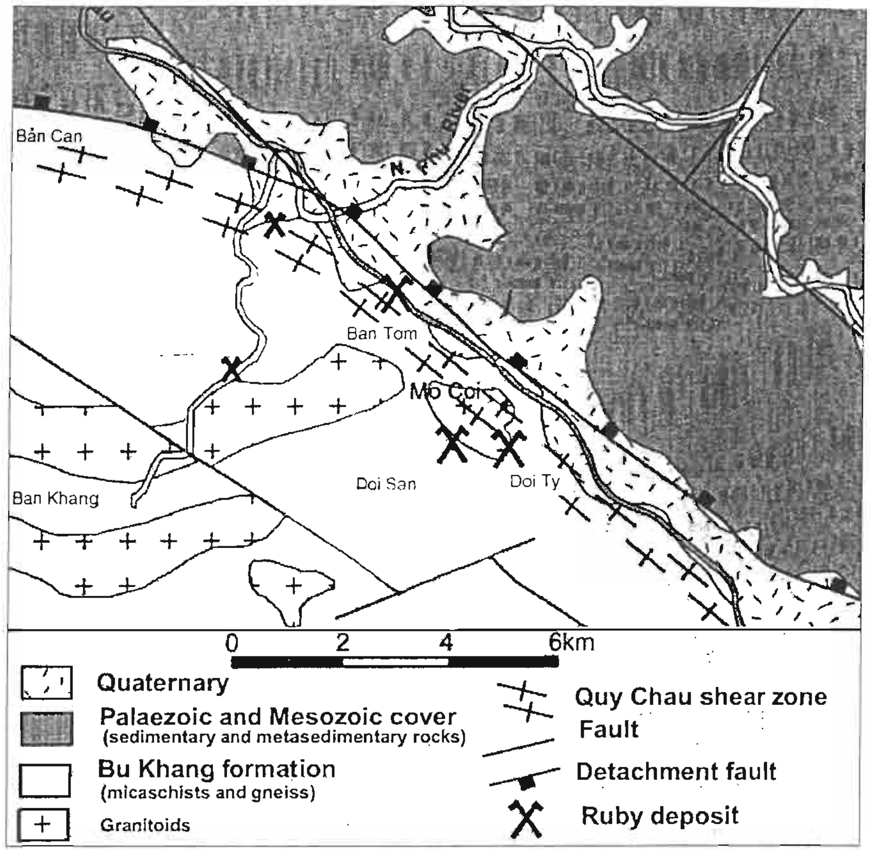

Figure 4: Geological map of Quy Chau area showing the location of the ruby deposit.

Colour: The colour of ruby from the Quy Chau deposits ranges from moderate to high saturation of purplish red to purplish pink. Meanwhile the rubies from Luc Yen deposits show red to pink tones The red colour of ruby from Luc Yen is usually less saturated than that from Quy Chau.

Refractive index (RI) and Birefringence: The RI of rubies was obtained at the VGGC using a GEM Duplex II refractometer with monochromatic light source. Birefringence values are within the normal range of corundum. All of the stones have an RI from 1.762–1.770, with a birefringence around 0.008.

Specific gravity (SG): The SG was determined by hydrostatic weighing method. The samples from Quy Chau (15 polished stones and 8 rough crystals) gave values between 3.94 to 4.05. The SG of 25 samples (20 polished stones and 5 rough crystals) from Luc Yen are between 3.92 and 4.01, little lower than those from Quy Chau.

UV luminescence: All of the red and pink samples from the Quy Chau and Luc Yen deposits fluoresced red when exposed to the long-wave (366 nm) and short-wave (254 nm) ultraviolet radiation. Stones from Quy Chau tended to fluoresce more strongly than those from Luc Yen.

