

## Investigation of the Bond Mechanism between Stones or Bricks and Grouts

Chryssi-Elpida Adami and Elizabeth Vintzileou

*National Technical University of Athens, Faculty of Civil Engineering, Laboratory of Reinforced Concrete, Athens, Greece*

Eleni-Eva Toumbakari

*Hellenic Ministry of Culture, Office for the Preservation of Acropolis monuments, Athens, Greece*

**ABSTRACT:** Within the present work, the mechanism of bond is studied in composite grout/substrate specimens. Three types of ternary (lime-pozzolan-cement) grouts are examined, combined with three substrates (two types of limestone and bricks). The interfaces between grout and substrate are characterized by means of mechanical tests in direct tension. The in-time development of the tensile strength is also investigated. In order to correlate the characteristics of the substrates and the obtained strengths, observations on their surface and porosity are made. The main conclusions of this study are that the studied ternary grouts can develop tensile bond strength comparable to Portland cement-based grouts, and that the value of the tensile bond strength is governed by the substrate characteristics. The results of this project confirm the efficiency of ternary lime-pozzolan-cement grouts with a Portland cement content of 30% for the repair and strengthening of historic masonries.

### 1 INTRODUCTION

#### *1.1 Design of grouts for the retrofitting of historic masonry*

One of the main issues in the field of conservation of the built heritage is the use of repair materials, which should be simultaneously (a) physico-chemically compatible to the *in situ* ones and (b) efficient for the repair and strengthening of historic masonries. Previous researches have established that the use of repair materials with a high cement content is not necessary for the level and, especially, the type of strength (compressive or tensile) required by a masonry structure (Vintzileou 2001, Toumbakari 2002). Focusing on grouts, the combination of microstructural and mechanical studies (Toumbakari et al. 1999) permitted the development of ternary lime-pozzolan-cement compositions with Portland cement content as low as 30%. The use of hydrated lime and pozzolans in various proportions ensures physico-chemical compatibility and permits the development of a wide range of mechanical properties, adequate for application in a variety of historic masonries. The efficiency of those compositions was proven through experimental investigation of their effect on three-leaf walls (Toumbakari 2000, 2002, Toumbakari et al. 2004). As far as compressive and tensile resistances are concerned, three-leaf walls grouted with ternary grouts and with a reference cement-based grout (Portland cement content 80%) presented a similar behaviour. The effect of each grout type was perceptible in the values of the modulus of elasticity and the transverse horizontal (out-of-plane) deformation of the grouted walls. It was shown that the main property affecting the behaviour of grouted walls is the shear bond strength of the grout-substratum interface. Higher bond strength makes that the separation between the external and internal leaves takes place at a higher stress level. Consequently, the efficiency of grouting is controlled by the bonding capacity of the grouts and not by their compressive strength. The importance of bonding and the very limited number of studies dealing with this issue in the field of historic masonry calls for a systematic study, which fo-

cuses on the bond properties of ternary grouts and the behavior of interfaces between existing materials and grouts (Adami 2006).

### 1.2 Parameters affecting bond strength

Bond between building materials presents two components, namely chemical and mechanical. Chemical bond is due to the different chemical reactions that develop between the mortar or concrete materials and the substratum. The first studies suggesting that a difference exists between the paste surrounding aggregate in concrete and the bulk paste, are those of Farran (1956). Later, Lyubimova & Pinus (1962) performed a series of microhardness measurements across cement-aggregate interfaces and found differences between the zone immediately adjacent to the aggregate and the bulk cement matrix. Those results suggested the occurrence of an area adjacent to the aggregate, whose properties differ from the rest of the cement matrix. Since then, this area, called Interface Transition Zone (ITZ), was extensively studied. Among the factors affecting chemical bond is the mineralogy of the substratum, the porosity of its surface, the type of the binder, the presence of pozzolanic or inert fines. Chemical bond prevents slip that can occur across an interface but it is destroyed when slip initiates. On the other hand, chemical bond is fundamental for durability issues.

Mechanical bond also depends on characteristics of the substratum, such as strength, surface porosity and pore size, roughness, aggregate water content and water absorption capacity. It depends, moreover, on properties of the binding material, namely its strength, and thickness, as well as the applied normal stress at the interface. The mobilization of the maximum resistance of mechanical bond is achieved for a slip value, which in turn depends on the materials' characteristics and the applied compressive force. A very important characteristic of mechanical bond is that part of bond strength is conserved even at high slip values. This issue is of great importance for the bearing capacity of structures, especially under earthquake actions.

