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RECOGNITION OF ANCIENT FIRES IN ARCHAEOLOGICAL SITES CONTAINING GYPSUM ROCKS

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Abstract

The presence in archaeological sites of architectural elements composed of gypsum rocks (selenite, gypsarenite and gypsrudite) is helpful to reveal traces of ancient fires because a characteristic white layer develop on gypsum surfaces directly exposed to temperatures in excess of 100° C. The white surface is generally up to a few centimetres thick and is the consequence of gypsum dehydration to form a mosaic of microcrystalline bassanite, soluble anhydrite and/or anhydrite, depending on the temperature. With the exception of anhydrite, all phases rehydrate rapidly back to gypsum because of atmospheric moisture and/or groundwater capillarity rise along the walls. The shape of the original gypsum crystal outline is still recognizable, but the rock fabric is now composed of a mosaic of secondary, white microcrystalline gypsum. Anhydrite may also be present if the surface temperature during the fire was in excess of about 252° C.
The final result of gypsum heating appears indistinguishable from natural dehydration, which may occur because of several geological processes, such as diagenesis and burial. In these cases, however, the white areas are massive or have nodular shapes and obviously formed before the rocks were quarried. Criteria to distinguish between artificial and natural dehydration are thus easily applicable. White surfaces caused by fires develop only on exposed face of blocks which were not shielded from the heat, are thin, and correlate across adjacent blocks showing different gypsum facies and crystal orientation.
The geological survey of gypsum facies in the source outcrops may reveal the presence of natural dehydration features, which may be misinterpreted as the effect of fires.
The spatial extent of correlable white surfaces in the buildings, their variable thickness and composition, may give indications on the severity of damages caused by fire events, providing significant informations to unravel the evolution of archaelogical sites.

Keywords: GYPSUM, ANHYDRITE, DEHYDRATION, BOLOGNA, ERACLEA MINOA, CRETE.

Introduction

Rocks consisting of gypsum (calcium sulphate bihydrate) are easy to cut and work and are available in many areas around the Mediterranean region, where Messinian and, more rarely, Permo-Triassic, Tortonian and Quaternary evaporites crop out. These characteristics favoured the use of Messinian gypsum for building purposes in a number of historical sites, such as the Roman and Middle-Age Bologna (Italy), the Greek and Roman Eraclea Minoa (Sicily; LUGLI, 1997) and the Minoan Palaces of Crete (Greece; Chlouveraki, this issue).

Gypsum blocks and slabs in archaeological sites can be used as indicator of ancient fires because relatively low temperatures, leaving no visible traces on bricks and other stones, may affect calcium sulfate in a permanent and recognisable way (*fig. 1*). A white, thin, layer forms on the surface of blocks exposed

Fig. 1 – Partially dehydrated gypsum block (white, center) located in a wall structure at Eracle Minoa (Sicily). The other gypsum block immediately on the right is unaltered, whereas the thickness of the white surface decreases toward the right, suggesting that a fire burned the left side of the wall decreasing in intensity and effectiveness to the right. Note that the other adjacent rocks, arenite (deeply altered, at bottom) and limestone (left), do not show traces of burning. The gypsum blocks are about 30 cm high.

to temperatures in excess of about 100° C. The «whitening» of gypsum rocks may be also a consequence of natural causes, but can be readily distinguished from post-constructional events because this phenomenon may occur only before the rocks were quarried and emplaced in the building.

The peculiar characteristics of gypsum rocks may provide a significant contribution to the interpretation of the evolution of archaeological sites, because it may help to demonstrate the occurence of extensive accidental or intentional fires. The purpose of this paper is to describe the development of white surfaces on gypsum rocks as result of burning and to provide criteria to distinguish the effect of fire from natural dehydration.
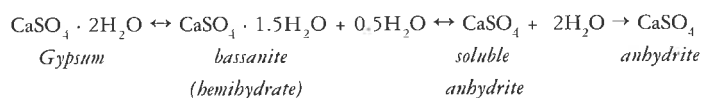
Characteristics of the white surfaces

The white surfaces are planar, generally less than 5 mm-thick (*fig. 2*) and affect totally or in part exposed faces of the blocks, which normally show a gray color (selenite, gypsarenite and gypsrudite). The boundary of the dehydrated surfaces commonly match across adjacent blocks of different shape and size showing different crystal orientation (3 and 4). These characteristic clearly indicate that the whitening should have occurred after the emplacement of the blocks. Moreover, the white surface commonly affect, and thus postdates, remnants of scretched lines produced by carving tools.