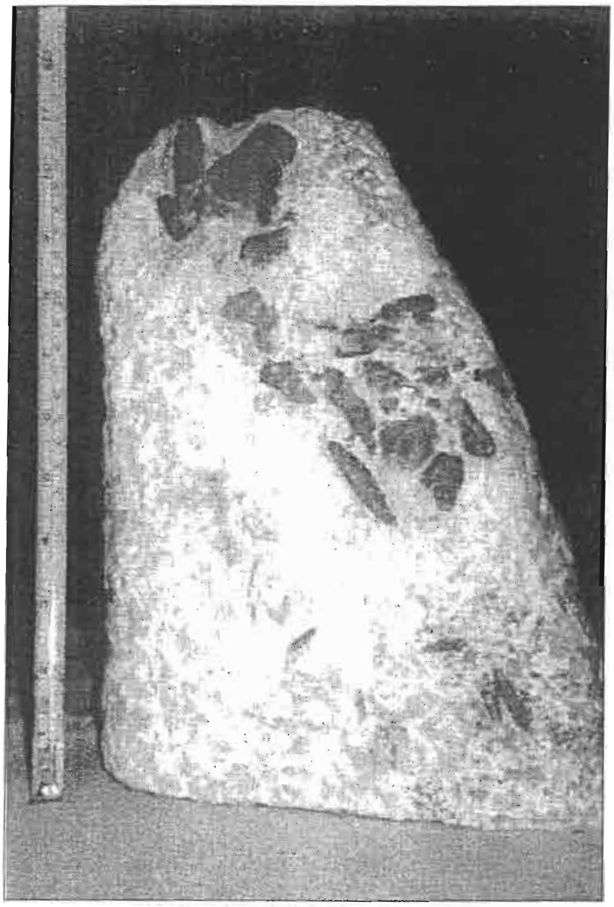

Figure 5: Hexagonal bipyramid of ruby crystals (range from 0.5-6.5 cm long) in marble from Luc Yen deposit. Photo by Pham Van Long.

Internal characteristics:

Twinning: Some samples from Quy Chau and Luc Yen showed twin features, including lamellar and polysynthetic twinning. The twin planes are usually emphasized by needle-like boehmite inclusions (Figure 6a). Boehmite needles in ruby from Luc Yen are usually thinner and longer than those from Quy Chau ruby (Figure 6b).

Growth features: Straight and angular parallel growth features are common in rubies. Other investigations (Kane et al., 1991) described swirl-like growth effects, which are common in rubies and fancy sapphires from Burma.

Colour zoning: Some of the specimens studied showed colour zoning features. The colour zones range in size from narrow to large areas related to growth planes. In some samples, dot-like and band-like colour zones were observed.

Solid inclusions: (See Table 2.) The most common mineral inclusions found in Luc Yen and Quy Chau rubies are apatite, calcite, dolomite, rutile, diaspore, zircon, and phlogopite. These minerals are also found in the marbles.

Calcite crystals are generally found in many different shapes. They can be relatively complete crystals or corroded (Figure 7a, b). In ruby from Luc Yen and Quy Chau, calcite inclusions are generally distributed together

