

## Gemmology in the service of archaeometry

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Archaeometric studies of ancient artifacts containing gems or gem-quality geological materials have an intrinsic complexity. The scientific questions to be answered are related not only to the type of material used (*e.g.* a mineral or poly-mineral geological material), but also to their age and provenance.

The answers can derive only from multidisciplinary study which combines experimental observations with information and clues from different disciplines.

This paper presents three case studies in which mineralogical knowledge and the non-invasive approach typical of gemmological analyses solve the problem of gem identification. The answer about the origin of gems and/or minerals is more complex because little is known about the *chaîne opératoire* that precedes the use of gems in artifacts. Little is known currently about the geological complexity of ancient ores, some are now exhausted. Moreover, the criteria used in choosing raw material are not known.

A multidisciplinary approach can lead to identification of the sources of supply of the material, understanding the choices made for the realization of the artifacts, defining the links with the geological, geographical and cultural realities that complete their context of origin. Correct archaeometric investigation must follow the 'four C's' rule and keep in mind the 'complexity' of the artifact, answer 'congruent' questions to the study, aggregate different 'competences' and be open to 'collaborations'.

### 1. Introduction

The use of geological materials as ornaments has evolved over time with a trend parallel to the increase in social complexity (Jessup, 1950; Arrhenius, 1988; Collareta, 2003, 2011; Mottana, 2006; Aimone, 2011; Hilgner, 2017; Bertelli, 2017a; Caselli, 2017). Gemmological materials used in the past can be restricted to a few dozens of mineral species with their many varieties, to some rocks and to a few products of the plant and animal kingdoms (Rapp, 2019). However, these must be juxtaposed with

several artificial and synthetic materials, including glass (De Michele and Manzini, 1993; Henig, 2008; Riccardi *et al.*, 2017), as well as with composite gems and with all those many gems that have undergone treatments to improve their appearance, transparency or colour (Nassau, 1994; UNI 10245: 1993; CIBJO, Confédération Internationale de la Bijouterie, Joaillerie, Orfèverrie des Diamants, Perles et Pierres, 2012).

Given the knowledge of the analytical methods applied to the study of geological materials for gemmological use (*e.g.* Artioli, 2010; Domínguez-Bella, 2012; Carter, 2016; Garrison, 2016), the current chapter is focused on an interpretative approach to these materials and derived objects with the aim of answering historical or archaeological questions. Such an approach goes beyond the simple recognition of the natural (mineral) or artificial phase used in the making of the object itself. When such historical objects are analysed it must be remembered that they represented a ‘status symbol’, and therefore the materials used are often rare in order to exalt this status.

As is the case for all geological materials used in ancient production cycles, including gems and gem materials, it is necessary to deal with the intrinsic complexity of geological contexts (Abdalla and Mohamed, 1999; Krippner *et al.*, 2014; Stutenbecker *et al.*, 2017; Aurisicchio *et al.*, 2018; see also: *The Canadian Mineralogist*, 2017, vol. 55), both in terms of the methodological approach and in the interpretation of data. The most common questions are related to the origin of the materials and, if the material underwent some manipulation with respect to the natural, what was the operational chain to obtain it or to mold it into an object.

The main question that drives archaeometric studies based on gems is identification of their origin. In fact, this information helps to outline routes and contacts for the exchange of goods and thus define trade patterns. This knowledge, together with historical and/or archaeological elements, provides clues to the economic systems and dynamics of societies in different historical periods. What is the path of raw material from its extraction to the use of the gem in an artisan workshop? Were those who extracted the rough gem also those who worked it? If not, where were the workshops? Were these workshops also hubs for the exchange of gems?

The link between object to be made, commissioning and the gem itself is very tight and changes over time. Consequently, the study of the provenance cannot be separated from the knowledge of the local and historical contexts and of the society within which the object and the material is used. Furthermore, it is often revealed that many gems have been used multiple times (De Michele and Manzini, 1993; Superchi, 1999; Calligaro *et al.*, 2007; Riccardi *et al.*, 2017). In the practice of the goldsmith’s art, the gold of old jewels is recast and the gems are recovered and reused in a spiral that we could define as ‘autarchic’. This is also demonstrated by the extreme variability of the dimensions and shapes the new bezels must fit.

A further complication is provided by the ‘provenance of raw materials’ concept itself. The believe behind the studies of the source of any raw materials has been explained in a work concerning a gem material, such as turquoise (Weigand *et al.*,

1977): “it is possible to recognize the source of raw materials if the chemical or mineralogical differences between different natural sources are greater than they are within each source”. This postulate highlights the importance of geological and mineralogical-petrographic knowledge when carrying out provenance studies. Nevertheless, much work remains to be done. Geo-mineralogical studies of mineral formation contexts must be conducted at an observation scale much more detailed than normally done for geological studies, being aware that the material extracted and harvested in the past may not be available today.

