

Mural Paintings in the Burial Chamber of Mastaba Idout: Cause of Deteriorations, Material Analysis, and Conservation Works

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イドウトのマスタバ地下埋葬室の壁画
——劣化要因、材料分析および保存修復——

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1 Causes of deterioration of the Wall Paintings¹

1.1 Introduction

The mural painting in the burial chamber in Saqqara Tombs symbolizes the most valuable and remarkable component of these archaeological sites. The techniques applied in the different period to create these pigments indicate the great skill, ability and experience achieved by the ancient Egyptian artists. The utilized technique for painting during the old Kingdom was the tempera technique and an organic binding material mostly of plant and animal origin was used [Lucas 1962; Nakhla 2003]. The preparation layer consists mainly of gypsum plaster and white wash. During the Roman times the Fresco technique was introduced in Egypt. In the Fresco technique, pigments are carried out when calcium hydroxide is still fresh and moist and they are bound to the surface during the carbonation process.

The protection and conservation of archaeological areas presents major tasks for the future and is a major duty for our existing generation. Generally, a lot of archaeological sites in the Saqqara

area, have recently suffered from severe degradation, compared to the conditions in which they were originally discovered and excavated, due to the lack of durable conservation practice, exposure to semi-arid climatic conditions, rising pollution, and ever-increasing pressure from tourism. The temples, pyramids and tombs of this region are composed of rocks of different types, most commonly limestones. Most of these remains show various signs of degradation [Nakhla 2003]. However, the degree of decaying depends mainly on the interaction of the factors related to the surrounding environmental and to those related to the rock texture and composition. Saqqara is the oldest Ancient Egyptian Cemetery, and lies south-west of Cairo (Fig. 1). It is considered to be one of the richest and most highly varied archaeological sites in Egypt. The Saqqara region is a plateau, formed mainly of well-stratified, yellowish, argillaceous limestone, marl and calcareous claystones, with a relatively high content of gypsum veinlets of late Eocene age [Youssef, *et al.* 1984]. In this area, many rock tombs, mostly dated to the Old Kingdom, have been hewn within these Upper Eocene sediments (Maadi Formation). Among these tombs is the Idout Tomb.

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Fig. 3 Shows big pile of the fallen fragments having various sizes.

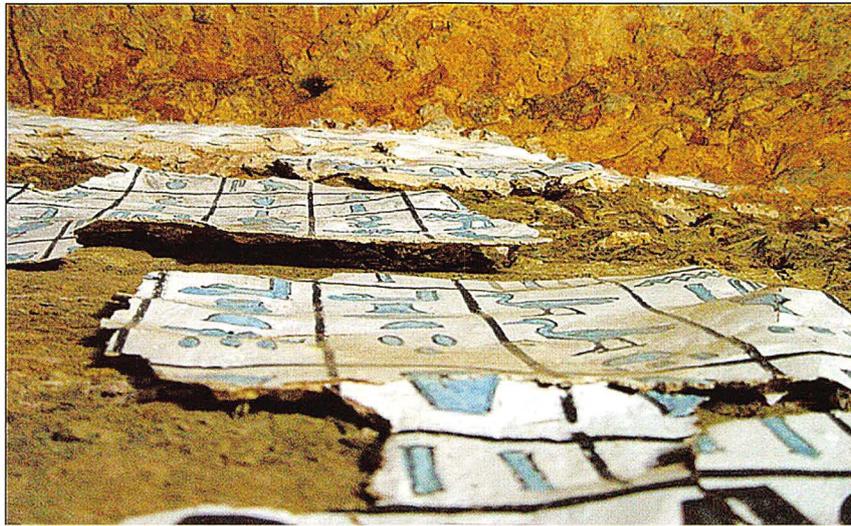


Fig. 4 Painting fragments highly separated from their wall support and being fallen down.

danger of being lost as a result of the separation of the painted plaster from the clayey limestone support (Fig. 3 and Fig. 4) was also apparent. There were signs of discoloration, color fading and salt crystallization, as well as mechanical deterioration caused by people. It seemed that a main reason of the deterioration of the wall paintings was the bed-rock composition, plaster, and paint layers.

Due to the extremely bad condition, the mission began to start a project for the preservation of the wall paintings at Saqqara area and picked up Idout Tomb as a case study. Hence, a three-phase program was formed to cover: (1) scientific study and analysis to evaluate the causes of deteriorations, (2) emergency stabilization of the wall paintings at the burial chamber, and (3) conservation management. It was decided that the first two years of the project would be fully dedicated to scientific studies of the causes of deterioration & emergency treatment.

The important question, however, was whether

the deterioration process was continuing and, if so, what the main mechanisms of deterioration were. It was therefore obvious to study the tomb from a geological, petrographical, mineralogical, and chemical point of view before deciding on and undertaking conservation treatment. This was important because the most important stage of any stone conservation process is the characterization of the stones' composition and the investigation into the causes of deterioration. If the chemical, mineralogical and petrographical properties of the rocks are not taken into consideration, the choice of an intervention technique may be unsuitable, to the extent that it damages the appearance or integrity of the stone. If the causes of the decay are not evaluated and corrective measures taken to stabilize the deterioration, successful intervention may, in fact, be only a temporary palliative [Espert *et al.* 1981; Preusser 1987; Price 1996]. It was therefore clear to interpret the causes of deterioration of the wall paintings through the examinations into the petro-

graphic characteristics and mineralogical composition of the wall painting support (mother rock or bedrock) and the preparatory plaster layer, in order to characterize the used rocks. It is hoped this strategy will enhance the stones' conservation and the development of suitable conservation/ restoration techniques. To achieve this, a polarizing microscope, X-rays diffraction (XRD) and thermal differential (DTA) are used; along with acid insoluble residues (AIR) being examined using the binocular microscope [for detail see Akarish 2007; Akarish and Shoeib 2011].

1.3 Petrography of the Bedrock

The bedrock comprises of two different lithotypes, limestone/argillaceous limestone and marlstone. The AIR of the argillaceous limestones ranges from 14-19%, whereas in the marlstones it is 43-53%. Binocular microscope examination of the AIR revealed that clay with subordinate sand plus a small amount of gypsum are the dominant components. The AIR of the argillaceous limestone is composed of 86-91% clay, 5-9% sand and 3-5% gypsum, whilst that of the marlstones is 78-84% clay, 9-13% sand and 6-9% gypsum.

Petrographically, the limestones are classified into biopelsparite [Folk 1959] or bioclastic peloidal grainstone [Dunham 1962]. The peloids and bioclastics are present in varying proportions (Fig. 5A and Fig. 5B). The bioclastic constituent is 15-25% of the rock volume and is formed of mollusc fragments echinoid debris, benthic and planktonic foraminifera, together with algae. Some of the fossil

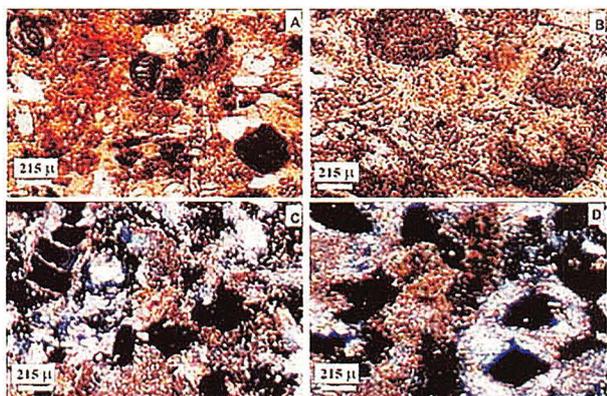


Fig. 5 A-Biopelsparite, limestone lithotope, P.P.L. B- Rounded peloids embedded in sparite matrix, P.P.L. C&D- Pore spaces (both inter- and intraskeletal) having various size and shape, biopelsparite association, C.N.

chambers are recrystallised and/or infilled with sparite, but have mostly been leached out. Peloids (20-35%), composed of structureless micrite, range in size from 20-110 μ m, and are rounded, ovoid, well-sorted and organically rich (Fig. 5B). They are formed due to fecal pelleting by certain organisms, such as molluscs and worms. The allochems are embedded in a sparitic matrix that is generally sub-translucent with a faint brownish cast in thin section. Pore spaces (both inter- and intraskeletal) are abundant (average 13%), having various size and shape (Figs.5C, D). They may have developed as a result of post-depositional diagenetic (dissolution) processes. The non-carbonates are represented by fine nodules of clay and fine detrital quartz grains (~8%).

The marlstones are very heterogeneous, but with different grain-sizes that range from 10 to 250 μ m. They have a higher percentage of terrigenous material represented by clays that admixed with the micritic matrix. The latter is composed of very fine microcrystalline carbonate that is commonly recrystallized into microsparite. These rocks contain <10% fossil fragments, randomly scattered throughout the recrystallized micrite matrix, and so can be tentatively classified as fossiliferous micrite [Folk 1959] (Fig. 6A) or lime-mudstone [Dunham 1962]. Some of these fragments are leached or replaced by recrystallized sparry calcite. They contain many micro-cracks, some of which have been filled with sparite.

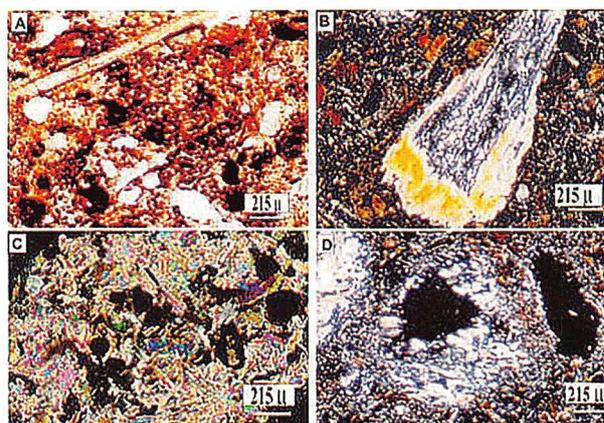


Fig. 6 A- Clayey fossiliferous micrite, micrite matrix commonly recrystallized into microsparite, marlstone lithotope, P.P.L. B - Coarse gypsum crystals (partly dehydrated) in fine grained gypsum groundmass, plaster preparatory layer, C.N. C - Well developed anhydrite crystals characterized by higher relief and stronger birefringence, C.N. D - Well developed pores in plaster layer, C.N.

1.4 Petrography of the Plaster Layer

A plaster preparatory layer was applied directly onto the bedrock with varying thickness. Plaster over the limestone is fine enough to allow relief carving or other manipulation. The rock surface was smoothed and covered by a reinforcing rough mortar layer, formed of coarse gypsum, in which thin limestone flakes powder, together with fine sand, was added as filler material. Over this coarse layer, a fine layer composed mainly of gypsum was applied. Therefore, the plaster layer was used to treat irregularities and smooth the surface of the bedrock [Lee and Quirke 2003]. Microscopically, the plaster layer is texturally heterogeneous. The groundmass consists mainly of finely grained gypsum in which coarse gypsum crystals are widely distributed (Fig. 6B). These crystals are occasionally dehydrated into anhydrite; characterized by higher relief and stronger birefringence (Fig. 6C). Voids (Fig. 6D) of various size and shape with sharp outlines, are occasionally delineated by fine gypsum grains, are additionally present. Some fine-to medium-sized quartz grains were also observed. Patches of iron oxide (hematite) occur as staining, with a red to blood-red color (Fig. 6B).