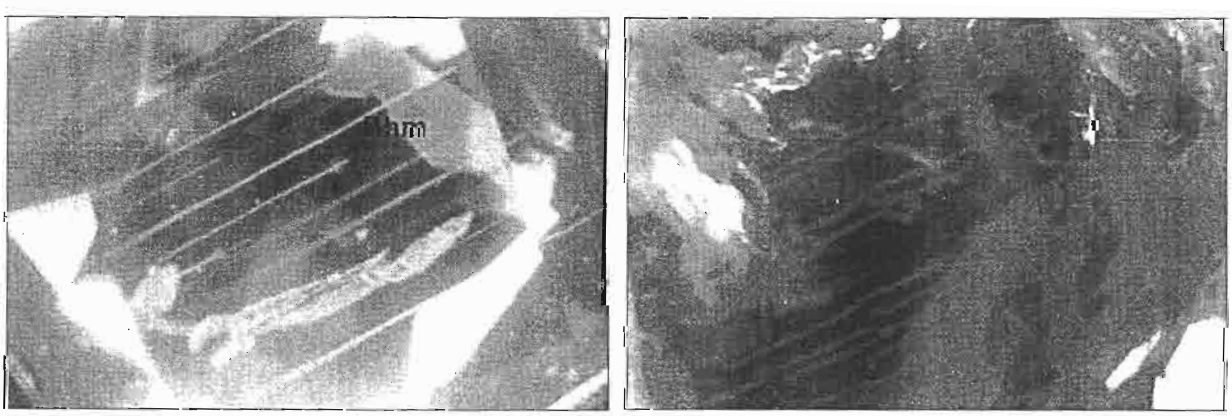

Fig. 6a (L). Needle-like boehmite inclusions (Bhm) in ruby from Luc Yen. Magnified 15x; Fig.6b (R). Boehmite inclusions in Quy Chau ruby. Magnified 15X. Darkfield illumination. Photos by Pham Van Long.

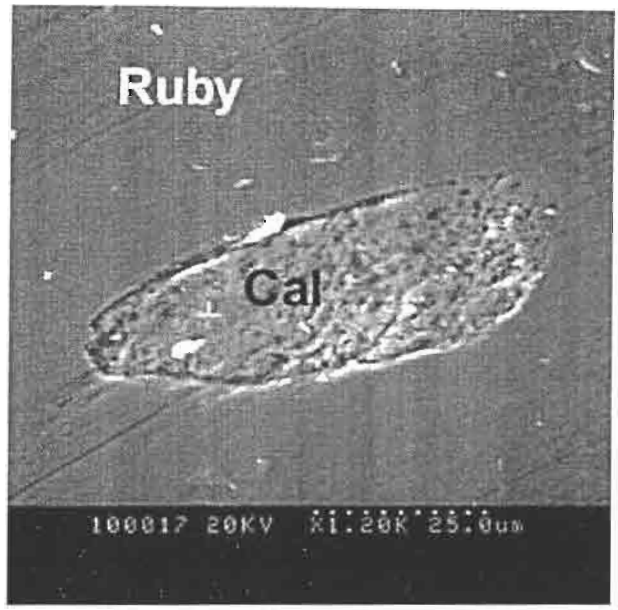

Figure 7a. (L) Photomicrograph of calcite (Cal) inclusion in ruby from Luc Yen. Magnified 25x. Darkfield illumination; 7b. (R) SEM image of calcite (Cal) inclusion in ruby from Quy Chau. Photos by Pham Van Long.

with zircon, corundum, apatite, and rutile.

In ruby, rutile exhibits many different shapes, including well-formed prismatic crystals (Figure 8), spindle-shaped, or fibre-shaped crystals, the result of exsolution. The rutile inclusions are of two types: short needles and twinned, plate-like crystals. It commonly appears as transparent, orange-brown or opaque crystals trapped along the growth zones. Rutile inclusions are generally reddish-brown in colour with metallic or semi-metallic lustre. Concentrations of the very thin needle and fibrous inclusions result in clouded, milky effects that are very popular.

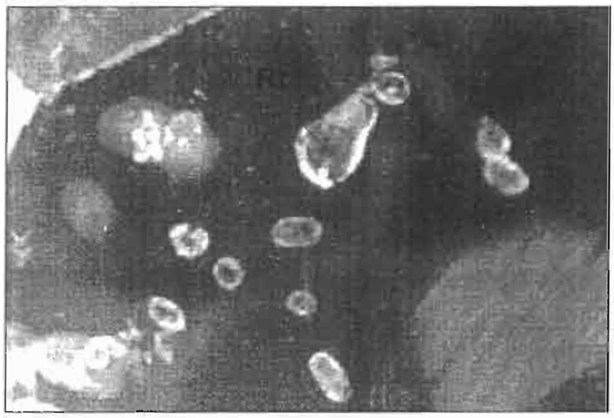

Figure 8: Syngenetic inclusion of rutile (pear shape) and protogenetic inclusion of apatite (oval) in ruby from Quy Chau. Magnified 30X. Darkfield illumination. Photo: Pham Van Long.