An example that can clarify the complexity of an archaeometric study of the origin of raw materials is that of garnets, which were used intensively in antique jewellery and in particular in Merovingian artifacts. Garnets are silicates comprising multicomponent solid solutions with the general formula  $A_3^{2+}B_2^{3+}Si_3O_{12}$ , where  $A$  may be  $Ca^{2+}$ ,  $Mg^{2+}$ ,  $Fe^{2+}$  or  $Mn^{2+}$  and  $B$  may be  $Al^{3+}$ ,  $Fe^{3+}$  or  $Cr^{3+}$ . Garnets are grouped in two series: those in which the  $A$  cation is  $Ca^{2+}$  (uvarovite, grossular, andradite) and those in which  $A$  is not  $Ca^{2+}$  and  $B$  is  $Al^{3+}$  (pyrope, almandine, spessartine). In nature, garnets are found commonly in metamorphic and igneous rocks and as debris in many sediments. Garnets have a broad chemical and geochemical footprint, depending on various geo-petrographic factors: the physical parameters of geological processes (pressure, temperature and time), the type of the parent rock, the composition of fluids, the microstructure of host rock and local chemical-physical equilibrium (microdomains). This compositional variability may be an aspect that complicates the search for sources of supply of raw minerals. In the archaeometric literature, there are many studies dealing with the characterization of the garnets of historical and archaeological objects, adopting non-invasive or micro-invasive analytical techniques (Arrhenius, 1971, 1985, 1988; Greiff, 1999; Hilgner, 2017), such as PIXE (Particle-Induced X-ray Emission) and Raman spectroscopies (Farges, 1998; Calligaro *et al.*, 2002; Dran *et al.*, 2004; Perin *et al.*, 2007; Mathis *et al.*, 2008), electronprobe microanalysis (Velde and Courtois, 1983; Rosch *et al.*, 1997; Quast and Shussler, 2000), X-ray fluorescence (Bimson *et al.*, 1982), X-ray diffraction (Schussler *et al.*, 2001) and laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) (Carter and Dussubieux, 2016; Carter, 2016). An important archaeometric study carried out at the laboratories of the Louvre Museum on >1000 specimens of garnets belonging to Merovingian goldsmithery has enabled the construction of a comprehensive compositional database for garnets of gemmological use. The database has been used to identify five compositional groups (I to V) well defined in terms of their major-element contents, some minor elements and crystalline inclusions (Farges, 1998; Calligaro *et al.*, 2002, 2003, 2007; Perin *et al.*, 2007; Horvath and Bendo, 2011). Comparison with data obtained from the literature concerning the composition of garnets (Greiff, 1999; Quast and Schüssler, 2000) and the reading of historical sources made it possible to identify the areas of origin: almandines of group I and group II come from India, group III from Sri Lanka and group V from the Bohemian Massif. Some uncertainties remain for group IV, which could be of European origin (Calligaro *et al.*, 2007; Carter, 2016). If the definition of the geochemical footprint is completed with the quantification of the minor and trace elements, and if the geological

sampling is expanded, the chemical compositional scenario becomes more complicated (Carter and Dussubieux, 2016): in the geographical area that includes south India and Sri Lanka, almandine garnets, indistinguishable in terms of minor elements, are subdivided into homogeneous groups according to their specific geographical areas of origin. However, the comparison on chemical and geochemical bases of these garnets with those used for the realization of almandine garnet beads of archaeological sites in the south Asia area (Thailand and South India) is not always reflected (Carter, 2016). Some geological sources are still partly unknown. The surprising fact is that all the data confirm that the archaeological garnets constitute very homogeneous compositional groups (Farges, 1998; Calligaro *et al.*, 2007; Horvath and Bendo 2011; Carter 2016; Bugoi *et al.*, 2016). This fact can be the expression of selection upstream of the processing and trade of raw material. A multidisciplinary study of this aspect could outline the criteria for the choice of geological garnets (colour, transparency, shape, dimensions, workability ...) and whether in the past, first- or second-quality materials were traded and for what uses. This would lead to an understanding of the conscious choices made in the past along the production chain of 'luxury goods', entering the minds of operators and artisans who managed part of the operational chain.

## 2. Some historical sources

A thorough examination of historical sources is of paramount importance in tackling the longstanding issues concerning the origin of gems. Archaeological studies are no less important, in particular those concerning the sites of departure, transit and arrival of luxury goods such as gems.

In analysing historical sources, the ambiguity of language used by different scholars may be an obstacle; for some, a given term defines a very precise mineral, while for others the same term represents a series of geological materials, similar in colour or ornamentation. Some terms are not easily interpretable.

In the Europe of the early Middle Ages, the first and only reliable written evidence on the eastern origin of certain gems is the *Christian Topography* by Cosma Indicopleuste (Wolska-Conus, 1968), a writer of the first half of the 6th century AD. A navigator, connoisseur of the Red Sea and the Persian Gulf, Cosma was stationed in the port and commercial centre of Adulis (60 km southeast of Massaua in Eritrea), the centre of commercial traffic not only with the Indies, but also with the southeastern and perhaps Kenyan–Somali coasts of Africa. Cosma was the first to record that the 'alabandinon' came from the east coast of India, and the 'hyacinth' from Taprobane, Sri Lanka. Important and extensive analytical researches on early medieval garnets of the Rhenish area led S. Greiff to conclude that the deposits of red garnet were indeed in India and Sri Lanka (Greiff 1999).

The testimony of Cosma confirms and completes what was written centuries earlier by Pliny the Elder, but to obtain more precise indications, it is necessary to read the Arab writers of the 8<sup>th</sup>–9<sup>th</sup> centuries, obviously unknown to Latin contemporaries and rediscovered starting from the 18<sup>th</sup> century. The famous doctor Yuhanna Ibn Masawayh

(777–857), known in Europe as Mesue, who lived in today's Iraq, published a book on gems and their characteristics, printed in Arabic in Cairo only in 1977 and translated into French in 1998 by G. Troupeau (1998). The deposits of India and Sri Lanka are described there, also recalling that on that island they were located at the foot of Adam's Peak (Adam Peak or Sri Pada, 2243 m), where the rich gem district of Ratnapura occurs.

More exhaustive is the treatise on gems of Abu al-Rayhan Mohamed ibn Ahmad al-Biruni (973–1048), translated by Fritz Krenkow in 1936 from the existing manuscripts, and since then reprinted several times in Muslim countries (Al-Beruni, 2007). Philosopher, scientist and traveller, Al-Biruni had direct knowledge of the Indian and Sinhalese deposits and also introduced the use of density for better characterization of gems. His essay confirms the origin of the garnet and sapphire sold in those countries around the year 1000.