## 2 EXPERIMENTAL STUDY

### 2.1 Materials

In this study, the investigation of the tensile strength of the interface between grout and substrate demanded the preparation of composite specimens, made of two aggregate pieces connected with a grout layer. The selection of three types of substrate was based on their frequent appearance in historical buildings: two types of limestone with different porosity (Dionysos marble and travertine) and bricks were used. According to tests carried out at the National Technical University of Athens (Vardoulakis & Kourkoulis (1997), Vardoulakis et al. (2000)), the compressive strength of Dionysos marble was equal to 83 N/mm<sup>2</sup> and 70 N/mm<sup>2</sup> in its strong and weak direction respectively. Its strength in direct tension was equal to 8.7 N/mm<sup>2</sup> in the strong direction of the marble. The available data regarding the tensile strength of the marble in its weak direction were not considered to be reliable (Vintzileou et al. 2006). The apparent porosity of the marble was equal to 0.2%. Referring to travertine, it has to be noted that this type of stone has a very variable quality. The presence or absence of a large amount of pores and argillaceous discontinuities affects mechanical performance. Indeed, the tests carried out showed a wide range of flexural (from 4.0MPa to 21.5MPa) and compressive strengths (from 12.1MPa to 95.5MPa) and apparent porosities (from 2.9% to 16.8%) of travertine stones. Consequently, it is expected that these characteristics would in some cases affect the failure mode and the value of the interfacial tensile bond strength of the respective composite specimens. Finally, the compressive strength of the used bricks was equal to 12.2MPa while their apparent porosity was equal to 21.4%.

The materials used for the grouts were: hydrated lime (HL), Portland cement CEM I 42.5 (C), commercial pozzolan from Milos (0-75 µm) (LA), commercial metakaolin METASTAR 501 (0-16 µm) (MK). The mix proportions of the grouts are presented in Table 1 (Adami, 2006). Grout G1 serves as reference. The Portland cement content of the ternary grouts is 30%. The selection of the lime to pozzolanic materials ratio is based on the estimation that, outside the used ratio, either the lime or the pozzolanic content would be too high to allow for the optimisation of the pozzolanic reaction: In either case, an important part of the material would remain unreacted and would therefore not contribute to strength through a hydration mechanism (Toumbakari,

2002). The W/S (solids) ratio was between 0.8-0.9 (for grouts G1 and G4) and 1.1 (for grout G2) in order to achieve penetrability to voids smaller than 0.3mm. To increase fluidity, superplasticizer was used. The grouts were prepared by using a mechanical mixer at 2400 revolutions per minute. The use of fine metakaolin did not require the use of specific mixing devices, even though the resulting increase of fineness led to an increase of the water content from 0.8-0.9 to 1.1.

Table 1 : Mix proportions of the studied grouts.

Ref.No of the grout	Cement	Composition [%-wt]		
		Lime	Metakaolin	Milos earth
G1	80	20	-	-
G2	30	35	35	-
G4	30	47	-	23

The main mechanical properties of the grouts are shown in Table 2. The development of the compressive strength of grouts is initially controlled by the cement content. This is expected, since the pozzolanic activity (with pozzolan grains ground to cement fineness) appears only after approximately 2-4 weeks. The compressive strength of the grouts containing 30%-wt OPC continued to increase due to the progress of the pozzolanic reaction. As far as the achieved strengths are concerned, grout G2 reached in 230 days the same compressive strength as grout G1 in 90 days, thus highlighting the potential of those ternary blends, provided the lime to pozzolan ratio is optimised. Despite some fluctuations in the obtained values, flexural strength of the ternary grouts generally increased (for grout G2) or remained constant (for grout G4). It is known that flexural strength is a mechanical property very sensitive to internal microcracking. The increase in the flexural strength of grout G2 highlights the presence of a microstructure with progressively increasing density, thanks to both the pozzolanic reaction and the fineness of the pozzolan. The stabilisation of the value of flexural strength of grout G4 shows a less dense microstructure, which nevertheless is resistant to microcracking (otherwise, flexural strength would have dropped with time).