In ruby from Luc Yen and Quy Chau, apatite inclusions can be at the same time protogenetic and syngenetic. As protogenetic inclusions, they are generally corroded, whereas syngenetic inclusions maintain well-formed original, hexagonal prismatic shapes. Apatite inclusions are generally yellowish to colourless, transparent, and in low relief.

Most zircon inclusions are protogenetic (Figure 9), and generally corroded with no well-formed crystal shape. Zircon inclusions in ruby from Luc Yen and Quy Chau are colourless and transparent with high relief and strong adamantine lustre. A characteristic feature is the presence of radiation "haloes" around inclusions (Figure 9a). These rims are the result of radioactive elements in the zircon.

In addition to the principal inclusions mentioned above, we found anorthite and andalusite (Figure 10), and mica inclusions that are

Figure 9a. (L) Photomicrograph of zircon (Zrn) inclusion with typical radiation "halo" in ruby from Luc Yen. Magnified 49X. Darkfield illumination; 9b. (R) SEM image of zircon inclusions (Zrn) in ruby from Quy Chau. Photos by Pham Van Long.

transitional between muscovite and phlogopite (Figure 11). The presence of these inclusions in Quy Chau ruby provided helpful information to debate the origin of the ruby (see the discussion below). Less frequently, inclusions such as spinel, dolomite, graphite, pyrite, phlogopite, and muscovite are also found in ruby from both deposits. Other studies found nepheline and sphene (Dao et al, 2001). Zoisite is the typical inclusion in ruby from Quy Chau, and it indicates the regional metamorphism of these stones.

Fluid inclusions: Three main types of fluid inclusions are recognised in the rubies from Luc Yen and Quy Chau on the basis of their respective chronology (Giuliani, et al, 2003a,b).

Type A inclusions (primary) occur as isolated or oriented clusters between 20 to 200 μm in size, present throughout from the core to the rim of the crystals (Figure 12), and are best observed in longitudinal sections. Type A inclusions are commonly two-phase fluid inclusions (liquid and vapour carbonic phases), composed of CO_2, H_2S and carbonyl sulphide (COS). They may also be associated in a single growth zone with single-phase carbonic-rich inclusions. The volumetric fraction of the carbon dioxide-rich liquid in the carbon dioxide-rich phase shows degrees of filling ranging between 60 and 100%. Type A inclusions show two kinds of morphology: (i) euhedral, negative crystals with polygonal or square outlines found in planes parallel to the basal pinacoid *c* (0001); (ii) flat or broad tubes

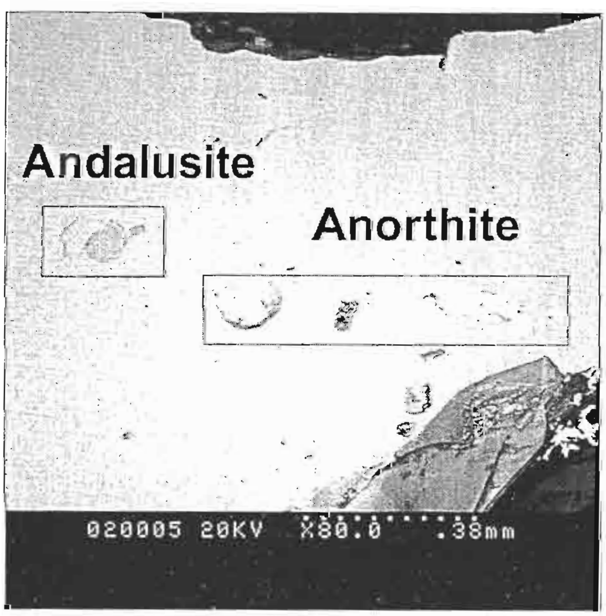

Figure 10: SEM image of assemblage of anorthite (Anr) and andalusite (And) inclusions in ruby from Quy Chau. Photo by Pham Van Long.

sometimes capped by mineral inclusions. Diaspore is found either as very rare solid inclusions, as prismatic crystals with a length up to 15 µm or as a non-visible film, 2–3 µm thick, coating the wall of the whole inclusion cavity. Diaspore was identified by its Raman lines at 331 and 448 cm^{-1}. Native sulphur is present at room temperature as very rare solid globules in the inclusions, but it is commonly nucleated from a non-visible thin layer during laser irradiation. S_8 was identified by its characteristic peaks at 220 and 462 cm^{-1}.