The Egyptian monopoly of the emerald trade was witnessed widely by the aforementioned Muslim writers and by Al-Masudi (896–956), historian, geographer and traveller, who lived in today's Iraq. In his monumental work, entitled *The Meadows of Gold and Mines of Gems*, known since the 19th century (Macoudi, 1861), important references are also found to the Egyptian emerald mines, still active at that time.

Thanks to the exceptional discovery of the 'Geniza' annexed to the synagogue in Cairo (a deposit of documents discarded but not destroyed, because of the Holy Name), S.D. Goitein and collaborators have published letters and documents from the centuries immediately after the year 1000. In these documents, are highlighted the dense network of commercial relations among the Egyptian Jewish community, often in societies with Muslim and Christian operators, with other communities scattered throughout the Mediterranean (Goitein, 1973, 2002; Goitein and Friedman, 2011).

### 3. The study protocol

The identification of the types of gems present in a historical artifact must follow a non-invasive approach. Very often the object cannot be transported from the place where it is stored (museum) to the laboratory, and this further limits the investigation protocol (the 'don't move, don't even touch' protocol). Traditional gemmological analysis is a method of study that meets the needs described above. It is also a quick, economic and non-destructive method to identify the gems found commonly in historical artifacts with sufficient security. It represents the premise of more complex investigations. Gemmological investigation is aimed at detecting certain simple physical parameters of minerals, rocks and other specimens.

If the gems are 'loose', *i.e.* not mounted in jewellery or other objects, it is possible to make optimal observations (*e.g.* measurement of density and refractive index). In most cases, however, the gems are mounted and cannot be removed from their settings other than for restoration needs. In case of mounted gems, the data collected are necessarily incomplete and this leads to a considerable level of uncertainty in the diagnosis. Fortunately, the ancient and medieval jewellery artifacts, especially the more voluminous ones, have only a few mineral species embedded, such as red garnet,

quartz in all its varieties, corundum, and beryl, easily identifiable even from partial detection of their physical properties.

In the two case studies illustrated below, gemmological analyses were carried out using common gemmological instruments: stereoscopic microscope with fibre optic illumination; refractometer; spectroscope; calcite dichroscope; polariscope; Chelsea filter; and short (256 nm) and long (360 nm) UV lamps. For a brief description of these instruments refer to Manutchehr-Danai (2009). The nomenclature adopted is that stipulated in the norms UNI10173 and UNI10245 with minor discrepancy concerning the aspect of cutting forms, not fully considered in the Standards. The evaluation of colour, for reasons of brevity, does not always correspond to the denominations contained in the reference standard (UNI9810). Measurement of density and/or refractive index is fundamental for the recognition of the gemmological species. One example is monocrystalline quartz in its most common varieties (colourless, citrine, amethyst). To facilitate this measurement, in many cases a minimum intervention in terms of the data collection (density and/or refractive index) of the settings was authorized.

The analytical protocol, therefore, started with the measurement of dimensional parameters by using digital callipers: the size, shape and type of cut of gems are important indications to evaluate (or estimate, in case the stone is still mounted) the carat weight. The use of the optical microscope or, where not possible, of a triplet lens has allowed determination of the conditions of the collet and to identify inclusions and/or defects in the gem, as well as the condition of its surface. The observation of the gem and its inclusions is a fundamental step: the association of inclusions represents an ‘identity card’ of the gem and sometimes proves decisive in defining its origin (Figs 1 and 2).

When possible, the measurements of refractive index and absorption spectrum have been associated with these observations. The refractive index is a discriminating measurement used to recognize a large number of gems by simply determining whether a gem is monorefringent or birefringent. Absorption spectra have been observed through a manual spectroscope (Fig. 3). They are diagnostic in the identification of the mineralogical species as well as of the gems embedded in an ancient jewel or artifact. Use of a spectroscope will distinguish a ruby from an almandine immediately (despite both being red) (Fig. 3a, c), an emerald from an olivine (green), or a sapphire from a synthetic blue spinel (Fig. 3b, d).

Note that, when studying gems of a complex artifact consisting of several materials, such as ancient goldsmith objects, the correct interpretation of the gems within the artifact also depends on the knowledge of other materials, metals above all, and on the set of historical and archival documents referring to the same object.

#### **4. Case study: the Iron Crown**

The Iron Crown (Fig. 4), so called for a dark grey metal strip on the inner side and considered to be one of the nails of Christ’s cross, is kept at the altar of the Teodolinda chapel, in the Cathedral of Monza, Italy. Even today, it remains an enigmatic object in its historical interpretation.