1.5 Mineralogy of the Bedrock

X-ray analysis of the bedrock indicated that calcite and little quartz are the main non-clay minerals recorded in all samples (Fig. 7A and Fig. 7B), whereas halite and/or gypsum are revealed in some marlstone samples. The clay mineral assemblages identified within the clay fractions (Fig. 8, as an example) include montmorillonite, kaolinite and

illite, in order of decreasing abundance. Both types of montmorillonite (Ca-,montmorillonite and Na-montmorillonite) were encountered in the studied Upper Eocene sediments. The semi-quantitative estimation of the recorded clays implies that montmorillonite (the expandable clay mineral) is the predominant clay, ranging from 61%- 70% with an average 66%. Kaolinite is the second in abundance, ranging from 23%-31% and averaging 26%, whereas illite averages 7%.

1.6 Mineralogy of the Plaster Layer

X-ray diffraction of the plaster (Fig. 9A and Fig. 9B) indicates that gypsum with subordinate anhydrite, in addition to bassanite and a little quartz are the main components. The differential thermal analysis (DTA) of the plaster is shown in Fig. 10. The DTA curves show the presence of two large endothermic peaks and one small endothermic peak. The first large endothermic peak occurs in the range of 145°C - 155°C, implying the formation of a hemihydrate phase [Flek *et al.* 1960]. The weight loss corresponding to this peak, as revealed from the TGA curve (Fig. 10), is about 9-12%; this represents about 75% of the total weight loss (~16%) of the sample; i.e. about 1.5 moles of the combined H₂O. The second large endothermic effect occurs in the vicinity of 185°C - 188°C, indicating a dehydration of hemihydrate to soluble anhydrite and loss of the remaining 0.5 mole of H₂O. Such total loss (~16%) implies that the present plaster layer is not formed of pure gypsum (theoretical total weight loss ~21%) but contains an amount of anhydrite and/ or bassanite; corroborating the re-

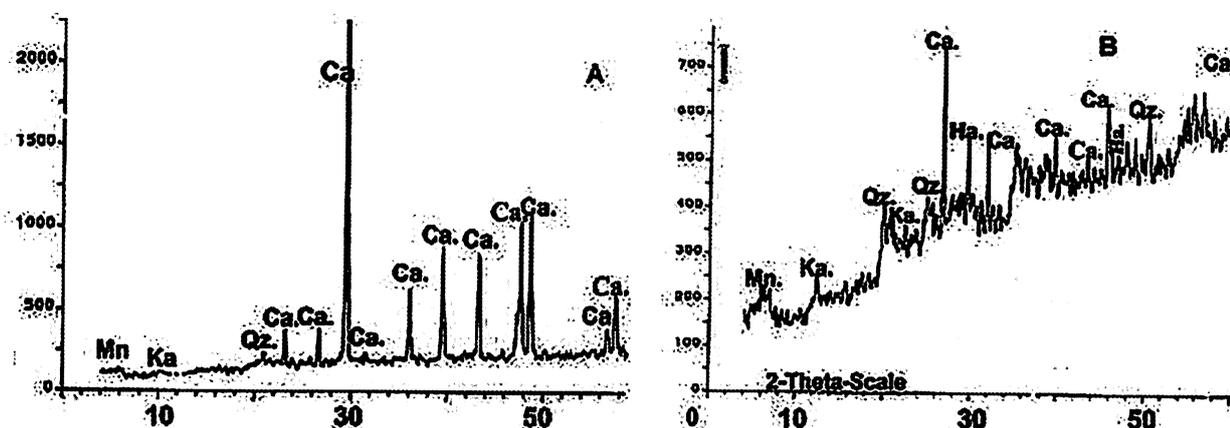


Fig. 7 X-ray diffraction patterns of some bulk samples of the bedrock, A- argillaceous limestone, B - marlstone. Qz =Quartz, Ca= calcite, Mn=montmorillonite, Ka= Kaolinite Ha= halite.

sults obtained from the petrographic studies, as well as the X-ray analysis. The very small endothermic effect may be attributed to the loss of the last traces of water held in the hemihydrate structure [Kuntze 1962].

1.7 Discussion

The process of decay of the wall paintings likely had already started in ancient times. It gradually progressed, over thousands of years, until the time of the archaeological discovery in the year 1927. Ancient burial chambers, because of their isolation from external factors, ensured a somewhat stable environment for the paintings. After the

tomb was opened, this environment had been totally distorted. Susceptible materials started to react to new temperatures and humidity conditions. The petrography and mineralogy of the bedrock contribute to the deterioration of the paintings and in turn, caused the mortar and the painted layer to detach from the bedrock. These rocks are too weak to resist the interaction between exogenic (climate, including rain water humidity, and temperature variation) and endogenic (related to the nature of the rock types) conditions, since their construction, thousands of years ago. These agents of deterioration often act in conjunction and also potentiate one another [Mora *et al.* 1984]. Our re-

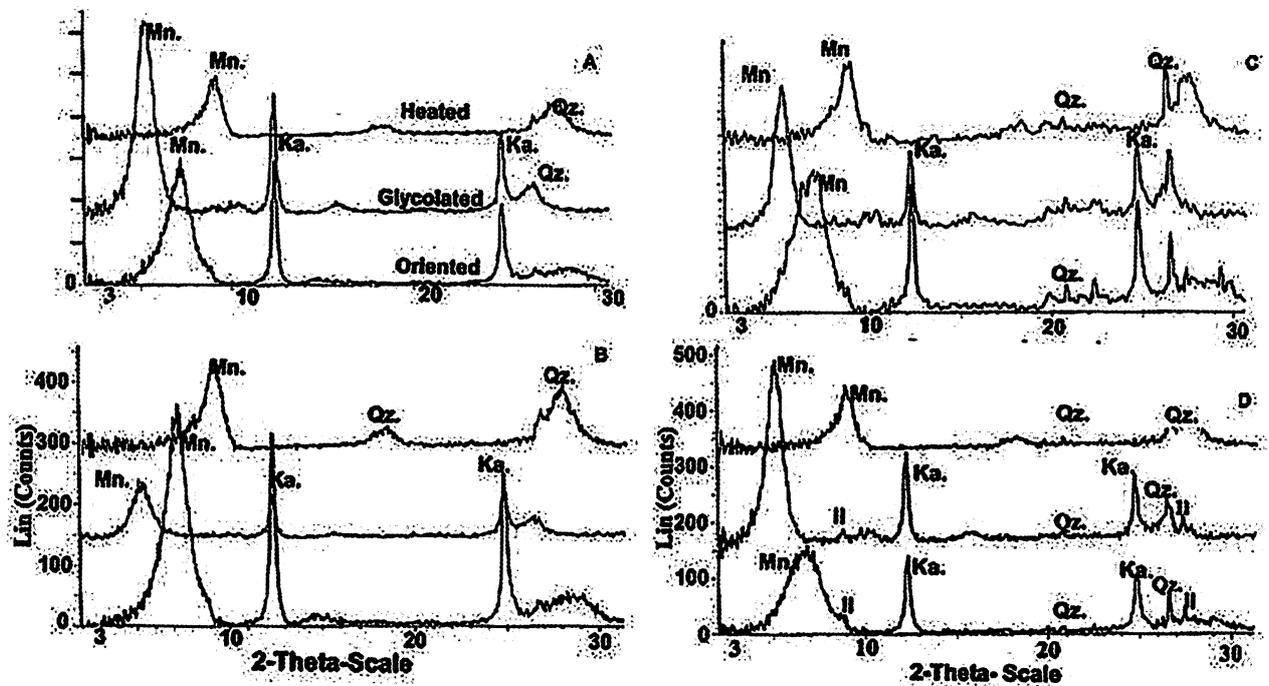


Fig. 8 X-ray diffraction patterns of some clay fractions of the bedrock. (A,B), argillaceous limestone, (C,D), marlstone; Mn=montmorillonite, Ka=Kaolinite, Il=illite Qz=Quartz.

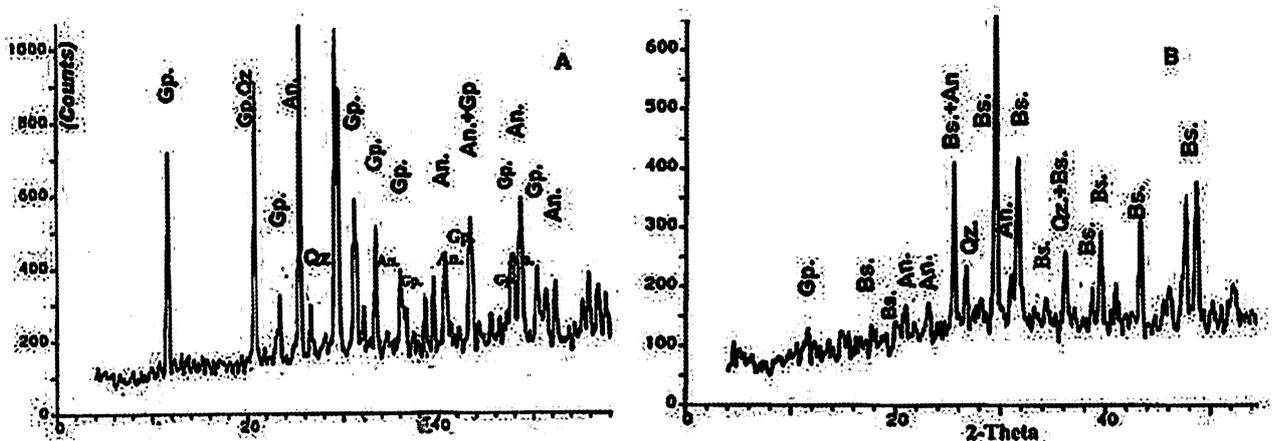


Fig. 9 Show X-ray diffraction patterns of the studied plaster, Gp.= gypsum, An.= Anhydrite, Bs.=Bassanite Qz=Quartz.

sults indicate that the limestone bedrock is composed of grain stones with a relatively low mud content, compared to the marlstones. Grainstones (sparites) are sufficiently permeable and hold porous facies, facilitating fluids to interact within the rock and influence their components [Sperber *et al.* 1984; Dawans and Swart 1988]. Moreover, the studied bedrock had originally suffered from diagenetic dissolution processes. It is believed that dissolution was mainly achieved by the action of circulating meteoric water in an active zone [Longman 1980]. Dissolution resulted in the development of inter- and intraskeletal pores through the removal of peloids, shell fragments and/or leaching of their cores (Fig. 4C and Fig. 4D). This process also produced irregular voids and vugs (Fig. 4D), with various sizes and shapes, or caverns (observed in the field). Such features might have led to a drastic increase in the bedrock porosity [Abu-Zeid 1989; Tucker *et al.* 2001]. Such sediments with high porosity and high pore connectivity favor the diffusion of rain water and humidity and help in water sorption, increasing the capillary pressure inside the rock, and finally giving rise to its disintegration, as indicated by Espert [Espert *et al.* 1981]. It is also evident that the higher the rock porosity the higher the

tendency to disaggregate [Espert *et al.* 1981].

It is worth mentioning that, in the Saqqara area, the relative humidity ranges from 26- 88% in the summer and from about 34-90% in the winter. On the other hand, the determined moisture content of the argillaceous limestone samples ranges from 0.9 to 1.6% and in the marlstones from 1.7 to 2.1% there statistics are in accordance with the results given by Soliman [Soliman 1998] and indicate a relatively high percentage.

The marlstones and argillaceous limestones of the bedrock contain a considerable quantity of clay, with a high percentage of expanding clay minerals; montmorillonite (average 66%) being the main clay mineral.