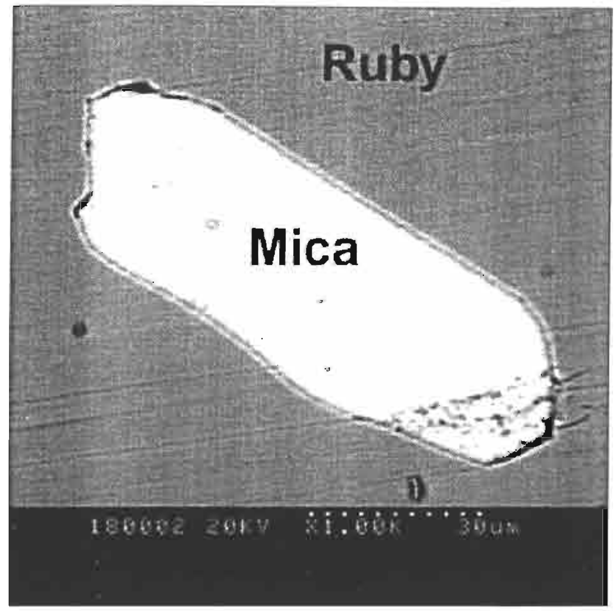

Figure 11: SEM image of mica inclusion transitional between muscovite and phlogopite. Photo by Pham Van Long.

Type B fluid inclusions (pseudo-secondary) appear as small clusters in crystals or as isolated inclusions mainly related to small intra-granular fractures. Fluid-inclusion planes are related to micro-cracks and healed fractures during crystal growth. At room temperature, Type B inclusions are single or two-phase (liquid and carbonic phases) inclusions with degree of filling between 40 and 100%. They have variable sizes (from 20–125 µm) and variable shapes close to a negative crystal. Some of the negative crystals are capped by mineral inclusions (mainly calcite) at one end, and contain calcite, rutile, and daughter crystals of diaspore and native sulphur, determined by Raman spectrometry.

Type C fluid inclusions (secondary) occur along healed fracture planes that cross-cut several crystal boundaries and have an elongated (Figure 13) or rounded morphology. Irregular or crescent shapes are also present, and are interpreted as the result of textural re-equilibration. Their size ranges from

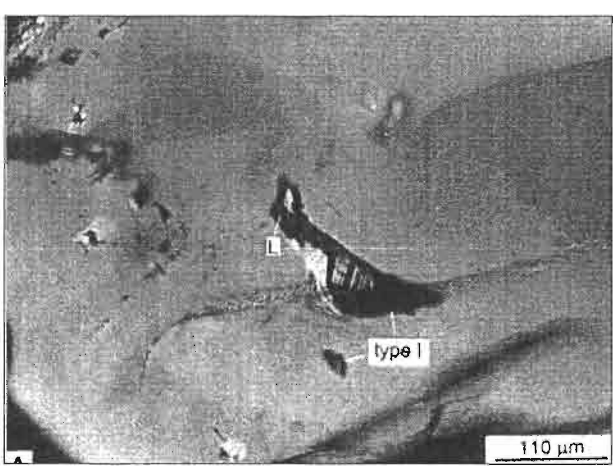

Figure 12: Isolated primary fluid inclusion (type A, labelled 1) of 100 µm long containing a liquid phase composed of CO_2, H_2S and COS. Photo by G. Giuliani.