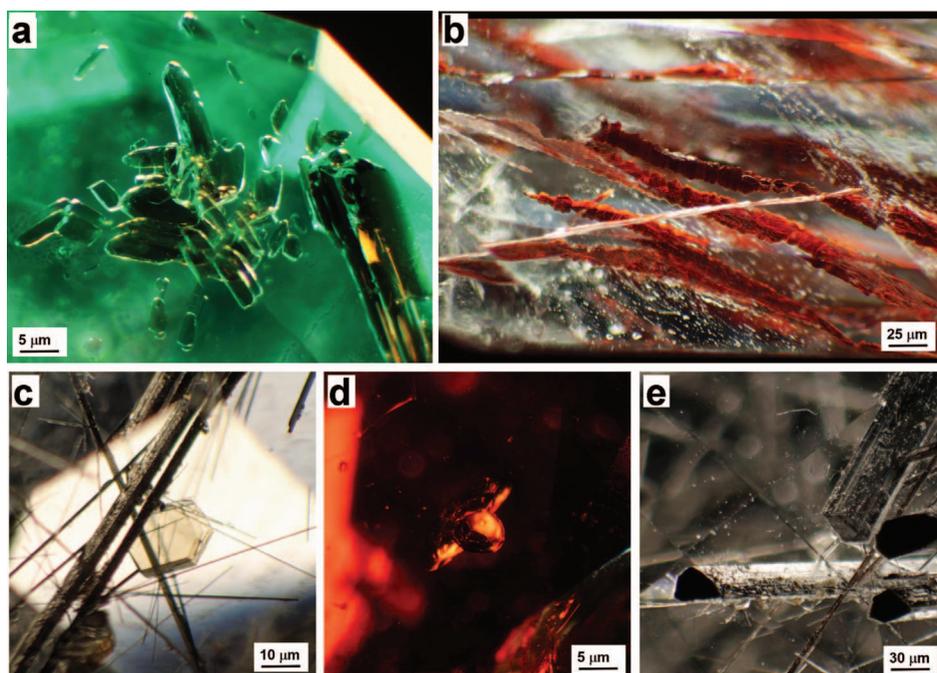


Figure 1. Optical microscopy images, dark field, of some inclusions in gems: (a) biotite in emerald; (b) hematite in quartz; (c) mica and rutile in quartz; (d) zircon with tension halos in garnet; and (e) tourmaline in quartz.

An interdisciplinary study was conducted over the years 1994 and 1995 (Lusuardi Siena, 1998; Bertelli, 2017b). The results of scientific investigations have raised new hypotheses and doubts about both the history of the object itself and its historical

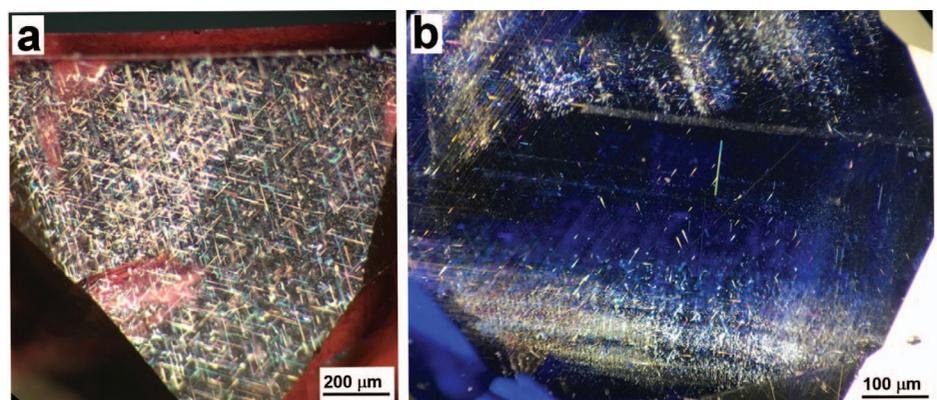


Figure 2. Optical microscopy images, dark field, of some inclusions in gems: (a) rutile in ruby and (b) rutile in sapphire.

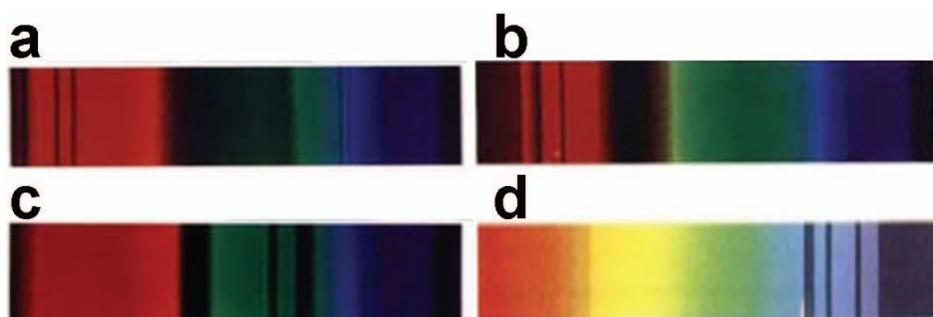


Figure 3. Examples of absorption spectra of some gems: (a) ruby; (b) emerald; (c) almandine garnet; and (d) olivine.

interpretation. The Crown shows signs of restorations to the structure, replacement of glazed plates and likely substitutions or perhaps only temporary removals of the gems. The questions about its interpretation which remain unresolved are summarized in the work of Bertelli (2017a,b), which provides an update on the historical studies of the object.

The Iron Crown is composed of six rectangular plates, P1–P6, attached to one another with their edges coffered on the outside to hold decorative enamels, rosettes and gems (Lusuardi Siena, 1998; Maspero, 2003, 2016). Plate P1 shows only two gems (Fig. 5), one placed in the centre with four rosettes and four enamels around it, the other is located between two rosettes in a narrow lateral strip. On each of the other five plates (Fig. 5 for P2 and P3; Fig. 6 for P4–P6) there are four gems: again, one occupies the



Figure 4. The Iron Crown (diameter 15 cm and height 5.5 cm) (photo by G. Monistier).