Montmorillonite is one of the clay minerals, belonging to the smectite group, all of which possess the property of being able to expand and contract their structures while keeping crystallographic integrity [Moore and Reynolds 1997]. This property is the main control on the physical characteristics of natural materials in which smectites are present. The swelling capacity of clays is a desirable feature [Bell and Maud 1995; Wilson *et al.* 2006]. In the presence of water swelling clay is one of the most significant factors of deterioration [Houben and

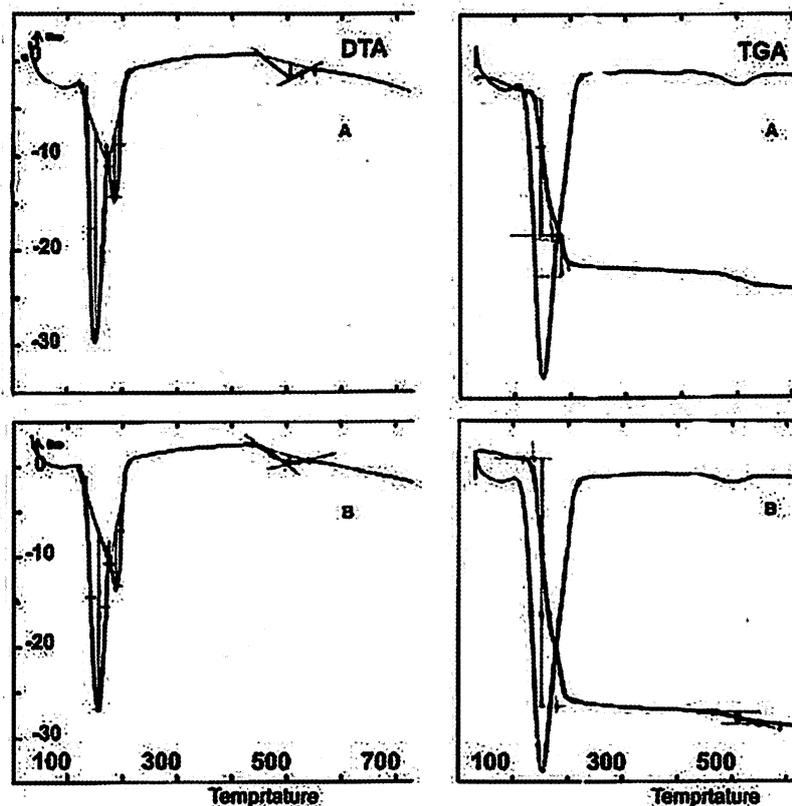


Fig. 10 DTA and TGA curves of the studied plaster.

Guillaud 1994; Nelson and Miller 1997; Meisina 2004]. Montmorillonite contains differing amounts of water due to a negative charge and the concomitant presence of exchangeable cations. These cations tend to form hydrates through attracting water molecules whenever some water is present. Ca and Mg ions retain H₂O more strongly [VanRanst 1993]. According to VanRanst (op. cit.), at every temperature, equilibrium exists between the amount of water found in the interlayer space and atmospheric water (relative humidity). As the relative humidity increases, the mineral will absorb more water out of the air until a new equilibrium is reached. Two types of clay swelling can be distinguished: a) osmotic or interparticle swelling, which takes place in any clay when saturated with water, and b) intracrystalline swelling, which only occurs in expandable clays (e.g., smectites); it may finally lead to structural failure [Rodriguez-Navarro *et al.* 1998] which is the case in the present study. The smectites can swell to a volume that can be several times the volume of the dry clay, depending on the amount of water available. In dry conditions, most of the adsorbed water is lost through evaporation, causing strong shrinking. It can therefore be presumed that periodic swelling and shrinkage during alternating wet and dry seasons, favored by humidity and temperature fluctuations, may have led to disintegration or severe damage to the bedrock. In turn this critically effects the stability of the wall paintings, especially with an increase in water content. Moreover, during the swelling process, the cohesion of the clay particles may have decreased, resulting in very low shear strength of the rock; the clay particles slide easily over each other and cause a lot of damage. Consequently, the bedrock has been damaged and in turn the plaster layer has collapsed and fallen down. It is well known that evaporites (gypsum, halite and other water-soluble salts) are originally present in the Upper Eocene limestone used for monument construction. They are mainly primary or secondary, due to the drainage [Soliman 1998]. The studied rocks have a considerable amount of gypsum and halite, as indicated from X-ray. Presence of soluble salts (especially halite) in our samples represent one of the most important causes of stone

decay and generate a serious damaging effect, as reported by many authors [e. g. Lewin 1982; Bongrani and Fanfoni 1995; Goudie and Viles 1997]. The salt-bearing solutions migrate through the rock, and under suitable conditions water evaporates and salts are deposited. The growth of salt crystals within the pores of a rock can generate stresses that are sufficient to overcome the rock's tensile strength and cause damage and disruptions, sometimes turning the stone to a powder [Jewaka 1981; Zehnder and Arnold 1989; Steiger 2005]. Moreover, crypto-efflorescence of these salts between the bedrock and plaster may have caused the detachment of the plaster and painting layers. The plaster contains anhydrite and basanite minerals. These minerals may be developed due to dehydration of gypsum. The dehydration process leads to net reduction in the bulk density and the resulting anhydrite is going to be more porous than its compact original gypsum [Goldman 1952]. Furthermore, rupture deformation may originate due to volume decrease during transformation. Such process as may also contribute to the detachment of the plaster layer. However, the main cause of the destruction of the paintings appears to be due to changes in the volume of the bedrock, due to the changes in humidity.

1.8 Conclusions

Petrography and mineralogy of the bedrock and the plaster donate to the deterioration of the paintings and, in turn, caused the mortar and the painted layer to detach from the bedrock. Their composition are too weak to resist the interaction between exogenic (climate, including rain water, humidity, and temperature variation) and endogenic (related to the nature of the rock types) factors of alteration that often work together and also potentiate one another, causing a lot of decay. The durability of the mother rock is mainly governed by clay content and porosity. We recommend that traditional methods of conservation cannot be applied to the studied tomb because of the critical conditions of the bedrock and the plaster that have suffered from severe conditions of deterioration that affected the wall paintings. Therefore, the best way of saving the wall paintings in the Idout tomb,

and in other tombs in the Saqqara area suffering from similar conditions would be to detach them from the harmful bedrock, using the Stacco technique. Then, place them on to new supports formed of synthetic materials and mount them back in their original locations. This is the only way to isolate the paintings from the influence of their original destructive support. This is the work that was applied by the Egyptian- Japanese mission through development appropriate conservation / restoration techniques suitable for the preservation of Idout mural paintings.

2 Pigments and Plasters

2.1 Introduction

This work presents the results of an investigation and characterizations of the inorganic paintings used in the burial chamber of Idout's Mastaba.

The Mastaba of Idout dates back to 2360 B.C and it is one of the well-known and beautiful in Saqqara area and also in Egypt. It was discovered in 1927 by Firth and later published in 1935 by Macramallah (for details see chapter causes of deterioration, this book). When it was excavated, the mastaba became known well due to it preserving the beautiful relief in its original brilliance. The burial chamber was hewn in calcareous rocks (mainly limestone and marl), several meters under the mastaba and connecting to the mastaba through a vertical shaft. The dimensions of chamber is approximately 10.5 m in north south, 4.5 m in east west, and 3 m in height. The limestone wall

(supported material) is fine enough to allow relief carving or other manipulation. The rock surface was smoothed and covered by a reinforcing rough mortar layer, formed of coarse gypsum in which thin limestone flakes or powder together with fine sand was added as filler materials. The mortar layer has various thickness from less than 1 mm to up to 10 cm. Over this coarse layer, a fine layer composed mainly of gypsum was applied (0.035-0.050 mm). Therefore, the plaster layer was used to treat irregularities and to smooth the surface of the bed rock [Aston *et al.* 2003]. On the upper half of walls, paintings of a variety of foods and offerings were depicted on fine plaster to secure the happy life of the deceased. Only on the west walls of the niche was the painting unfinished. The walls of the tomb and its burial chambers were decorated with brilliant colors, which kept their original hue in their brilliance till now (Fig. 11). The colors are mainly blue, red, yellow, brown and white in addition to black and faint green (Fig. 12). On the walls (South wall), the colors appear to be as fresh as when they were applied. The pigments adhered to the plaster gypsum substrate surface by use of a binding medium of animal origin without remoistening of plaster surface, i.e. Tempera technique.

Pigments vary with deference to their chemical properties, because they are composed of a broad variety of chemical compounds. The shade (hue) and clarity of the colors are mainly related to color absorption [Gettens and Stout 1966]. The size, shape, and texture of the pigment particles are also due to the presence or absence of impurities.

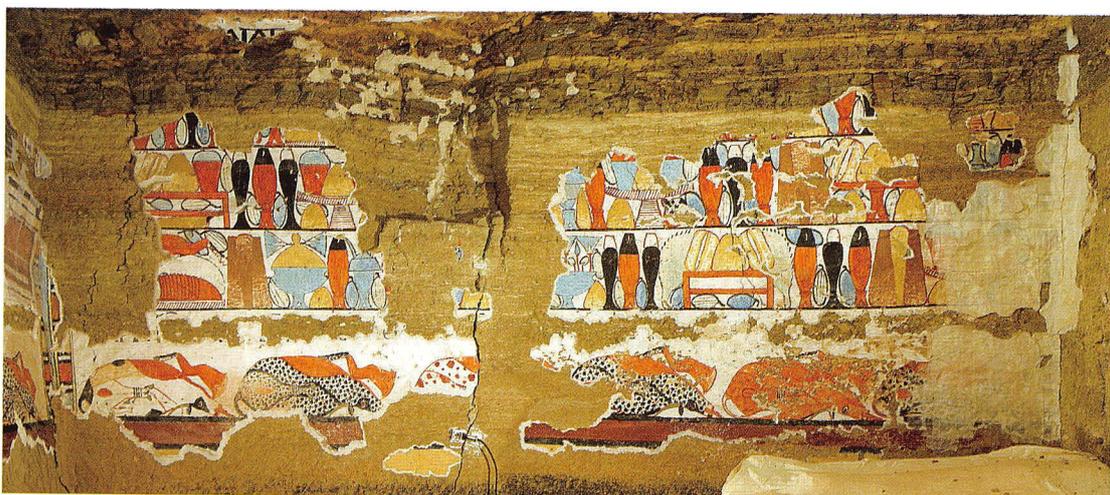


Fig. 11 The walls of the burial chambers decorated with brilliant colors.

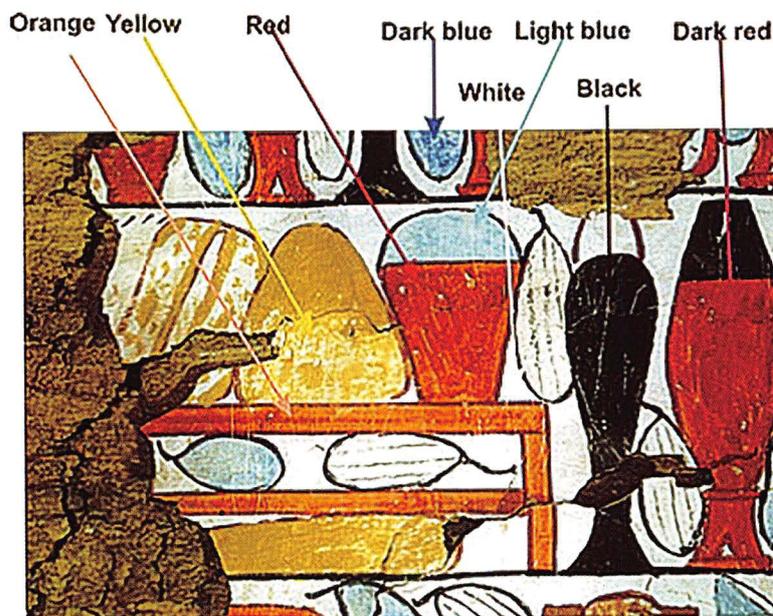


Fig. 12 Inorganic pigments used in Old Kingdom decoration of Idut's burial chamber.