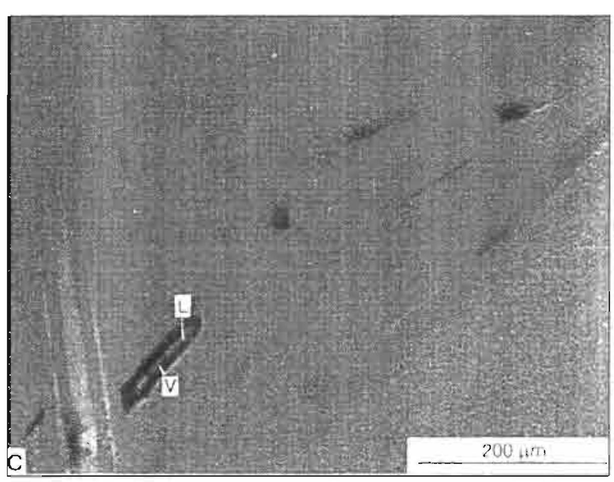

Figure 13: Secondary fluid inclusion (type C) in ruby from Quy Chau distributed along the healed cracks, that contained liquid (L) and vapour (V) phases. Photo by G. Giuliani.

10–100 μm. At room temperature, they contain one or two phases, and the volumetric fraction of the carbonic-rich liquid in the carbon-rich phase is between 35 and 100%.

Raman analyses of the three types of fluid inclusions revealed that the carbonic phase is always composed of CO_2, H_2S and carbonyl sulphide (COS).

CHEMICAL COMPOSITION

Chemical analysis of ruby from Luc Yen and Quy Chau revealed chromium, iron, titanium, vanadium, silicon, manganese, magnesium, calcium, gallium, germanium, scandium, and zinc. In both mines, the average content of chromium is usually higher than that of iron and titanium combined. In Luc Yen, the average contents of chromium, iron, and titanium are 0.227%, 0.083%, and 0.050% respectively. Meanwhile, in ruby from Quy Chau the figures are 0.295%, 0.086%, and 0.045% respectively (Pham Van Long, 2003). On average, corundum from the Quy Chau deposits contains slightly more chromium than the corundum from Luc Yen (0.295% compared to 0.227%). As well, Quy Chau ruby usually has a higher ratio of chromium to iron plus titanium (2.252 to 1.707). This explains the widespread occurrence of rubies at Quy Chau, whereas there is a wide range of colours of ruby and other sapphires at Luc Yen.

DISCUSSION

The mineralogical and gemmological characteristics of rubies from marble in Luc Yen and Quy Chau are very similar, reflecting similar formation conditions and origin of both mines. Inclusions in Luc Yen and Quy Chau rubies are very diversified and abundant, and are typical of a regional metamorphic process.

The composition of inclusions in rubies from Luc Yen and Quy Chau

Table 2. Chemical analysis of mineral inclusions in rubies from Luc Yen by SEM methods.

	Compositions (%)											
	SiO_2	Al_2O_3	MgO	K_2O	TiO_2	MnO	FeO	H_2O	ZrO_2	HfO_2	Na_2O	CaO
Phlogopite	40.98	20.00	17.16	10.52	0.17	-	6.56	4.00	-	-	-	-
Muscovite	44.11	37.34	0.34	10.98	2.08	-	0.46	4.46	-	-	-	-
Rutile	-	5.54	-	-	93.93	-	-	-	-	-	-	-
Spinel	-	71.55	27.11	-	-	0.12	1.03	-	-	-	-	-
Zircon	32.10	-	-	-	-	-	-	-	65.00	1.95	-	-
Hercynite	-	59.54	-	-	1.45	-	38.65	-	-	-	-	-

results from regional metamorphism; these inclusions are anatase, diaspore, muscovite, andalusite, anorthite, zoisite, and CO_2-H_2S-COS-S_8-AlO(OH)-bearing fluid inclusions. The co-existence of anorthite and andalusite inclusions in ruby from Quy Chau is suggestive that the host ruby formed from mica minerals according to the reaction:

$$3CaAl_4Si_2O_{10}(OH)_2 + SiO_2 \rightarrow 3CaAl_2Si_2O_8 + Al_2SiO_5 + 2Al_2O_3 + 3H_2O$$

margarite silica anorthite andalusite ruby

Analysis of a mica inclusion in Quy Chau ruby (Figure 11) showed SiO_2 = 44.44%; Al_2O_3 = 33.26%; MgO = 8.20%; K_2O = 10.00%; TiO_2 = 1.56% and F = 2.54%. The contents of aluminum and magnesium indicate that this mica inclusion is transitional between muscovite and phlogopite. We therefore conclude that a metamorphic reaction resulted in the formation of ruby from dolomite and muscovite as follows:

$$3CaMg(CO_3)_2 + KAl_3Si_3O_{10}(OH)_2$$

dolomite muscovite

$$\downarrow$$

$$KMg_3(AlSi_3O_{10})(OH)_2 + 3CaCO_3 + Al_2O_3 + 3CO_2$$

phlogopite calcite ruby

CONCLUSION

Characteristics of marble-hosted rubies from Luc Yen and Quy Chau deposits are the same in mineralogical and gemmological properties, including trace-element chemistry, typomorphism of crystals, inclusion composition, and other features.

The appearance and properties of the marble-hosted rubies from Vietnam are characteristic of the geological environment in which these rubies form. Different types of corundum in Vietnam occur in amphibolite, gneiss, marble, basalt, pegmatite and metasomatised pegmatite. The ruby from marble deposits in the Luc Yen and Quy Chau districts plays an important role in the gemstone market. Details of the nature of solid and fluid inclusions in these rubies indicate that high-grade regional metamorphism (amphibolite facies) accounts for their origin.

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Table 1. Gemmological properties of rubies from Luc Yen and Quy Chau deposits.

PROPERTY	Quy Chau rubies	Luc Yen rubies
Colour	Purplish red to purplish pink; high saturation	Ranges from red to pink; saturation usually lower than for Quy Chau ruby
Clarity	Translucent to opaque	Translucent to opaque
Refractive index	1.762–1.770	1.762–1.770
Birefringence	0.008	0.008
Optic character	Uniaxial negative	Uniaxial negative
Specific gravity	3.94 to 4.05	3.92 to 4.01
UV luminescence Long wave	Very strong red	Strong red
Short wave	Strong red	Medium red
INTERNAL FEATURES		
Colour zoning	Colour zones range in size from narrow to large areas that follow the growth plane of the stones. Dot-like and band-like colour zones were observed.	Colour zones are usually in large area. Dot-like and band-like colour zones were rarely found.
Twinning	Frequent lamellar and polysynthetic twinning	Similar to Quy Chau rubies
Growth features	Straight and angular parallel growth features; swirl-like growth effect commonly found	Straight and angular parallel growth features; swirl-like growth effect is sometimes found
INCLUSIONS		
Solid inclusions		
Primary	Anatase, andalusite, anorthite, apatite, biotite, boehmite, brookite, calcite, corundum, diaspore, dolomite, graphite, margarite, muscovite, phlogopite, pyrite, rutile, spinel, zircon and zoisite. Other studies found sphene and nepheline (Dao et al, 2001), nordstrandite (Kane R. et al, 1991).	Anatase, apatite, biotite, boehmite, calcite, corundum, diaspore, graphite, hercynite, margarite, monazite, muscovite, phlogopite, pyrite, rutile, spinel, tourmaline, and zircon. Other studies found sphene and nepheline (Dao et al, 2001; pyrrhotite and nordstrandite (Kane R. et al, 1991).
Secondary	Needle-like inclusions of rutile and boehmite; clouds and milky effects of rutile silk; orange cracks.	Similar to Quy Chau rubies
Liquid inclusions	CO_2-H_2S-COS-S_8-AlO(OH)-bearing fluids with native sulphur and diaspore daughter minerals.	Similar to Quy Chau rubies

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