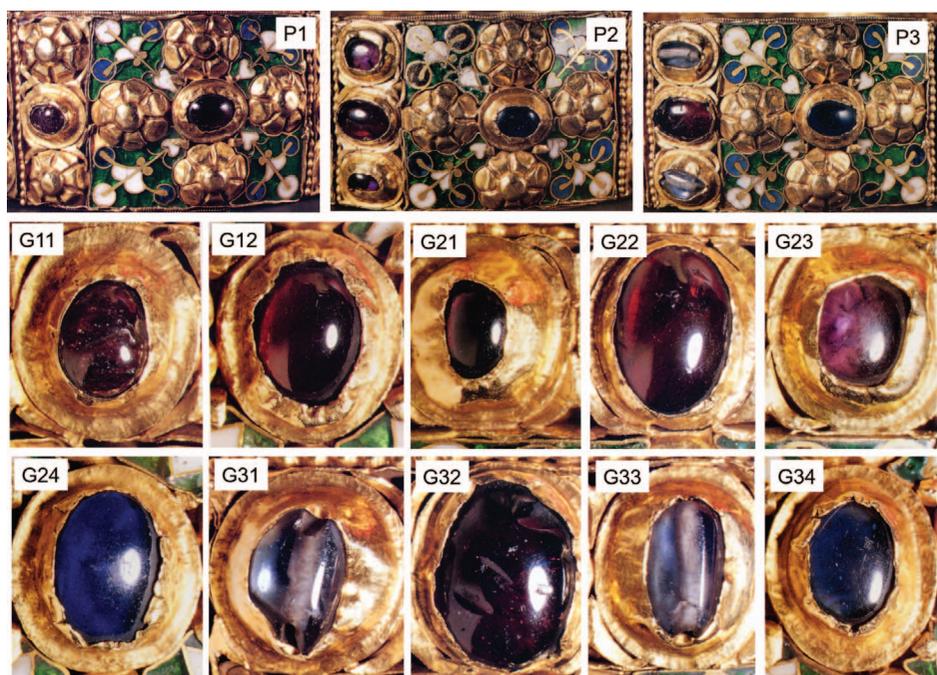


Figure 5. Plates P1, P2 and P3 of the Iron Crown and related gems. The dimensions of each element are given in the text and in Table 1 (photos by G. Monistier).

central position amongst four rosettes and enamels, while the other three are arranged vertically in the narrow lateral sector. We note that the Crown is currently assembled with plates P1, P2 and P3 (Fig. 5) with the jewelled border on the left, and P4, P5 and P6 (Fig. 6) with the jewelled border on the right. P1 and P6 are facing each other on their jewelled border sides (Fig. 4). Historians have hypothesized that the plates were originally assembled with the border all on the same side, and that the Crown had at least one more plate (Marimonti, 1841; De Michele *et al.*, 1986). All the gems are fixed with semi-occluding truncated cone settings with a wide collar, very similar to those of the middle border on the reliquary of Saint John the Baptist (Monza) and the reliquary-bag of Saint Stephen (Vienna), both attributed to the 9<sup>th</sup> century.

There are 22 gems set on the crown (Figs 5 and 6): corundum (7), garnet (7), quartz (4) and artificial glasses (4).

*Corundum*: The corundum gems in the crown are quite light-coloured and almost all are perforated longitudinally (Table 1). The hole shows helicoidal marks left by the drill, and in the case of gem G31 (Fig. 5) there is some encrusted material which, if analysed, might identify the abrasive used. This thus suggests that it is again a case of recycled material, with physical characteristics that may allow determination of its place of origin in Sri Lanka (Hughes, 1990; Gubelin and Koivula, 1992). Stone G42 (Fig. 6) has, in fact, kept the typical pebble shape of products from the gemmiferous gravels of this island.

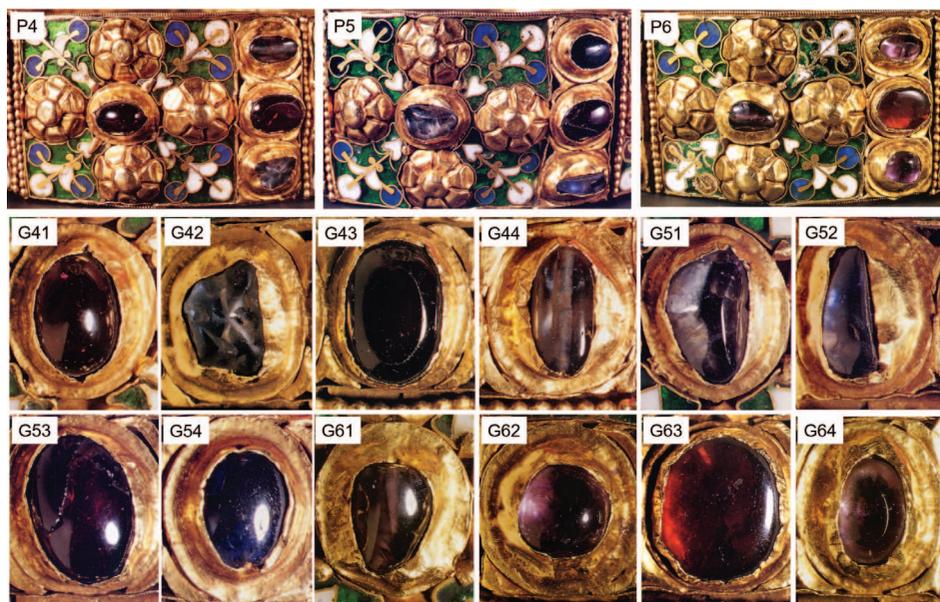


Figure 6. Plates P4, P5 and P6 of the Iron Crown and related gems. The dimensions of each element are given in the text and in Table 1 (photos by G. Monistier).

**Garnets:** The garnets of the Iron Crown are pyralspite (rhodolite), very close to almandine, as borne out by the refractive index and absorption spectra (Gunther, 1988; Stockton and Manson, 1985; Rouse, 1986). Inclusions indicate that the stones are from Sri Lanka (Gubelin and Koivula, 1992). The presence of surface grooves might be interpreted as a way to remove showy and heavy solid inclusions and give vivacity to the gem, but they are generally indicative of a re-use of the material.

**Quartz:** Quartz appears in an amethyst variety of a light violet colour (Table 1). One of the four specimens has an oval shape with mixed cabochon and table cut (Figs 5 and 6).

**Glasses:** Three specimens are coloured blue (from cobalt, as shown by the absorption spectra), and one is orange with a mass of aligned bubbles.