The white areas show the same macroscopic characteristic of the unaffected surfaces of the gypsum blocks because the shapes of selenite crystals are still recognizable (pseudomorphs), made evident by enclosing dark envelopes of clay or microcrystalline carbonate, which are present also in the unaffected zones. In some cases the white surface boundary cuts directly across arrow-head gypsum twins, so that the only difference between the two parts is that one appears white and the other gray-translucent (unaffected crystals).

Examination at the optical microscope of some examples shows that the white surface is made of microcrystalline gypsum (alabastrine gypsum) pseudomorphs after selenite single crystals containing small anhydrite crystals. The cleveage planes from the original gypsum and also the impurities located in the core of the unaffected crystals, such as clay and carbonate, are still recognizable in the microcrystalline white groundmass. The observation of selenite monocrystals only partially affected by the white surface shows that the mycrocrystalline mass start to grow along cleveage planes and crystal boundaries. The described characteristics are the same that can be observed in gypsum rocks experimentally heated to temperatures in excess of 100° C.

Origin of the white surfaces by artificial dehydration of gypsum

Gypsum blocks directly burnt or exposed to temperature in excess of 100° C develop white surfaces. The white surfaces formation is due to thermal dehydration of gypsum to form a mosaic of fibrous microcrystalline hemihydrate, soluble anhydrite and/or anhydrite, depending on the maximum surface temperature, according to the following reactions:

$$CaSO_4 \cdot 2H_2O \leftrightarrow CaSO_4 \cdot 1.5H_2O + 0.5H_2O \leftrightarrow CaSO_4 + 2H_2O \rightarrow CaSO_4$$

| Gypsum | bassanite (hemihydrate) | soluble anhydrite | anhydrite |

Abr iel and Reisdorf (1990) investigated the transition temperatures by heating powdered gypsum with particle size <50 μm. Subhydrates (bassanite) form from gypsum starting from 57° C. At 101° C gypsum has been totally consumed and at 207° C soluble anhydrite starts to form. Stable anhydrite starts to form at 252° C and coexists with soluble anhydrite up to 352° C. At temperatures above 352° C only stable anhydrite is present.

The transition temperatures are variable depending on steam pressure, crystal size and probably crystal orientation. Because dehydration proceed starting from crystal boundaries

Fig. 2 – Top view of a partially dehydrated gypsum block in the Palace of Minos at Knossos (Crete). Note the white surface running parallel to the vertical face of the block (left). The maximum thickness of the white surface is about 4 cm.

and cleveage planes, as suggested by observation at the microscope of experimentally heated gypsum, when large size gypsum crystals in a coherent rock are heated, the transition temperatures should necessarily be higher. Experiments better resembling the behaviour of «burning» gypsum in a building are probably short-term differential analyses (DTA) in air. Popp and Kern (1993) showed that a natural fine-grained polycrystalline gypsum rock composed of crystals ranging from 0.1 to 1 mm in size starts to dehydrate rapidly at around 100° C, value which is significantly higher than 57° C. No data are available to evaluate the shifting of the transition temperatures on crystalline rocks like the Messinian selenite, gypsarenite and gypsrudite facies composed of crystals ranging from millimetres to decimenters. In particular, no data are available to evaluate the temperature at which stable anhydrite starts to form. The stable anhydrite formation temperature is probably higher than 252° C (Abriel and Reisdorf, 1990), value which can be considered as the lower limit of a range with unknown upper limit.