We categorize some of the inorganic painting materials used for the creation of painted plaster (rock substrate, plaster layers and pigments) belonging to the Old Kingdom at Saqqara. The technical features of the painting components were investigated using optical microscopy (OM) and Scanning Electron Microscopy Equipped with an Energy Dispersive X-ray Spectrometer (SEM-EDS), XRD and VHX-100 Series Digital Microscope. The components of the substrate and preparatory layer of the painted areas were characterized using confidential chemical analyses [Akarish *et al.* 2011].

2.2 Rock substrate

The rock substrate (bed rock) was formed mainly of limestone (argillaceous in some parts were) and marlstone. Its chemical analysis (Table 1) implied that they were formed mainly of calcite, judging from the high content of CaO with little quartz and clays, as can be indicated by the acid insoluble (AIR) content. This was confirmed by the mineralogical and petrographic studies [details of these are given by Akarish and Shoeib 2011 and in chapter of causes of deterioration].

2.3 Plaster Layer

Using Digital Microscopic investigations, we distinguished two main layers of the plaster (Fig. 13) used to overcome faults in poor stone and to produce flat and smoothed surface for painting.

The base coarse layer mainly consisted of gypsum, calcite and quartz (XRD analyses), while the top fine coat layer was mainly based on gypsum with or without very fine sand. Chemical analyses of the coarser layer (Table 2) revealed a high content of acid insoluble materials, AIR, (37% in average), reflecting the high amount of sand. It contained relatively lower amount of combined water (average 4.5%). The average content of CaO and SO_3^- were 26.5% and 9.5%, respectively. Moreover, Na_2O , K_2O and Cl- concentrations were low. However, Na_2O and Cl- showed relatively higher amounts in some samples, implying the presence of salts, a feature that was not indicated through X-ray analysis. The sulphate (SO_3^-) content was too small to match with the content of carbonate, reflecting that much of CaO was present as carbonate (calcite), confirmed by less content of combined water. This suggested that thin limestone flakes or powder, together with fine sand, were added as filler materials during the preparation of the mortar. Presence of calcite was confirmed by XRD analysis. Absence of carbonate in the finer layer and plaster samples corroborated such interpretation. On the other hand, the finer layer was characterized by (1) a low content of AIR, (2) a high content of combined water, indicating a higher percentage of gypsum, and low contents of Na_2O , K_2O , Cl (Table 2). The CaO concentration matches very well with sulphate SO_3^- content and also corroborates with the amount of

Table 1 Partial Chemical analysis of the wall support substrate.

Component (%)	Wall support samples	
	Limestone	Marlstone
AIR*	18.58	52.38
CaO	44.39	20.08
MgO	Nil	Nil
Na ₂ O	0.48	1.69
K ₂ O	0.06	0.10
Cl ⁻	0.62	1.77
SO ₃ ²⁻	0.24	1.08
LOI **	35.87	24.40

Table 2 Chemical analysis of preparatory plaster layer.

Component (%)	Preparatory plaster layer			
	Rough coat		Finer coat	
	A	B	A	B
Insoluble Residue (AIR)	37.32	37.33	4.93	4.86
CaO	26.43	26.67	32.96	35.55
MgO	---	---	---	---
Na ₂ O	1.13	0.48	0.07	0.06
K ₂ O	0.16	0.10	0.06	0.05
Cl ⁻	0.54	0.48	0.20	0.27
SO ₃ ²⁻	9.17	9.99	45.40	42.51
Water loss at 230°C	5.38	3.72	14.58	12.37

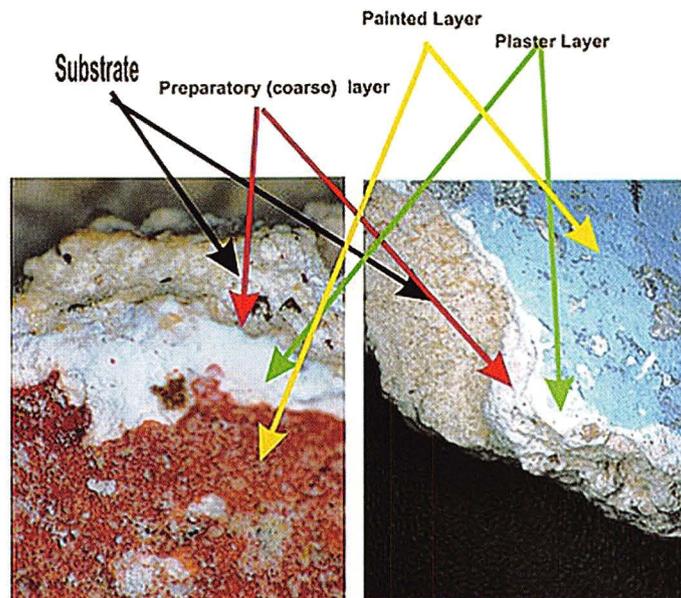


Fig. 13 Microscopic observation of the two plaster layer and the painted layer produced on the limestone substrate.

water content, which reflect that CaO was present only in a form of calcium sulphate minerals; it is evident that the higher the water contents, the higher the percentage of gypsum was. This is in accordance with the results obtained from the mineralogical analyses.

2.4 Paint Layer

We started by characterizing the inorganic components of the illustrative layers. The color interpretation can be done by understanding the character of the pigments, their features such as morphology and thickness and the sequence of the colored layers.

(1) Blue color

The Egyptians developed a wide range of pig-

ment variety including what is now known as “Egyptian Blue,” which was the first of its color at the time of its development. The first recorded use of Egyptian Blue as a color name was in English 1809 [Maerz and Paul 1930]. The earliest evidence for the use of Egyptian Blue is in the 4th Dynasty (ca 2575-2467 B.C.). It continued to be used as a pigment for the decoration of tombs, wall paintings, furnishings and statues. It was used continuously throughout the Late Period and Greco-Roman Period, only dying out in the 4th century A.D., when the secret of its manufacture was lost [Chase 1971]. Egyptian Blue is a synthetic blue pigment; its color is due to a calcium-copper tetrasilicate $\text{CaCuSi}_4\text{O}_{10}$ of the exact same composition as natural mineral, cuprorivaite. It was produced by mixing silica, lime, copper compound and alkali flux (natron or

plantash) to reduce the required temperature [Hatton *et al.* 2008]. This mixture was heated to a temperature between 850 and 1000 C (basing on the amount of alkali used) to produce a colored glass or frit and later ground to powder for use. The result is cuprorivaite or Egyptian Blue, carbon dioxide and water vapor: $\text{Cu}_2\text{CO}_3(\text{OH})_2 + 8\text{SiO}_2 + 2\text{CaCO}_3 \rightarrow 2\text{CaCuSi}_4\text{O}_{10} + 3\text{CO}_2 + \text{H}_2\text{O}$.

Two types of blue pigments used in Idout burial chamber were distinguished, dark blue and light blue (Fig. 14). Both microscopic and SEM observation showed that the dark blue color consisted of very coarse to coarse rectangular layers of coarse-medium sized blue crystals (Fig. 15), cluster together formed of cuprorivaite. It was thickly ap-

plied with coarse particles up to 0.04 mm across (Fig. 16), giving the dark blue hue appearance. In contrast, the light blue color has fine tabular crystals about 15 μm to 30 μm that are uniformly distributed with some unreacted quartz (Fig. 17). The light blue color containing white color may be of gypsum. But, it is not certain if it is an additive to the blue or appears as contamination from plaster layer.

The shade of blue, as revealed in our study, was related to the coarseness and fineness of Egyptian Blue as it was determined by the degree of aggregation of its particles. Coarse Egyptian Blue, has a thick form, due to the large clusters of crystals, which adhere to the unreacted quartz.

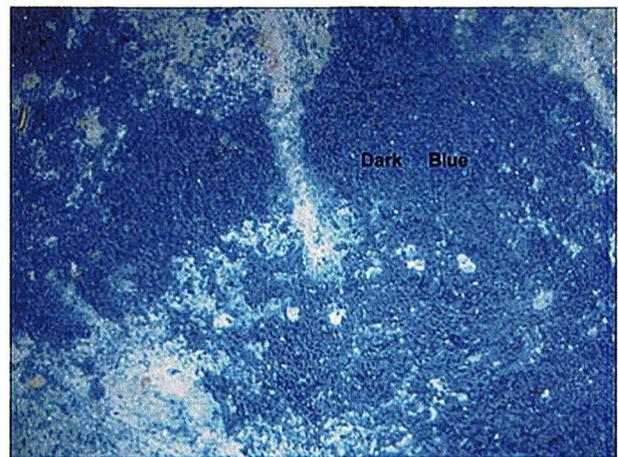
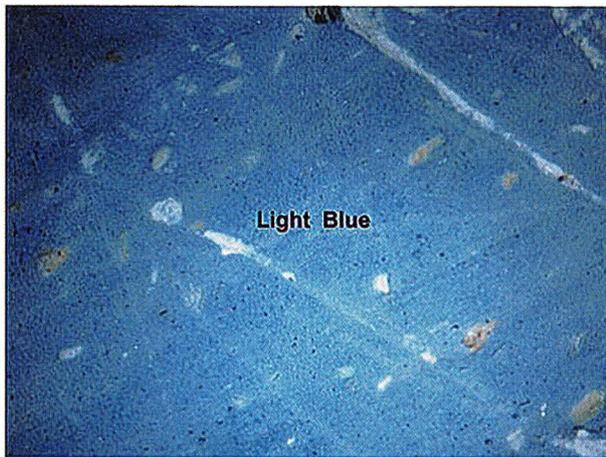


Fig. 14 Microscopic view for light blue formed of fine particles (left) and dark blue formed of coarse particles of caprorivaite(Egyptian Blue pigment).

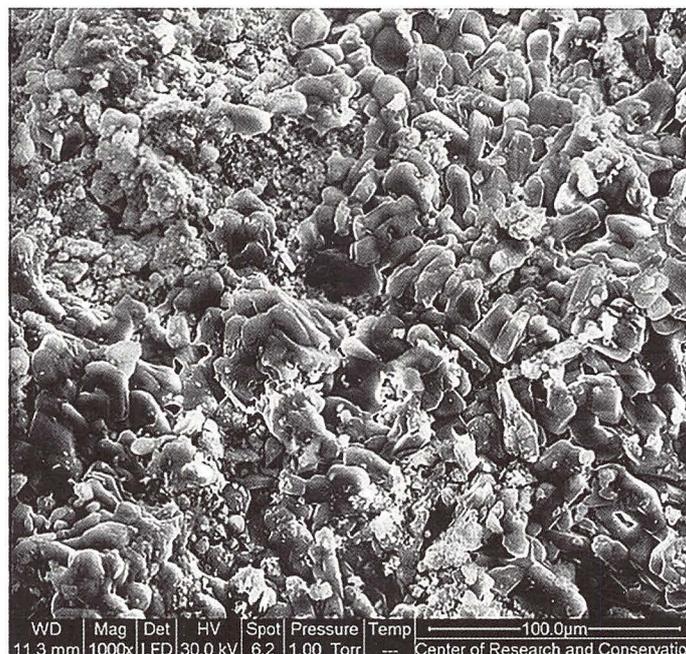


Fig. 15 SEM photomicrograph showing coarse tabular crystal of the dark blue color.