The gems of the Iron Crown have undoubtedly suffered losses and substitutions, as signs of damage to the gold lamina of the mountings testifies. Moreover, Fontanini, in the third edition of his *Dissertatio* (Fontanini, 1719?) writes that in 1299 “*unus lapis pretiosus*” was missing, having been substituted with a triangular stone which scarcely matched the quality of the others. Moreover, Bellani (1819) recorded that in the inventory drawn up in 1353, the Crown was found to have seven gems missing, yet at the same time the treasure included batches of loose gem-stones. Because the shapes of cuts underwent no changes until the 1500s, recognizing a substitution means relying on a reconstruction of the original chromatic pattern assigned to the gems and enamels. However, if the substitution respects the original colour of the gem to be replaced, even this criterion becomes invalid.

Table 1. Gemmological characteristics of the gems of the Iron Crown.

| Gem | Dimensions (mm) | Colour           | Clarity | Morphological and textural features | Measures          | Notes                           | Classification |
|-----|-----------------|------------------|---------|-------------------------------------|-------------------|---------------------------------|----------------|
| G11 | 10 × 7.8        | Red              | TP      | oval; cabochon                      | AS; RI            | fractures                       | garnet         |
| G12 | 13.5 × 9        | Red              | TP      | oval; cabochon                      | AS; RI            |                                 | garnet         |
| G21 | 9.8 × 7         | Purple           | TP      | oval; cabochon                      | RI                | inclusions                      | amethyst       |
| G22 | 16.8 × 11       | Red              | TP      | oval; cabochon                      | AS; RI            | inclusions                      | garnet         |
| G23 | 11 × 9          | light purple     | TP      | irregular olav; cabochon            | RI                |                                 | amethyst       |
| G24 | 12 × 8.2        | Blue             | TP      | oval; cabochon                      | AR; RI            |                                 | glass          |
| G31 | 13.5 × 9        | pale blue        | TP      | oval; cabochon                      | RI                | crosswise hole                  | corundum       |
| G32 | 17 × 12         | Red              | TP      | oval; cabochon                      | AS; RI; incl.     | superficial grooves, inclusions | garnet         |
| G33 | 14 × 7          | pale blue        | TP      | oval; cabochon                      | AS; RI            | crosswise hole                  | corundum       |
| G34 | 14 × 10         | Blue             | TL      | oval; cabochon                      | AS                |                                 | glass          |
| G41 | 11.8 × 9.5      | Red              | TP      | oval; cabochon                      | AS; RI; incl.     |                                 | garnet         |
| G42 | 12 × 8/8.5      | pale blue        | TL      | irregular olav; cabochon            | H > 8             | crosswise hole                  | corundum       |
| G43 | 16 × 12         | Red              | TP      | oval; cabochon with table           | AS; RI            | inclusions                      | garnet         |
| G44 | 14 × 7.5        | pale blue        | TP      | oval; cabochon                      | AS; RI            | crosswise hole                  | corundum       |
| G51 | 14 × 9          | pale blue        | TP      | irregular olav; cabochon            | AS; RI; FL        |                                 | corundum       |
| G52 | 14.5 × 5.5      | pale blue        | TP      | irregular oval                      | RI; FL            | fracture; crosswise hole        | corundum       |
| G53 | 15.5 × 11       | Red              | TP      | oval; cabochon                      | AS; RI            | fracture, inclusions            | garnet         |
| G54 | 12 × 9.5        | Blue             | OP      | oval; cabochon                      | AS                |                                 | glass          |
| G61 | 12.5 × 5/8      | pale blue-violet | TP      | irregular oval                      | AS; RI; incl.; FL | crosswise hole                  | corundum       |
| G62 | 9.5 × 8.5       | light purple     | TP      | oval; cabochon                      | RI                |                                 | amethyst       |
| G63 | 16.5 × 11.5     | Orange           | TP      | oval; low cabochon                  | FL                | Inclusions                      | glass          |
| G64 | 12 × 7.5        | light purple     | TP      | oval; cabochon with table           | RI                |                                 | amethyst       |

TP: transparent; TL: translucent; OP: opaque; AS: absorption spectrum; RI: refractive index; H: hardness; FL: fluorescence.

Certainly, identification of the materials constituting the Iron Crown has not solved all the interpretative problems that even today keep scholars busy dealing with medieval goldsmithery. The small number of gems, many of which are reused from older artifacts, the association of a few varieties of minerals, such as garnet, quartz (amethyst) and corundum, as well as the use of glass, show clearly that it is not the gems which are the 'star' materials but probably the enamels, which are very abundant in all the elements of the Crown and the metals.

For these materials and for metal alloys, data collected using portable XRF are available (Milazzo and Ciccardi, 1998). These first data, as often happens in archaeometric studies, raise new doubts: the composition of metals showed that, despite the name, in the Crown there is no iron and that the inner lamina is made of silver. The enamels in the slabs of the most ancient series (Lusuardi Siena *et al.*, 1998; Milazzo and Ciccardi, 1998) seem to have been made with a potassium flux, while the restoration plates show the use of a sodium flux. At the same time, the glazes of the oldest slabs have tin as opacifier, while those of restoration contain Ca antimonates; the cobalt-blue coloured restoration enamels also contain Zn, perhaps associated with Pb. This in turn would indicate north European mines as a source of supply (Gratuze *et al.*, 1992), known only from the 10<sup>th</sup>–11<sup>th</sup> centuries (De Launay, 1913). Could the constant presence of Pb, detected by surface measurements, be justified by the intrinsic enamel composition or by the superficial processing of glazes?