Table 2 : Compressive and flexural strength of grouts [MPa].

days	G1(C=80%, L=20%)		G2(C=30%, L:MK=1:1)		G4 (C=30%, L:LA=1:2.5)	
7	13.3	3.7	5.1	1.3	1.0	0.6
28	14.6	3.4	9.9	2.0	3.3	1.7
90	17.9	4.5	13.6	1.0	7.6	1.9
230	-	-	17.9	3.3	7.3	1.7

## 2.2 Test program and investigated parameters

In order to examine the in-time development of the tensile strength of the interface of the composite specimens, tests at the ages of 28, 60, 90 and 180 days were performed. The choice amongst the possible combinations of substrate and grout was based on the following assumptions: 1) termination of the pozzolanic reaction of the binders for the compositions containing metakaolin or milos earth by the age of 180 days, 2) practically, no further development of the compressive strength of the cement-based grout G1(C=80%, L=20%) after the 28<sup>th</sup> day. A total of 111 composite specimens were prepared and tested. The parameters selected for investigation within this study, dictated by the relevant literature, were: the type of the aggregate (physico-mechanical characteristics), the type of the binders and their main mechanical and microstructural characteristics and, finally, the age of the composite specimens.

## 2.3 Specimen preparation

Dionysos marble, travertine and brick tablets were cut in rectangular prisms and then glued with epoxy resin in order to form "T"-shape substrate specimens. To simulate the roughness of the inner layers of multi-leaf masonries, the tablet face to be bonded with a grout was mechanically chiselled. The contact surface was cleaned using high-pressure air and then the T-shape speci-

mens were stored for at least two weeks in the humidity chamber at 95% R.H. and 20°C. The preparation of the composite specimens followed. For the injection of the grout in the (3mm thick) joint of the composite specimen, a disposable syringe of a 10 ml capacity (without the needle) or a 20ml capacity (with a 1mm vent needle) was used. Special care was taken, to avoid air penetration in the grout layer: the grout was set very slowly and moreover, the injection was carried out in two phases: firstly, half of the quantity of the grout was injected and then condensed with the use of a metal wire. After that, the rest of the grout was poured following the same procedure. It has to be noted that during the whole procedure of injection and curing, the composite specimens were stored in horizontal position, while the width of the joint remained constant by a confinement system consisting of steel rods and plates. Finally the composite specimens were cured at 95% R.H. and 20°C until the day of testing.

#### 2.4 Experimental Procedure

For the measurement of the tensile strength of the composite specimens the fragment-test method (Tassios et al., 1989) was applied, using a device (Figure 1) that allows for testing in direct tension (Katsaragakis, 1987). The forces were applied by a hydraulic press (max capacity of 300KN). A DBBSE loadcell (max capacity of 10 KN) was bolted to the press and connected to a computer, to which load measurements were transferred.

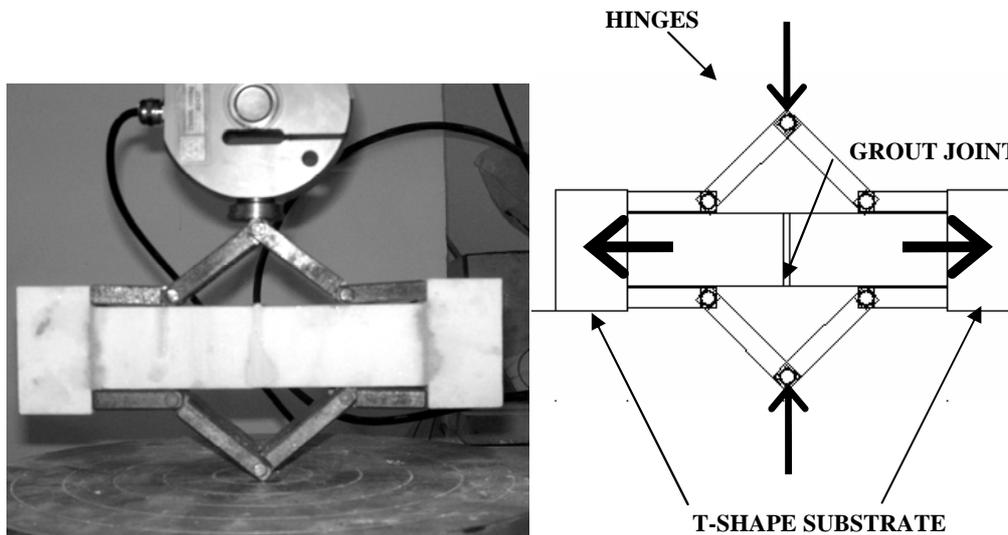


Figure 1: Experimental apparatus

### 3 EXPERIMENTAL RESULTS

#### 3.1 Failure modes of the composite grout/substrate specimens

Four different modes of failure were observed (Figure 2):

1. Mode ITZ (Failure at the interface zone): The grout detaches from the substrate along one interface, whereas it remains attached to the other interface with the substrate. In other words, the plane of failure occurred along a grout/substrate interface.
2. Mode Z: Similar to failure mode ITZ. In this case, however, practically the one half of the grout remains attached to the one tablet whereas the other half remains attached to the second tablet.
3. Mode E: Failure within the grout (tensile failure of the grout).
4. Mode S: Failure at the substrate.