This clustering resulted in a dark blue color that was the appearance of coarse Egyptian Blue. Alternatively, fine-textured Egyptian Blue consisted of smaller clusters that were uniformly interspersed between the unreacted quartz grains and tended to be light blue in color [Tite *et al.* 1987]. The hue of the colors also depended on the level of alkali added to the mixture and, therefore the more alkali, the more glass-formed, the more the diluted ap-

pearance was gained. The major components of the studied blue pigments are cuprorivaite and quartz (unreacted), as indicated by mineralogical analyses (XRD), and is confirmed by micro-chemical analysis (Fig. 18). The later revealed that the main constituents are SiO₂ (62.12-63.83%), CaO (12.63-13.54%) and CuO (13.48-15.83%), which fall in the ranges given by Tite *et al.* 1987 for Egyptian Blue in Egyptian antiquities and elsewhere; their data indicated

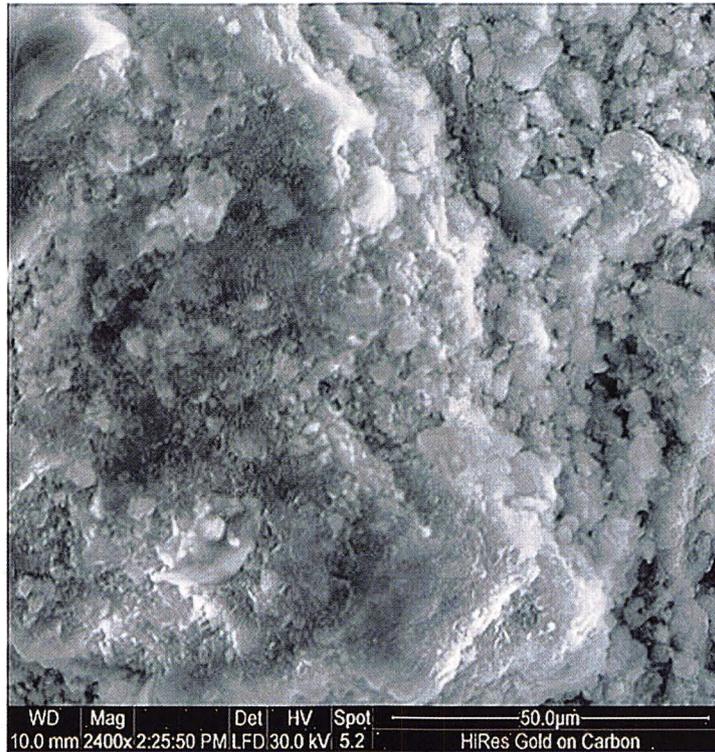


Fig. 16 SEM microphotograph showing thickly applied layers of coarse dark blue crystals.

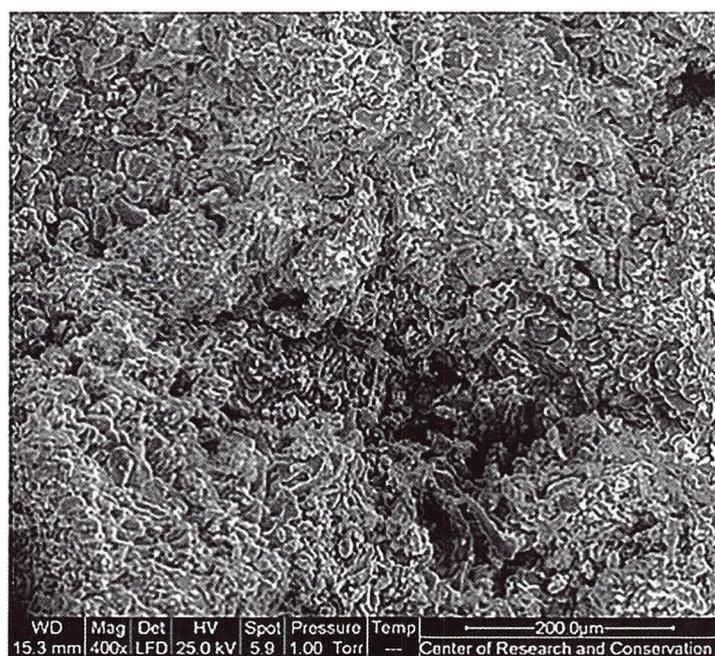


Fig. 17 SEM photomicrograph showing fine crystals of light blue color.

ranges of SiO₂, CaO and CuO are 60-70%, 7-15% and 10-20%, respectively. SEM-EDS semi-quantitative analyses are shown in Table 3. The total alkali content in the analyzed samples was greater than 1%, suggesting that the alkali was introduced into the mixture and not as an impurity from other components. The sources of alkali may be natron as evident from the absence of phosphorous and low content of magnesia. It may be from Wadi el Natron. The presence of some metals (e.g. titanium) in the studied samples could be a result of the presence of Ti-bearing minerals found in desert sand. Its presence in Egyptian Blue indicates that quartz sand in the area of manufacturing was used as silica source.

(2) Red color

Red pigments show the massive granular aggregate particles appearing to be uniform (Fig.19). The results obtained from the XRD analysis indicate that the red color was formed mainly of hematite (red iron oxide, Fe₂O₃). The EDX analysis (Fig. 10) confirmed the presence of iron oxide by the presence of the iron peak (~37%). Presence of aluminium and silicon detected by EDS analysis suggests the existence of aluminosilicate materials (clay minerals etc.), together with the presence of traces of minerals (Ti, V, Co) (Fig. 20), judging that the red ochre was used as red pigments in Idout tomb. Red ochre is a natural mineral pigment colored by the presence of the anhydrous, iron oxide, hematite. Ochre forms a very wide class of natural inorganic pigments. Ochre is characterized by having a wide

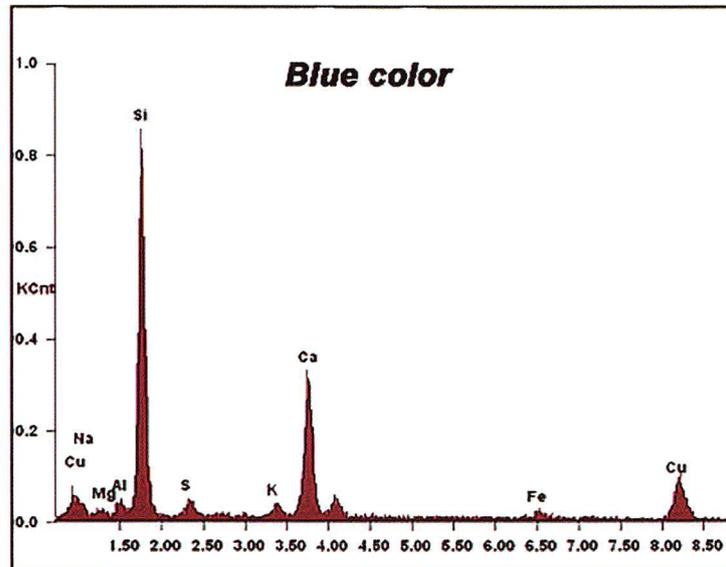


Fig. 18 Micro chemical analyses of blue pigments.

Table 3 EDX analysis of the blue color compared with Tite et al., 1987, Cuprorivaite composition.

Element (%)	Sp.1	Sp.2	Sp.3	Tite et al. 1987	Cuprorivaite composition
Na ₂ O	1.87	1.99	1.96		
MgO	1.10	1.25	1.30		
Al ₂ O ₃	2.25	2.40	2.44		
SiO ₂	63.83	62.12	62.47	60-70	64
SO ₃	3.95	3.09	4.56		
K ₂ O	1.43	1.25	1.28		
CaO	12.76	12.63	13.54	7-15	15
Fe ₂ O ₃	1.34	1.45	0.92		
CuO	11.48	13.83	11.55	10-21	21

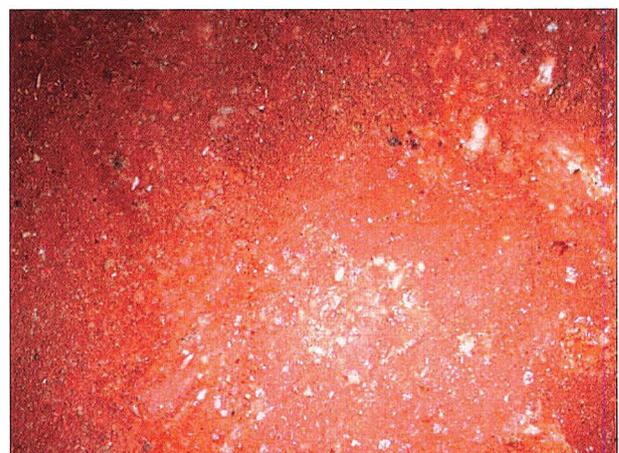


Fig. 19 Microscopic view massive granular aggregate particles appear to be uniform of red pigments.

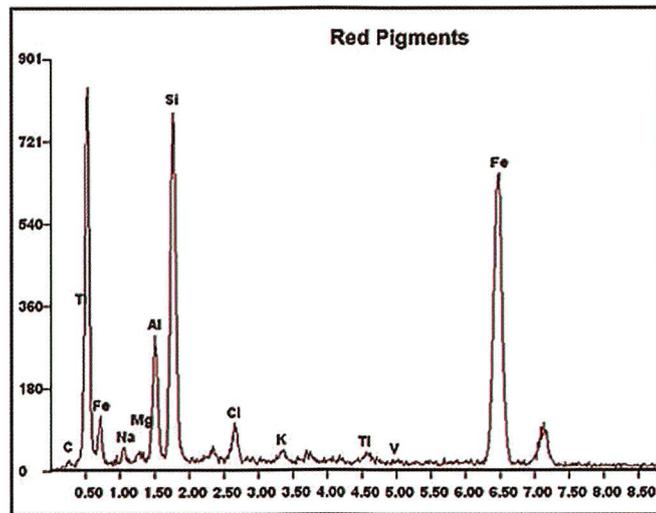


Fig. 20 EDX analysis of the red pigments.

color range that can differ from deep red or brownish to orange and finally to bright yellow. Red ochre was used in Egypt from the 5th Dynasty till the Roman times. Lucas [Lucas 1962] recorded the Dakhla Oasis, Western Desert as provenance for good ochre.

(3) Yellow color

The yellow layer was compact and homogeneous. Its chemical constituent confirmed that yellow ochre, where the yellow was given by goethite, had been mixed with gypsum. Inclusion of red which, when confirmed by analysis, showed that its major component was iron.

(4) Brown color

It was formed mainly of brown ochre as hematite mineral (Fe_2O_3) was the main component identified, plus aluminum and silicon detected by EDS analysis, suggested that the brown ochre was used as brown color. The brown color resulted to the contained manganese as indicated from the EDS analysis (Fig. 21 and Fig. 22).

(5) Grey color

Grey pigment was produced by applying black (carbon black) over white gypsum layer or mixing of black with white gypsum (Fig. 23), while dark grey pigment may be applied over an unprepared surface as we can see that the painting layer shows irregular lines with different thickness.

(6) White color

White pigment produced by spreading a film of gypsum plaster (hemihydrate, $\text{CaSO}_4 \cdot \frac{1}{2} \text{H}_2\text{O}$), which was the same as the one used for preparatory layer but was less thick and had a compact surface.

(7) Black color

The black pigment layer is uniform and contains fibrous particles, sometimes being applied directly on the coarse irregular plaster layer (Fig. 24). The presence of fibrous particles suggested that the black color was produced using carbon black, deriving from wood charcoal [Pallecchi et al 2009] indicating that the anatomical structure of wood was one of the first characteristic elements that stood out in the fibrous particles. Black charcoal was found to be mixed with white or applied over white in grey samples.