### 5. Case study: King Tutankhamun's pectoral and Libyan Desert Glass

Libyan Desert Glass (LDG) (Fig. 7) is a natural glass consisting mainly of silica (SiO<sub>2</sub> ~98 wt.%) and traces of Al, Fe, Mg and other metals (Fudali, 1981; Koeberl, 1986, 1997; Barrat *et al.*, 1997; Greshake *et al.*, 2010, 2018). The H<sub>2</sub>O content is ~0.1 wt.%. It was P.A. Clayton, in 1932, who first saw and reported the presence of abundant fragments of glass, some large, in the western part of the Egyptian desert, in the area between the Siwa oasis to the north and the Gif Kebir plateau in the south (Clayton, 1933, 1998; Clayton and Spencer, 1933, 1934). LDG can be opaque, translucent or transparent; the colour is mainly greenish yellow and variable from dark green to blackish.

The raw material can be found on the surface, in the inter-dune corridors of the Great Sand Sea, as fragments of various sizes, sometimes even >30 kg, with the surfaces worked by the wind. Flaking is absent and the fracture is conchoidal.

Prehistoric man used LDG in artifacts (Oakley, 1952; Roe *et al.*, 1982; Negro and Damiano Appia, 1992). Despite its local use in pre-dynastic times, this glass does not occur in historical Egyptian objects (Lucas, 1962).

LDG is not an important gemmological material, but because of its likely genesis, it has taken on a very strong symbolic meaning. Apart from the first gemmological indication by L.J. Spencer in 1933 in the journal *The Gemmologist*, and an extended quotation in a gemmological text (Spencer, 1936), the glass of the Libyan Desert has not been mentioned further. The reasons for this are its scarce use in historical goldware



Figure 7. Chromatic and textural varieties of Libyan Desert Glass (photos by V. De Michele).

artifacts, and, in part, its widely agreed lack of aesthetic properties. To obtain gems with intense but bright colours (brown, yellow-brown, brown-green), macroscopic samples showing dark a colour, almost black, must be chosen, while rough samples of a beautiful green colour are weakly yellow or even colourless after the cut (Fig. 8). The commercial value of the LDG does not reside in aesthetic properties, therefore, but in its symbolic meaning, as discussed below.

The discovery of the use of this material for the funeral of the great Egyptian king Tutankhamun was the result of a happy intuition, almost a doubt, during the examination of King Tutankhamun's pectoral (Fig. 9) held at the Cairo Museum (De Michele, 1998). The scarab-shaped central motif of one of Tutankhamun's pectoral



Figure 8. Gems obtained from some Libyan Desert Glass (photos by V. De Michele).



Figure 9. King Tutankhamun's pectoral (photos by J. Bodsworth/Wikipedia).

(14<sup>th</sup> century B.C.) was carved from a material strikingly similar to LDG in appearance. The pectoral with the large translucent green-yellowish scarab (18 mm × 28 mm) was found by Howard Carter in a chest (n° 267) in the so-called 'treasury' room beyond Tutankhamun's burial chamber (Carter, 1972). The true nature of the scarab material was not established with certainty. In book texts, this material was classified as chalcedony, a 'fibrous' variety of quartz (Desroches-Noblecourt, 1963; El Mallakh and Brackman, 1978; Aldred, 1979; Saleh and Sourouzian, 1987; Andrew, 1990). The material is cloudy, translucent, and shows a vitreous lustre. The lines showing the insect anatomical shape are fine, sharp grooves, indicating that the raw material is harder than the 5<sup>th</sup> degree on Mohs' scale. These features are compatible with chalcedony, but on the other hand, the colour is not compatible with any known variety of chalcedony.

The colour is a peculiar feature: fairly intense green-yellow, known by those who have had the opportunity to examine the glass pieces in the LDG area. By using optical fibres and a 10 × loupe, and by observing the textural details in the scarab, flow texture

is observed, accompanied by small, round, white inclusions, very similar to spherules of cristobalite occurring in LDG (Fig. 10). The scarab is optically isotropic. The refractive index of the scarab's curved surface was measured using a refractometer (liquid  $n = 1.79$ ) with the spot method. The spot method is a rapid method to read the refraction index on facets or cabochon-like curved surfaces. Precision is low, though the index values can be useful for identification. The refractive index of LDG was measured firstly by Spencer (1933; 1936), who obtained, by sodium light, a value of 1.4624; Weeks *et al.* (1984) obtained values ranging from 1.4590 to 1.4650 using the Becke line test. The refractive index measurements were particularly difficult because of the large size of the pectoral, therefore they were repeated several times and gave average values of  $\sim 1.48$ . In comparison, the refractive index of a fairly intense green-yellow LDG cabochon was measured using the same method and instrument and a similar value of 1.47–1.48 was obtained. Subsequent measurements conducted on other LDG products were carried out with the same refractometer and registered values ranging between 1.462 and 1.470. By considering the values reported in the literature and those measured on the artifact and on materials collected *in situ*, it can be concluded that the refractive index of LDG can vary from 1.459 to 1.470, according to the bulk chemical composition as well as the textural variations of the material itself (Table 2).

The data collected automatically exclude chalcedony, which would have registered a value of 1.53–1.54 (Table 2). Similarly, green Pakistan alabastrite, which is a concretionary calcite, can be excluded. Other minerals characterized by a similar green-yellow colour show refractive index values very different from LDG (Table 2). Moreover, artificial glass would give a minimum value for the refractive index of 1.50–1.52, therefore an unlikely green obsidian, never found in Nature, can be also excluded.