The observation that in the studied sites the thickness of white surfaces is generally limited to a few centimeters suggests that vaporization of the crystallization water may significantly slow down the rate of temperature rise into the rock. Until the liberated water is not completely vaporized, the block surface remains at a temperature of about 100° C retarding the initiation of the reaction deeply into the rock. The reaction penetration rate is probably reduced also by the growing dehydrated layer on the surface. It is not clear at present how the reaction penetration can be influenced by time. Single blocks completely covered by a thick burning detritus may remain at temperatures above 100° C for a long time (days?) and may possibly develop a thick dehydrated coat. No data are available to evaluate how thick this coat could grow in a burning building and how thick a white surface can eventually grow on a block encased in a wall during a particularly severe fire.

Fig. 3 – Partially dehydrated gypsum blocks (white) in the western façade of the Palace of Minos at Knossos (Crete). Note that the boundary of the white surface correlates across different blocks, indicating that the blocks were dehydrated after their emplacement. The lower portion of the gypsum blocks are unaltered (dark gray), suggesting that the socle may have acted as a sort of shield against the fire («shield shadow»). This interpretation is supported by the observation that the gypsum blocks are completely affected by the white surface in those tracts where socle is missing (not visible on picture). The gray areas on gypsum blocks are concrete restorations. Vertical blocks are about 60 cm high.

Bassanite and soluble anhydrite are very unstable and rehydrate rapidly back to gypsum just because of atmospheric moisture and/or by capillarity rise of water from the floor and along the walls. Stable anhydrite tends to rehydrate back to gypsum as well, but the reaction is very slow. In a dry climate, such as in Sicily and Crete, anhydrite probably needs several millennia to be rehydrated back to gypsum. In more humid climate, such as in Northern Italy, anhydrite is still present in burnt gypsum blocks after centuries from the fire event (observations in the S. Silvestro abbey church, Nonantola; LUGLI, 1995).

From the above considerations it follows that severity and extent of a fire in an ancient building may be evaluated considering the thickness of the white surfaces and their anhydrite content. The coexistence of anhydrite with gypsum suggests surface temperature in excess of 252° C, whereas the sole presence of anhydrite indicates temperature in excess of 352° C.

After rehydration of the metastable phases, the shape of the former crystals is still recognizable, but the crystals have been replaced by a mosaic of white microcrystalline gypsum (pseudomorphs). For this reason the effect of fires are recognizable only on macrocrystalline gypsum facies, such as selenite and gypsarenite, which are made of translucent crystals and generally show a grey color (*fig. 1*). These facies are typical of the Messinian evaporites and are generally missing in the Permo-Triassic Formations, which are mostly composed of microcrystalline white («alabastrine») gypsum. There would be no possibility to recognize effect of fires on such rocks, because

the starting material does not differ from its dehydration-rehydration product.

Unaffected zones: the «shield shadows»

Most of the gypsum blocks in the studied sites are affected by white surfaces totally or in part. The unaffected areas are commonly in the lower part of the blocks and correlate across the walls (*figs. 3* and *4*). The presence of sharp unaffected surfaces in the white blocks could be interpreted as the shield effect to direct heat by fallen blocks, bricks, roofing-tiles, beams or other objects. In some examples in the Minoan Palace of Cnossos the shield against fire may have been the socle (*fig. 1*). This interpretation is supported by the observation that gypsum blocks are completely affected by white surfaces in those tracts where socle is missing.

Natural or artificial dehydration

The final result of gypsum dehydration-rehydration due to burning may be indistinguishable from its natural equivalent, which may occur because of several geological causes during diagenesis or burial evolution of the rock. In case of natural dehydration, however, the white areas differ from those formed by artificial dehydration because the former are massive or have nodular shapes and obviously formed before the rocks were quarried. The criteria to distinguish between artificial and natural dehydration are thus easy (*Tab. 1*). White surfaces caused by fires:

1) develop only on the exposed faces of the blocks (*figs. 1* and *2*),
2) are a few centimeters thick and thin away from burned zones (*figs. 1* and *2*),
3) may contain anhydrite in amounts which are decreasing away from burned zones,
4) are correlable across adjacent blocks made of different gypsum facies together with their «shiel shadows» (*figs. 1* and *2*).