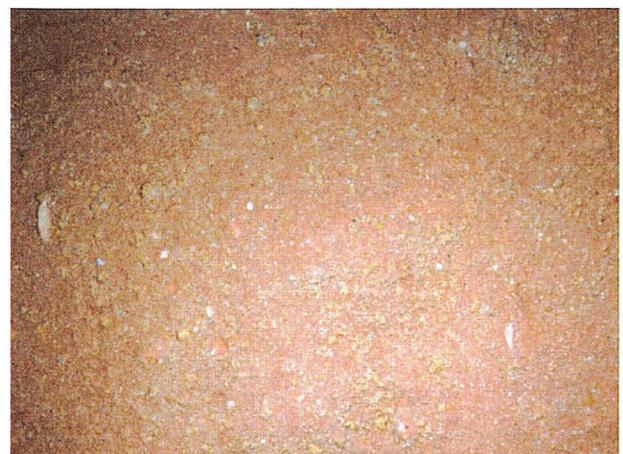


Fig. 21 Microscopic view massive granular aggregate particles appear to be uniform of brown pigments.

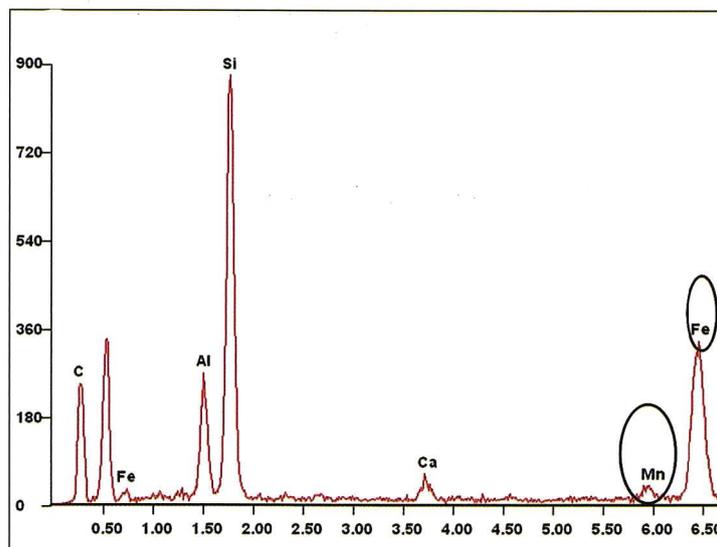


Fig. 22 EDX analysis of the brown pigments.



Fig. 23 Microscopic view show grey pigments prepared by applying black carbon over white (left) and dark grey pigment may be applied over an unprepared surface, note, the fibrous present.

2.5 Conclusions

The paintings of the inorganic pigments from Old Kingdom were performed, signifying extensive usage of pigments frequently used in ancient Egyptian wall paintings. The paintings in Idout burial chamber were produced with a palette formed of five basic colors: white, red, blue, yellow and black that were produced using various hues. Red, yellow and brown ochres were used for the creation of red, brown and yellow pigments; hematite in the first and second case, and goethite in the third, are the iron minerals that provide the colors. White pigments produced by spreading a thin film of gypsum, which was the same as the one used for the preparatory layer. Blue appears to be produced from pigment taken from Egyptian Blue, based on the presence of mineral cuprorivaite. Two types of



Fig. 24 Microscopic view show the uniform black pigments, note the fibrous particles of the wood charcoal.

blue pigments (dark and light blues) were distinguished. Natron was the alkali used and might come from Wadi el-Natron. Quartz sand present in the area of manufacturing was used as silica source.

Two layers of plaster were noticed: the coarse one with higher thickness, consisting mainly of quartz, calcite and gypsum: the fine white wash with thin irregular layers consisting mainly of gypsum with little or without quartz.

3 Conservation and Treatments of the Mural Paintings Inside the burial Chamber

3.1 Introduction

This work builds upon our team. Earlier bibliographies including those that appeared in Akarish and Shoeib [2007; 2011], Akarish *et al.* [2011], Shoeib *et al.* [2007; 2011], Suita *et al.* [2007], clarify the causes of deterioration and the conservation and treatment work.

In this section (chapter) we are going to shed light on and provide more than ten years of the conservation and treatment work of the wall paintings inside the burial chamber of Mastaba Idout.

Our previous studies that lasted more than two years (see causes of deterioration, this book) indicate that the bedrock of the burial chamber in which the painted plaster was applied on mostly very fragile marlstone and argillaceous limestone), including a high proportion of expandable (smectite) clays. Previous petrographical and mineralogical investigations, carried out by our mission, showed the presence of several problems that led to the detachment of the paintings from their original support (bedrock), especially the paintings of West, North and East walls. The mother rock of these walls contributed to the deterioration of the paintings and, in turn, caused the plaster and the painted layer to detach from the bedrock. These rocks are too fragile to resist the interaction between exogenic (climate, including rain water, humidity, and temperature variation) and endogenic (related to the nature of the rock types) conditions, since their construction thousands of years ago.

These agents of decay often act in conjunction and also potentiate one another [Mora *et al.* 1984]. As a result, the bedrock has suffered from severe deterioration resulting in the painted plaster preserved only in fragments. A large area of the plaster has already fallen off the walls, and has been broken down into smaller pieces that accumulated on the floor [Akarish 2007; Akarish and Shoeib 2011; Shoeib *et al.* 2007; Suita *et al.* 2007].

These conservation problems made it clear that the traditional methods of conservation could not be applied to the studied tomb. Therefore, it was assumed that the best way of saving the paintings in the Idout burial chamber would be to detach them from the bad bed-rock; this is the only way to isolate the painting from the influence of their original destructive support. A decision was made to detach the paintings of the North, East, and West walls using a "Stacco" technique [Mora *et al.* 1984] and then to place them on an auxiliary support formed of synthetic materials and to mount them back into their original location. Stacco technique is the mechanical detaching of the paintings together with a part of the plaster layer.

The International Charters for the conservation of monuments [ICOMOS 1964 and 2003] state: "A mural painting is an integrated part of the architectural it completes. Therefore, any separation of the painting from its original constitutes an essential and irreversible alteration of both" (Article no. 7 and article no. 63 respectively). In exceptional circumstances, however, as in the case of catastrophic situations, detachment may be necessary. Accordingly, we were forced to use the detachment process for these walls because of their severe condition.

3.2 Restoration and Treatment Works

In practice, there are many kinds of materials for facing. Also, there are many techniques and skills detachment methods. In the beginning, we had to take into consideration of the environment peculiar to the subterranean and closed room of the burial chamber. This means that the common solvents like acetone or toluene, used in abundance in many processes of restoration works, might be dangerous and harmful for workers, for they are

flammable and toxic. Their use should be limited to inevitable works as much as possible. The work that needs the largest quantity of solvent is concerned with the facing process: the consolidation of some number of flexible materials on the plaster surface with resin, and the dissolving of the resin at the removal of facing layers. Then, we have adopted the Japanese traditional technique of facing: Funori glue, rayon paper, and Kozo paper. The Funori is a natural resin, *gloiopeltis* glue, made of seaweed, which needs just water as solvent. Rayon paper and Kozo paper are flexible and strong. The latter, Kozo paper is a Japanese paper, made of a kind of mulberry tree. This method combination is being recognized among restorers in the world nowadays. The most merit of this method is due to it being "environment-friendly." It means there is no harm to workers and no bad influence on ancient materials in the future.

We started the restoration works in 2005 by developing this new technique applied for Egyptian mural paintings in the Saqqara area. A series of successive processes of restoration and treatment works were achieved and described here as shown below.

A. First Rescue

We picked up hundreds of fallen fragments that were accumulated on the floor as a "first rescue." Then we cleaned, numbered, consolidated, documented the fragments and kept them into

plastic containers. Smaller fragments were also collected through sieving the sands of the burial chamber floor. A plan for reassembling these fragments and reattaching them back to their original places is in progress by the mission

B. Detachment

In the burial chamber, there are four walls of painting, all of which are in severe condition. Among them, the west wall was most damaged since the opening of tomb in the early 20th century. The plaster of the west wall is very thin and it peeled off more than on other walls. So, we had a plan to detach the west wall at first, then the north wall adjoining to the west, next, the east wall. As for the south wall, it is the most beautiful part in Idout burial chamber. We treated it in situ as will be explained here after.

(1) Fixation

The first step of the restoration was the fixation work. Fixation treatment was executed on the paintings using lime-and-sand fillets and through use of a painting knife alongside of the edge of painting plasters, to prevent the plasters from falling off any more (Fig. 25).

(2) Facing

Before applying the facing layers, the paintings were consolidated and fixed with a very dilute baraloid B72 dissolved in acetone. Next the pre-



Fig. 25 Fixation

pared surface was covered by facing. The Japanese paper carriers were applied with Funori glue. First thin Rayon Paper was applied, followed by thick Rayon Paper and Kozo Paper. The last layer was an Egyptian cotton canvas (Fig. 26).

(3) Detachment Processes

The paintings were detached from their original support using long knives and an electric saw. After detaching the paintings from the walls, the paintings were hung on strips of canvas, protecting them from falling and being crushed (Fig. 27). Small pieces of the paintings were left on the wall during the detachment processes (Fig. 28) due to the insufficient strength of the Funori facing. These pieces were separately detached and re-put into their original places again.

(4) Backside Treatment Work

A. Smoothing and Strengthening

The paintings were transferred to a working table in an upside down position (facing down). The next stage was to smooth the back side. Excessively protruding fragments were sanded down and the cavities were filled with new mortar. The flatter the surface of the back, the easier it is to bind it with subsequent layers. Partial removal of the mortar helps to reduce the thickness and the weight of the paintings, which is in return beneficial for mounting them back in to their original location. The flatter the surface of the back, the easier it was to bind it with subsequent layers. The smoothed surface was covered with an insulation layer of ARGE-ARD, or Primal AC-33. Next, the cotton canvas was attached on the back side using a dispersion of polyvinyl acetate as a lining layer

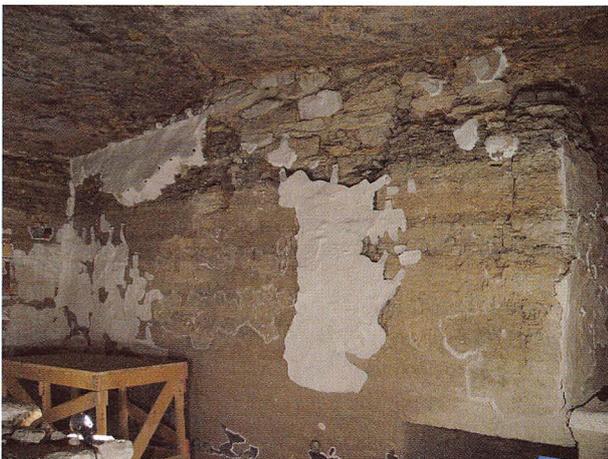


Fig. 26 Facing Layers (Kozo, Rayon and Cotton Materials Laminated with Funori glue)