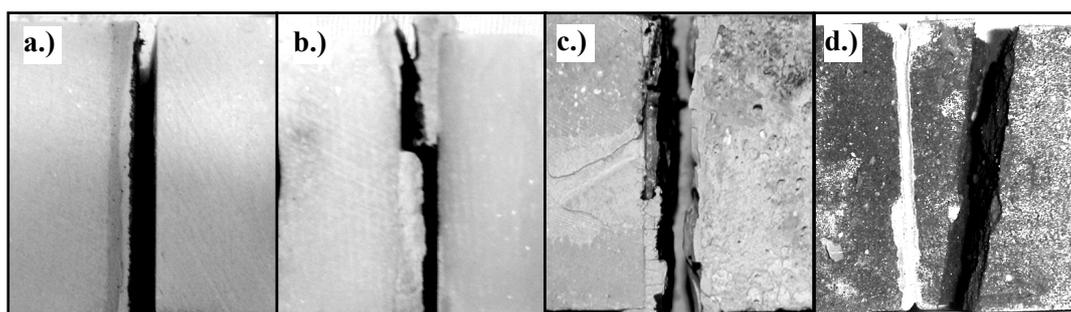


Figure 2 : Failure modes: (a) ITZ, (b) Z, (c) E, (d) S.

### 3.2 Results and Discussion

In this section, the main findings of the research are presented, i.e. the obtained tensile strength for the examined grouts, as well as the failure modes observed for the three different types of substrates. It should be noted that some of the test results are not evaluated here: Specimens in which the grout joint was not full or those with a large amount of air in the joint (due to defective injection procedure) were discarded. Moreover, specimens with partly carbonated grout in the joint were not taken into account because they are not considered representative of the grout situation at the interior of a masonry wall, where carbonation is not expected to occur. Furthermore, specimens that failed in the substrate away from the interface are excluded as well.

### 3.3 Composite marble/grouts specimens:

In Figure 3, bond strength of the interfaces between the three grouts and marble substrate are plotted in function of the age at testing. The lowest tensile strength (0.23MPa) was measured for the ternary grout G4 with Milos Earth (pozzolan), whereas the highest tensile strength value (1.72MPa) was obtained for the metakaolin based grout G2.

Regarding the in-time development of interface strength, the following can be observed: for grout G1(C=80%, L=20%), the tensile strength exhibited a small decrease between the 28<sup>th</sup> and the 90<sup>th</sup> day (from 0.93MPa to 0.85MPa respectively). This reduction lies within the acceptable margins of scatter of experimental results, related to a very sensitive property like tensile strength. For grout G2 (C=80%, L:MK=1:1) the early formation (at 28<sup>th</sup> day) of the C-S-H gel provided the mechanical interlock between marble and grout G2 and thus enhanced the tensile bond strength. Then, an unexpected fall of the strength occurred, which cannot exclusively be attributed to the scattering of the testing method. It has, however, to be noted that the flexural strength of this grout also decreased between the 28<sup>th</sup> and 90<sup>th</sup> day (Table 3). It is possible that this strength drop is responsible for the drop in the bond tensile strength as well. Further research is therefore needed, in order to understand this strength evolution. Finally, the increase between the 28<sup>th</sup> day and the 180<sup>th</sup> day (from 0.33MPa to 0.59MPa) of the tensile strength of the interface of the composite marble/G4 (C=30%, L:LA=1:2.5) specimens was attributed to the densification of the interface due to the pozzolanic reaction, which is slower with the coarser Milos Earth pozzolan. As a summary, the tensile bond strength between marble and grout G1(C=80%, L=20%) or grout G2 (C=80%, L:MK=1:1) was about 1MPa and already completed by the 90<sup>th</sup> day. On the contrary, the ascending curve of the marble/G4 bond tensile strength with time, probably due to a slowly developing pozzolanic reaction, permits to expect a further increase of bond tensile strength at a later age.