Fig. 4 – Close-up of the wall structure of *fig. 3* (Palace of Minos, Knossos, Crete). Note that the white surface cut through blocks with different crystal orientations, indicating that dehydration occurred after emplacement of the blocks.

The geological study of gypsum facies in the outcrops, which were possible source of building materials, may reveal the presence of natural dehydration features, which could be misinterpreted as effect of ancient fires. In some cases, the dehydration zones may have been totaly quarried away, it follows that the possible occurrence of natural dehydration in the former source gypsum could be evaluated only by geological considerations.

Recycling of burnt blocks and «artistic» burning

In some cases it has been possible to observe old dehydration surfaces located on block faces oriented toward the wall interior, whereas the exposed face was unaffected. This case could be explained by recycling of old blocks from previous buildings. Because fire represented one of the most common causes of destruction for ancient buildings, it follows that recycled blocks could have been dehydrated in more than one occasion before definitive emplacement. This possibility must be kept in mind when surveying white surfaces in ancient buildings. Recycling of dehydrated old blocks appears unlikely in all those cases when «shield shadows» correlate along adjacent blocks.

Another aspect that should be taken into account is that gypsum rocks may have been intentionally burnt to produce candid blocks or slabs. These «special» architectural elements may have been used to provide a spectacular and prestigious decoration for rooms dedicated to particular porpouses, such as ritual or reception ceremonies. This could be the case in some of the Minoan sites in Crete, but the hypothesis should be fully investigated. Intentional burning for artistic purposes can be excluded where partially dehydrated blocks are common and «shield shadows» are correlable along the walls.

Conclusions

The study of white surfaces on gypsum blocks composed of selenite, gypsarenite and gypsrudite indicate that traces of ancient fires in archaeological sites can be recognized. The spatial extent of correlable white surfaces in the buildings, their variable thickness (millimeters to centimeters) and composition (gypsiferous versus anhydritic), may give indications on the severity of damages caused by accidental or intentional fire events providing significant information to unravel the evolution of archaelogical sites.

In order to avoid the risk of misinterpretation, the study of dehydration surfaces in archaeological sites should be corroborated by a geological survey of evaporite facies in the source areas, to indicate whether naturally dehydrated blocks could have been used in the buildings.

Acknowledgements

References

ABRIEL W. and REISDORF K., 1990, Dehydration reactions of gypsum: a neutron and X-ray diffraction study. Journal of Solid State Chemistry, 85, 23-30.

CHLOUVERAKI S., this issue, Exploitation and utilisation of gypsum in Minoan architecture.

LUGLI S., 1995, Blocchi di roccia gessosa nella chiesa abbaziale di S. Silvestro a Nonantola (Modena): caratteristiche geologico-petrografiche e ipotesi di provenienza. Atti della Società dei Naturalisti e Matematici di Modena, 124 (1993), 137-160.

LUGLI S., 1997, Il gesso in natura e nell'arte. Atti della Società dei Naturalisti e Matematici di Modena, 127 (1996), 31-47.

POPP T. and KERR H., 1993, Thermal dehydration reactions characterised by combined measuements of electrical conductivity and elastic wave velocities. Earth and Planetary Science Letters, 120, 43-57.

Table 1

	Original gypsum rock (selenite, gypsarenite, gypsrudite)	Artificial dehydration (immediately after burning)	Artificial dehydration (final product)	Natural dehydration (final product)
Color	gray	white	white	white
Composition	gypsum	± gypsum ± bassanite ± soluble anhydrite ± anhydrite	gypsum + anhydrite (T>252° C) anhydrite (T>352° C)	gypsum ± anhydrite
Crystal size	millimeters to decimeters (monocrystals)	micrometers (pseudomophic mosaic)	micrometers (pseudomophic mosaic)	micrometers to millimeters (pseudomophic mosaic)
Type of white surfaces	–	planar	planar	planar, curved, nodular
Thickness of white surface	–	millimeters to centimeters	millimeters to centimeters	centimeters to meters
Age of whitening	–	after block emplacement	after block emplacement	before block emplacement

Characteristics of gypsum rocks and their artificial and natural dehydration products.

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