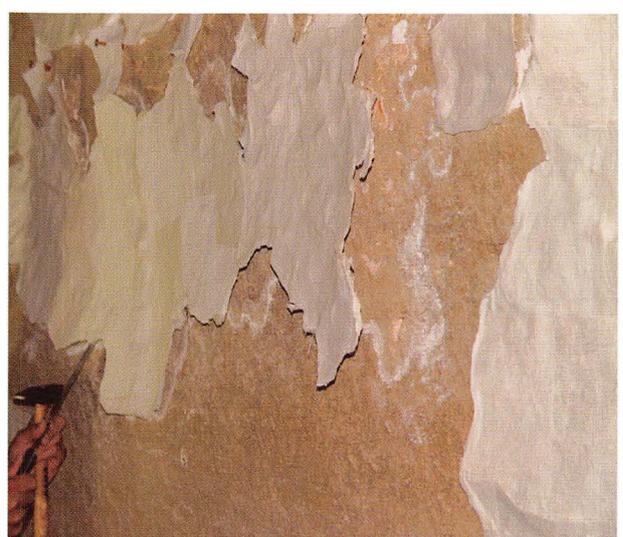
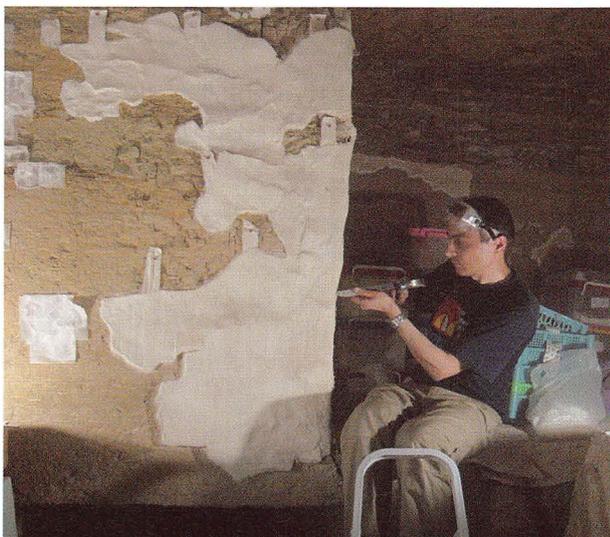
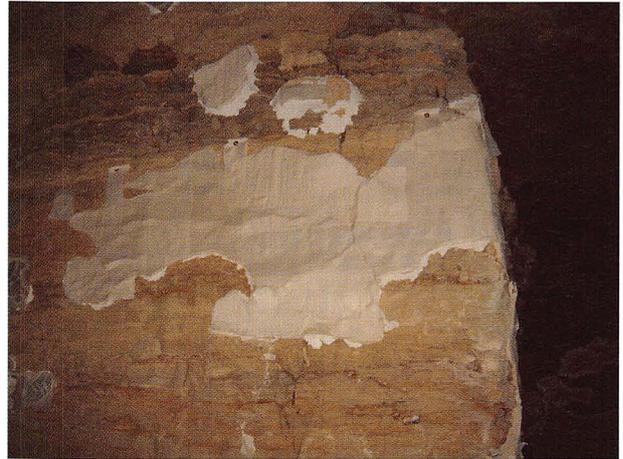


Fig. 27 Detaching of a painting fragment (left) and paintings are hanging on the straps of canvas (right).

(Fig 29 and Fig. 30).

B. Auxiliary Support

To assure the reversibility of the treatment, a polystyrene foam (as an intervention layer) was glued on the lining layer with thermoplastic glue. The paintings were placed on a sandwich-type support, light and durable, which consisted of three layers. First, a layer of carbon fiber, saturated with epoxy resin, was attached. Next, a core-layer of polyester non-woven tissue, Firect Coremat, was attached, followed by a second layer of carbon textile. All the layers were saturated with the same resin. Firect Coremat in contact with the resin became ductile and could be shaped easily. Afterward, the excess of the auxiliary support was cut off, the border of the pieces (consolidation with 20% aqueous dispersion of Primal AC 33) was protected, and the cavities were filled with white, acrylic mortar (Fig. 31 and Fig. 32).

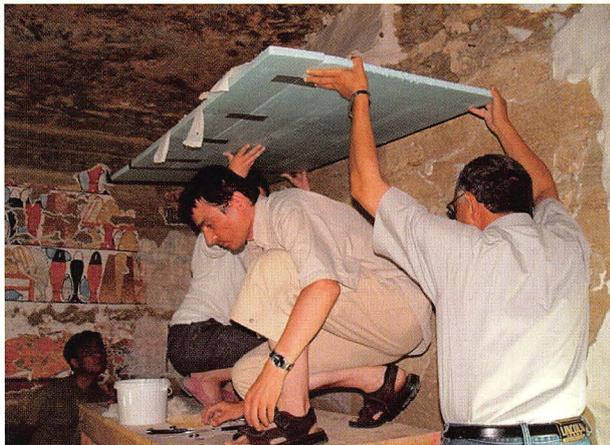


Fig. 29 Transferring the detached Paintings from its walls support to a working table.



Fig. 30 Back side treatments: reducing plaster, smoothing the back side and filling cavities with new mortar.



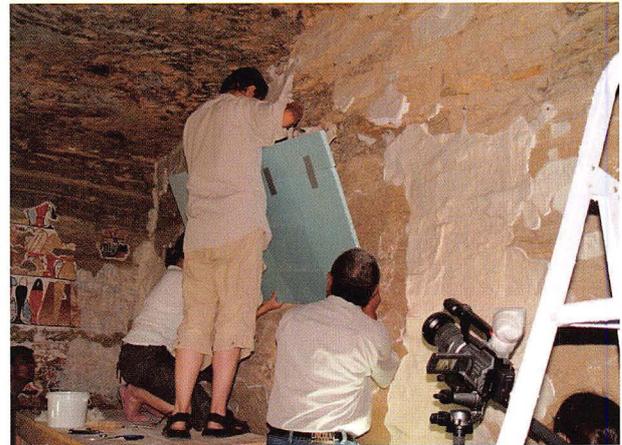
Fig. 28 Missed fragments during detachment processes.

(5) Removal Facing Layers

After turning the paintings face-up, the facing was removed with water. The process of removing the facing turned out time-consuming and required much caution. Funori dissolves in water very well and keeps a good fixing on the paintings with a water-resistant agent. These features are crucial for the safety of the procedure (Fig. 33).

(6) Tentative Re-attachment

Finally, handles were stuck to the back of the piece with epoxy resin, and the paintings were mounted back, tentatively, to their original location



on the wall support (Fig. 34). This way, we were able to isolated the paintings from the influence of their original destructive support.

3.3 Restoration and Treatment Works of South Wall

(1) Condition of South Wall

In contrast to the bad condition of the paintings on the West, East and North walls, the condition of the paintings on the South wall was quite good. The bedrock of the South wall consists mainly of calcite and quartz. Petrographically it consists mainly of micrite that mostly recrystallized into micro sparite. The rock is highly silicified, where silica has replaced carbonate grains and/or filled the pores, which may have helped to straighten the rock. This helped it to survive, to some extent, the deterioration process that affected the other walls

[see Shoeib *et al.*, 2011]. However, the painting has suffered from several problems. The support has a large absorptivity, meaning that the mortar has bent which caused the painting to become slightly separated from the support. Also, the plaster ground was fragile; it had a lot of fine fractures and required strengthening (Fig. 35). In many places, the ground was separated from the wall and some places, brown patches are present on the surface of painting layer (Fig. 36), probably due to the effect of microorganisms. The pictorial surface showed many fractures. For these reasons, the South wall also required some conservation such as (1) strengthening the support, the ground and the painting layer, (2) replacing some of the mortar bends with new ones, (3) removing the rayon paper, and (4) increasing the adhesion of the paintings to the support. In addition, the causes of the ob-



Fig. 31 Polystyrene as an intervention layer.

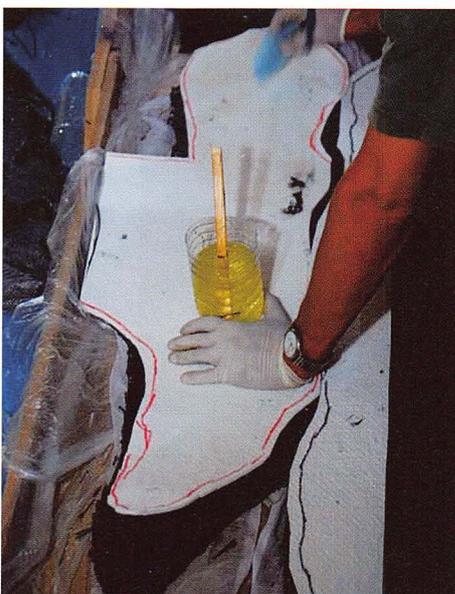


Fig. 32 Execution of new "Sandwich" type of auxiliary support: separation sheet made out of a fire coremat (left), a layer of carbon fiber (right).

served brown spots were investigated.

Accordingly, an in situ treatment using suitable consolidants should be carried out. On the wall at the burial chamber, some consolidate materials have been tested [see Shoeib *et al.* 2011].

Field and laboratory tests of the selected consolidants (Nonorestore, Seven Chemical and Silicon Wacker Steinfestiger OH K5E 300) showed that Silicon Wackor OH and Nonorestore could be used for the consolidation process. However, we found that Nanorestore is preferable because it adhered

well to the separated and disintegrated particles of the substrate. Also, Nonorestore is formed of a Nano- Lime dispersion and as a calcium hydroxide Ca (OH) dispersed in a mixture of ethylic- isopropilic acid. Therefore, it can be applied safely on carbonates of archeological wall paintings.

(2) Field Treatments

The following treatments were carried out to conserve the paintings of the South wall.



Fig. 33 Removal of the Funori facing from the surface of painting.

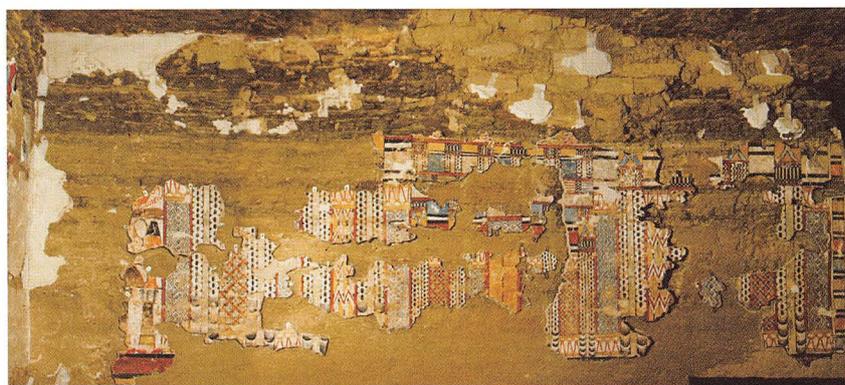
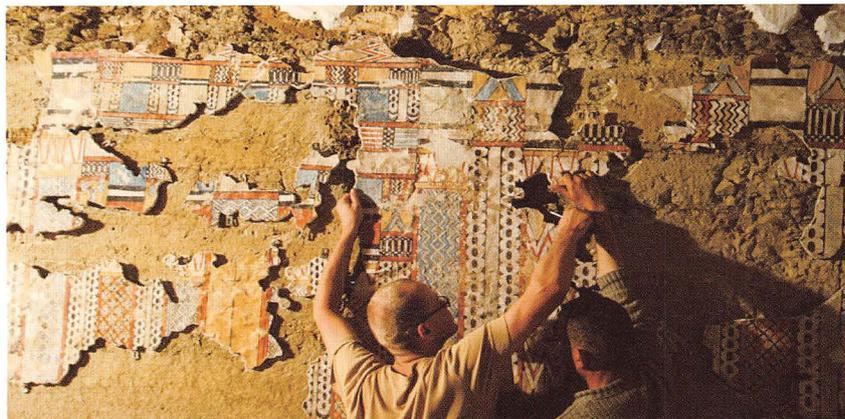


Fig. 34 Reattaching (tentatively) in the Right Order, to its original locations.

A) Microorganism treatment: Preventol RI 80 biocide was tested as an antifungal treatment and produced good results. Therefore, the biocide was used as an antifungal to disinfect the brown patches present on the surface of painting layer. Preventol RI 80 (3% in water) was normally applied three times on the surface of the brown patches through spraying.

B) As mentioned above, Nanorestore was used as an inorganic consolidation material for conservation of the South wall. Nanorestore was applied using the injection method at the back of the painting. The application was done four times and did not cause any color change of the painting layer and was enough to strengthen the painting. Nanorestore was also used to strengthen the support (applied three times). The injection was done specifically through the existing fractures and holes. Mortar bends were also covered by Nanorestore, by brushing it five times (Fig. 37).