Regarding the failure mode, the following observations were made: all composite specimens with grouts G1(C=80%, L=20%) and G4(C=30%, L:LA=1:2.5) failed along the interface (Mode ITZ). For grout G2 (C=80%, L:MK=1:1), the specimens failed either in Mode ITZ or in Mode Z or due to tensile failure of the grout (Mode E). The observed failure modes are considered to prove the improved interface properties due to the presence of the fine metakaoline particles. Finally, no noticeable cracking in the grout joint for this type of substrate occurred.

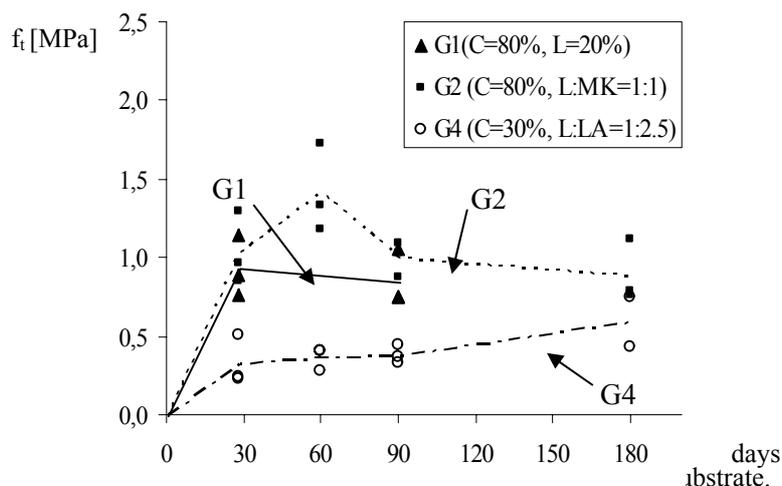


Figure 3 : Bond tensile strength for marble substrate.

### 3.3.1 Composite travertine/grouts specimens

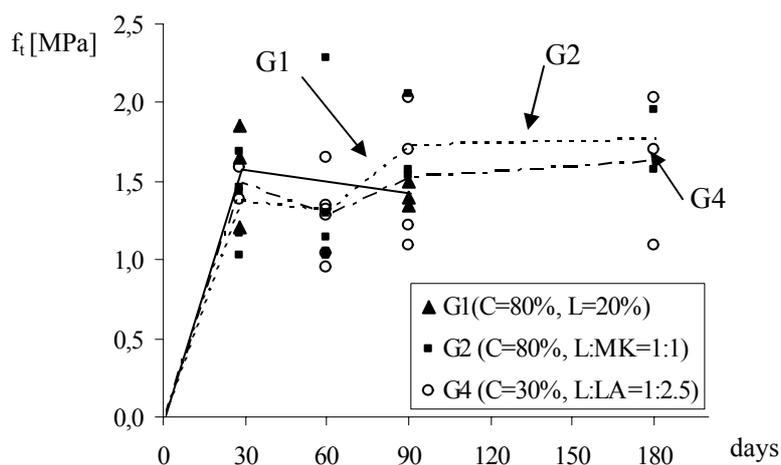


Figure 4 : Bond tensile strength for travertine substrate.

In Figure 4 the experimental results on the tensile bond strength of the interfaces between the three grouts and travertine substrate are presented. The values of tensile strength for grout to travertine interfaces are by more than 50% higher than those obtained for marble interfaces. This can be explained by the higher porosity and surface absorption of the travertine, which led to a better interlocking of the binder to the substrate, as well as a reduction of the water available at the interface. Since high injectability grouts contain a great amount of water, this water reduction does not affect the hydration process. On the contrary, it results to lower interface porosity and a more dense grout/travertine interface. The higher values for the tensile bond strength could possibly be explained also by the diminished wall effect that is induced for this substrate in regard with marble substrates. The mean value of tensile bond strength of the interface for grout G1 (C=80%, L=20%) showed a drop from 1.57MPa at the 28<sup>th</sup> day to 1.41MPa at the 90<sup>th</sup> day. This was again attributed to the scattering inherent to the tensile tests. A growth of the strength occurred for grout G2 (C=80%, L:MK=1:1): the tensile strength of the interface, (equal to 1.35MPa at the 28<sup>th</sup> day) rose up to 1.72MPa at the 90<sup>th</sup> day and then stabilized at 1.75MPa at the 180<sup>th</sup> day. This result, as well as the increase of the tensile strength of the interface of the composite travertine/G4 (C=30%, L:LA=1:2.5) specimens can be explained by the consolidation of the interface caused by the pozzolanic reaction. The value of the travertine/G4 bond ten-

sile strength was 1.48MPa at the 28<sup>th</sup> day and 1.65MPa at 180days. It is remarkable to notice that composite specimens grouted with materials with very different compressive and tensile strength presented a similar bond tensile behaviour of the interface with travertine.