Autoptic observation, inclusion and optical properties therefore point to LDG as being the raw material used to carve Tutankhamun's scarab. This material obviously went through the Nile Valley from the remote area  $>700$  km away, to be used as a rarity

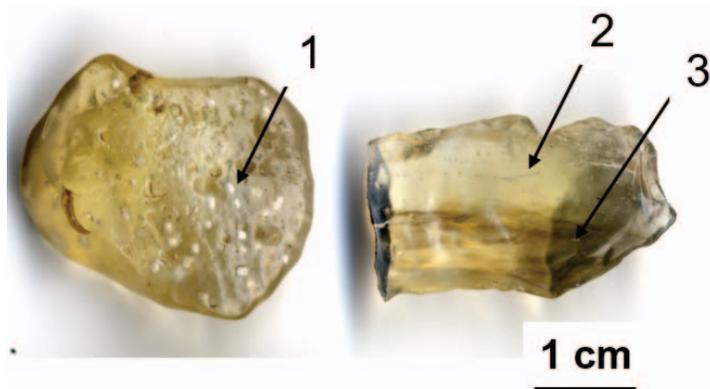


Figure 10. Textural details of some samples of Libyan Desert Glass: (a) green-yellowish variety with inclusions of cristobalite (1); and (b) transparent green-yellowish varieties with a fluid texture showing an alignment of bubbles (2) and dark brown bands (3).

Table 2. Gemmological materials with a yellow/yellowish tinge similar to that of Libyan Desert Glass.

| Mineral  | Refractive index | Hardness |
|--|------------------|----------|
| ————— Singly refracting materials —————                |                  |          |
| LDG  | 1.459–1.470      | 6        |
| Fluorite   | 1.434            | 4        |
| Opal   | 1.44–1.45        | 5–6      |
| Obsidian   | 1.49–1.50        | 5.5      |
| Glass (artificial)                                     | 1.50–1.70        | 5        |
| Hydrogrossular var Transvaal ‘Jade’                    | 1.72–1.73        | 7        |
| ————— Double refracting materials (spot methods) ————— |                  |          |
| Talc var. Steatite                                     | 1.54             | 1–2.5    |
| Chalcedony   | 1.53–1.54        | 7        |
| Serpentine   | 1.56–1.57        | 4        |
| Muscovite  | 1.58             | 3        |
| Pyrophyllite var. Agalmatoite                          | 1.58–1.59        | 1        |
| Nephrite and other Actinolites                         | 1.6–1.61         | 6        |
| Prehnite   | 1.63             | 6        |
| Apatite  | 1.64–1.65        | 5        |
| Alabastrite  | 1.64–1.65        | 5        |
| Jadeite  | 1.66             | 6.5      |
| Vesuviana var. Californite                             | 1.72             | 6.5      |

\* Eppler, 1984; Gunther, 1988; Author’s personal observations

by the New Kingdom pharaohs. This finding from Tutankhamun’s tomb is evidence of the appreciation the young pharaoh reserved for the latest technological discoveries and resources of the mineral world.

The extraterrestrial nature of the iron used to make King Tutankhamun’s dagger, suggested in a recent paper (Comelli *et al.*, 2016), and perhaps also the other 19 iron objects (Carter, 1996), offers an imaginative and romantic suggestion about the choice of materials for the king’s grave goods, a choice linked with strong symbolism, perhaps to indicate the long journey of the king after his death. There were also unique funerary objects for kings of the Bronze Age.

## 6. Concluding remarks

The archaeometric study of historical artifacts, and in particular of the goldsmith’s objects, has the merit of highlighting complex problems related to the characterization of objects and materials, to which doubts and questions are added. This is because,

although it is sometimes forgotten, the methods of science, as well as literary acquisitions, open new horizons for investigation, rather than immediate certainties. However, this concept is included in the same definition of 'History', which is revived when there is convergence of attentions from a plurality of points of view.

Thus, while on the one hand, the materials offer interesting insights for the understanding of the object, on the other hand, they ask for further in-depth study, not only for the understanding of their intrinsic characteristics, closely related to the choices made for their use, but also in terms of their geological, geographical and cultural realities that complete their context of origin.

For a correct investigative method, we suggest here the 'four C's' rule (Fig. 11):

- *Complexity*: never forget that the object of study is complex, has a long history, is almost always poly-material, and each material preserves the memory of human and technical processes that include many variables. The reconstruction work itself is complex: investigators must take into account changes in time scale and changes in landscapes and territories. Materials are expressions of technical knowledge, even in their choice and express the historical memory of the communities. An object is always the communication of the strong link between territory, community and culture.
- *Congruity*: ask the right question with respect to the objectives of the investigation, and the right question must be calibrated in a highly interdisciplinary context.
- *Competence*: everyone contributes to the survey with their own specificities and the baggage of their personal and professional experiences.
- *Collaboration*: being open to other points of view and receptive to other disciplines.

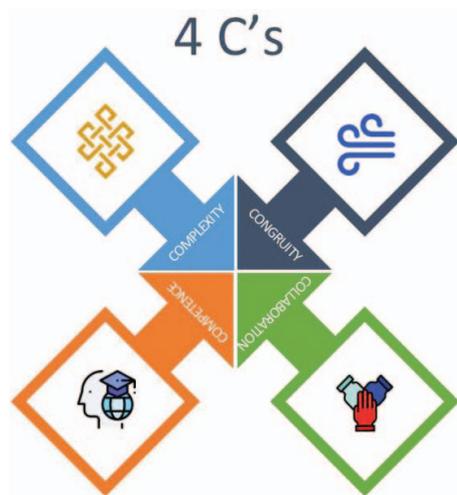


Figure 11. The four C's Rule.

In this perspective, the definition of ‘archaeometry’ (Mannoni, 1990) is still current and appropriate: “The more you get rid of the blinders of the history of idealistic art, and study using the scientific method any type of product, even those considered trivial, the more you realize that almost always the choices of the materials were optimal in relation to the resources of a given territory, and that already in the prehistory, very rare materials, which even today can be considered the best to produce the objects for which they were intended, were sought and exchanged over very long distances”.

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