C) One of the largest obstacles to conservation problems was the fast absorptivity of the support. In order to reduce this absorptivity, tests using a solution of thermoplastic resin Paraloid B-72 [Lascaux] and a solution of Plexisol P550 were carried out using the injection method. In addition, Malta 6001 (Bresciani), hydraulic lime-based liquid mortar, was applied directly on to the support. Choosing the most suitable resin was important for reducing absorptivity, helping the support consolidate, and increasing the adhesion of Malta 6001. Plexisol P550 (5% in Acetone) was applied three, five and seven times. However, it did not strengthen the support. Malta 6001 did not show good adhesion to the rock which in turn, crumbled. Paraloid B-72 (5% in Acetone and Alcohol 1:1) was applied three times and gave the best results. Accordingly, Paraloid B-72 was used to help in the consolidation of the support. As mentioned, Paraloid B-72 was carried out through injection. Malta 6001 did not crumble and provided good adhesion to the support, after Paraloid B-72 was injected. Malta 6001 was used as a filler for the air-pockets.

D) The existing mortar bends, separated from the support and the painting, were replaced with new ones (Fig. 38). Some of the mortar bends which had good adhesion to the painting were left,

and the paintings were only strengthened with the new lime-sand mortar layer mixed with a small amount (Fig. 14). Mortar bends were covered by Nanorestore five times, by brushing of Acril 33 (acrylic resin in water dispersion), which was then covered by Nanorestore a few times

E) Cleavages were reattached with the solution



Fig. 35 Mortar bends slightly separated from the support plaster upper and Fine fractures on plaster ground lower.



Fig. 36 Brown patches on the surface of painting layer.

of Paraloid B-72 (10% in Acetone). In the larger area losses (cracks, holes), the filling was applied with a spatula using lime-sand mortar mixed with Acril 33.

F) Small and big air pockets remained as a conservation problem, posing a danger to the constancy of the object. Smaller air pockets were filled with Malta 6001 in addition to Preventol 1 RI 80, which was injected because of the microorganisms. The bigger air pockets were not filled because of the load. The blisters were saturated with Alcohol (99%) before hand (Fig. 39).

G) Finally, the facing layer was removed with water. However, in some parts of the paintings it was too dangerous to remove the layer and the rayon papers were left there. Fig. 40 show the paintings of the South wall after the treatment processes were completed.

3.4 Conclusions

The wall paintings of the West, East and North walls inside the burial chamber of Mastaba Idout were detached from their harmful support using the "Stacco method". With this, the fragments were

detached from the walls. The backside of the fragments were sanded down and leveled, and the fragments were consolidated and lined with a cotton canvas and polyvinyl acetate glue. After the intervention layer and auxiliary support were applied, their extensions were cut down. The facing was also removed and the cavities were filled with white, acrylic mortar. Handles were stuck to the back of the pieces with epoxy resin and the pieces were reattached on the walls.

The condition of the paintings on the South wall were better than on the other walls. Accordingly, an in situ treatment using Nanorestore, as an inorganic consolidant, for conservation of the South wall was carried out. Nanorestore was applied to consolidate both the paintings and the support by using the injection method. Paraloid B-72 with a different concentration was also used to reduce absorptivity, to help in the consolidation of the support, and to improve the adhesion of Malta 6001 to the support and to fill cleavages. Malta 6001 was also used to fill the air pockets. Preventol RI 80 biocide was used as an antifungal to disinfect the brown patches present on the surface of painted layer and biocide was applied through spraying, three times, on to the surface of the brown patches.

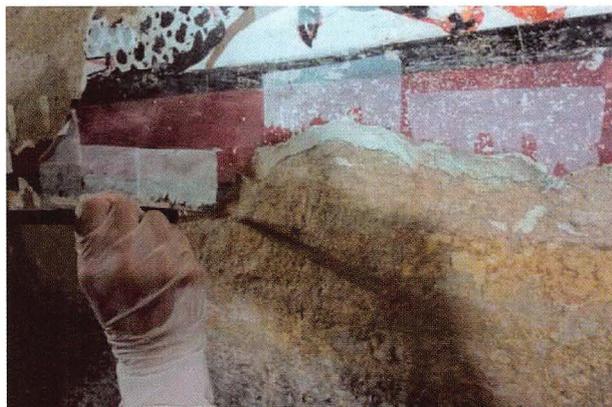


Fig. 37 Mortar bends covered by Nanorestore five times by brushing.

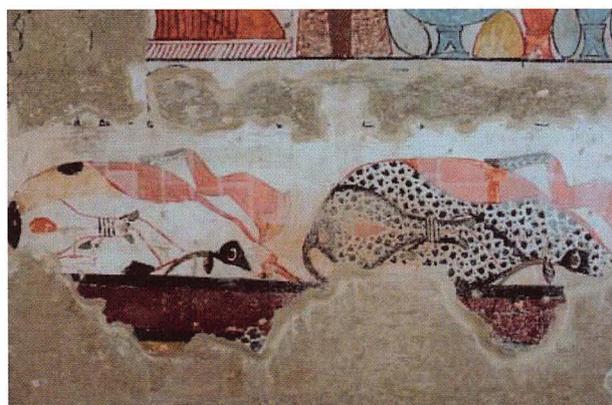


Fig. 38 New mortar bends

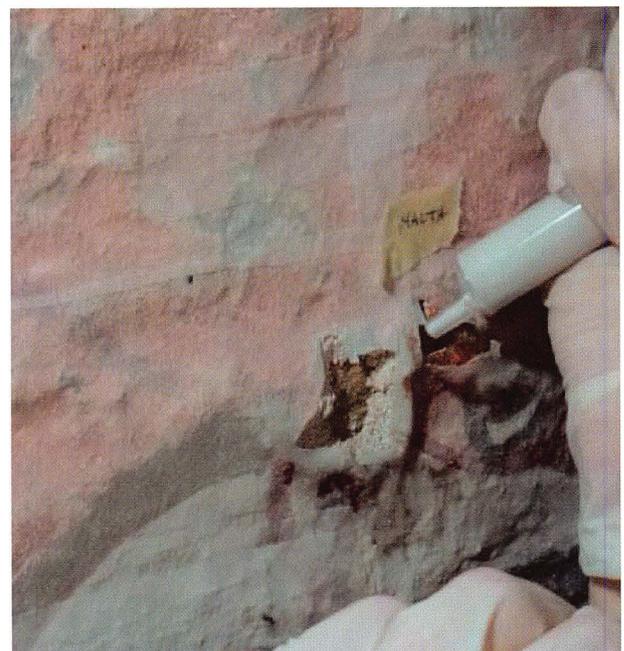
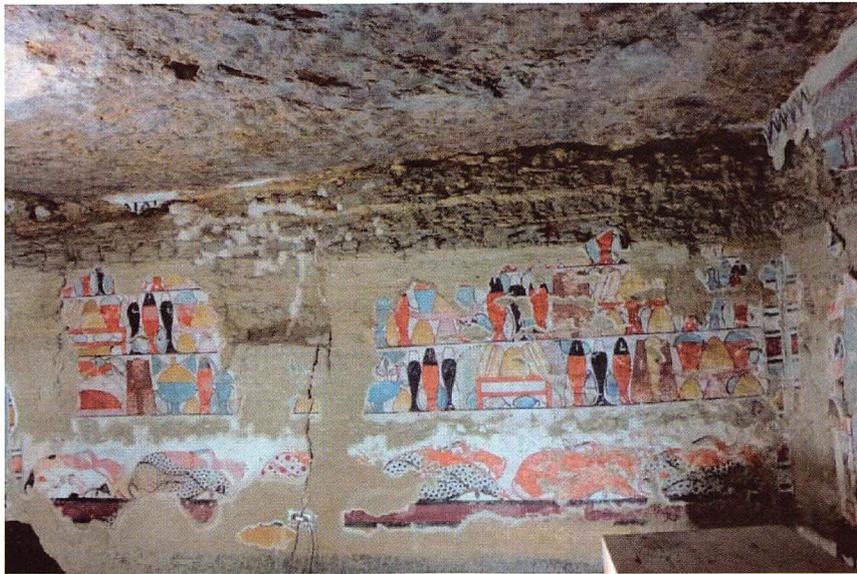
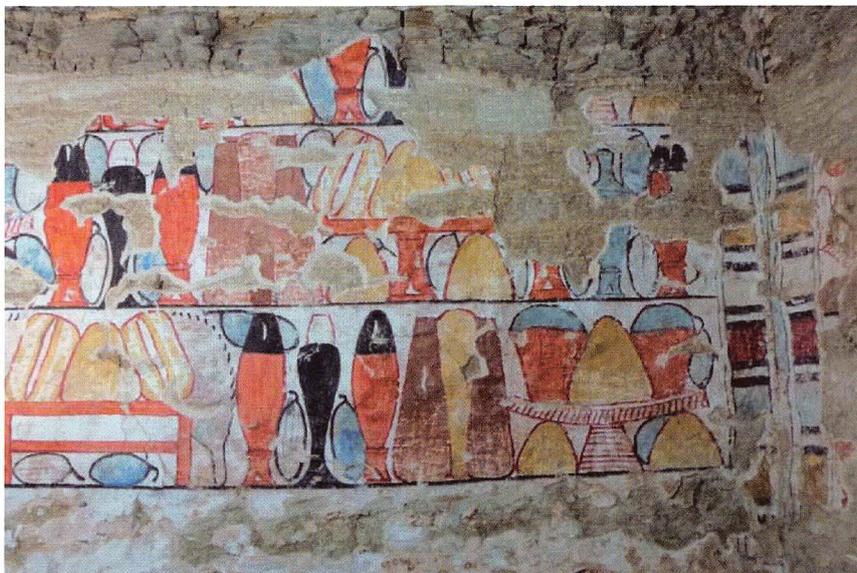


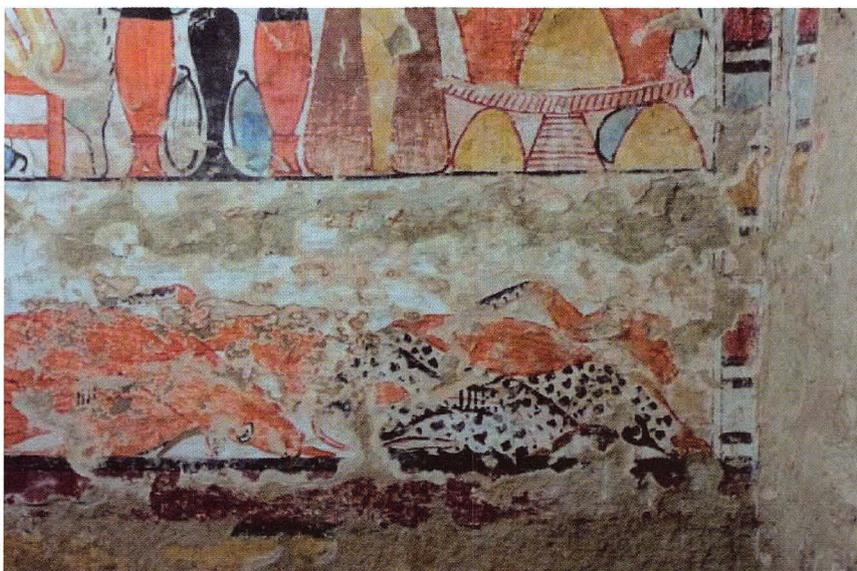
Fig. 39 Air pockets filled with Malta 6001 and Preventol using injection.



A



B



C

Fig. 40 The paintings of south wall after treatment processes.

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