Regarding the failure mode for all composite specimens with grouts G1(C=80%, L=20%) and G4(C=30%, L:LA=1:2.5), the plane of failure occurred along the interface surface (Mode ITZ or Z). For grout G2 (C=80%, L:MK=1:1) the specimens failed mainly due to tensile failure of the grout (Mode E), therefore it can be said that for the latter composite specimens the interface was stronger than the respective bulk paste.

### 3.4 Composite brick/grouts specimens:

For all the composite specimens with brick as a substrate the failure occurred in the brick regardless the grout composition or the age of the specimen. Therefore the results of the test gave the tensile brick strength which was 0.80MPa. Referring to the tensile strength of the interfaces between brick and the three specific types of grouts it can be said that it is bigger than the obtained value of the brick tensile strength (0.80MPa).

### 3.5 Comparison with test results form the literature

At this point, it is necessary to compare with the results of the previous work on ternary grouts (Toumbakari, 2002). The bond behaviour of ternary grouts of various lime:pozzolan contents with a 30% Portland cement content (similar to G2 and G4) as well as a reference cement-based grout identical to G1 was studied at the age of 60 days in tension and in shear on two different substrata (a porous brick and a dense limestone) and the results were correlated to the interface failure mode and microstructure. In the case of brick substratum, the bricks failed in tension instead of the grouts. The obtained bond tensile strength of the ternary grouts in a limestone substratum varied between 1.35 to 1.63 MPa, whereas the reference grout reached 1.76 MPa in 60 days. Those results are in very good agreement with the present study. Bond tensile tests between various dry stones with a grout (joint thickness: 3mm) containing 50%-weight cement, lime and silica fume (compressive strength: 10-12MPa) produced bond tensile strengths ranging between 0.8-1.6 MPa in 28 days depending on the type of the substrate (Miltiadou 1990). Those values fall also within the range reached by ternary grouts made with a lower (30% instead of 50%) cement content and coarser than the used silica fume pozzolans (Rheinisch trass, Milos earth, metakaoline). In the same study, a grout made with 75% cement and 25% lime, thus similar to our reference grout, reached bond tensile strength values between 1.1 to 3 MPa in 28 days.

## 4 CONCLUSIONS

Due to the many factors controlling bond, the efficiency of ternary grouts with a given substrate should be evaluated in comparison to a reference cement-based grout with identical injectability properties. On the basis of the approach, the following conclusions can be drawn:

1. The good properties of high injectability ternary grouts (with 30% Portland cement content and various lime and pozzolan proportions) were confirmed.
2. As far as tensile bond strength of the interfaces between substrates and grouts is concerned, the studied ternary grouts can develop tensile bond strength similar to Portland cement-based grouts, thanks to an improved interface transition zone and also a good bulk matrix microstructure. In this place it has to be made clear that, other grout materials or ternary grouts with different mix design logic may develop different bond tensile properties.
3. The value of the tensile bond strength developed by the reference and the ternary grouts, as designed for this study, is controlled by the substrate characteristics, mainly porosity.
4. The tensile strength at 28 days for marble substrates is similar (1MPa) for grouts G1 and G2. Practically, no further in-time increase of the tensile bond strength is obtained. On the contrary for grout G4 further strength increase is expected.

5. Higher values of the tensile bond strength ( $>1.50\text{MPa}$ ) are obtained for travertine substrate. An in-time increase of the strength occurred for the tripartite grouts G2 and G4, which reached 1.75 and 1.65MPa respectively at the age of 180 days.
6. The tensile strength of the brick/grout interface is higher than 0.80MPa.

#### ACKNOWLEDGEMENTS

The first author gratefully acknowledges the financial support of Onassis Foundation and Leventis Foundation for her doctoral research at the NTUA.

The materials were offered by IMERYS Minerals Ltd and SIKA Hellas.

#### REFERENCES

- Adami, Ch.-E. 2006. Development and study of the physico-chemical and mechanical properties of high injectability lime-metakaolin based grouts. An investigation of the behaviour of the interface zone of existing and retrofitting materials; *Doctor Thesis (under preparation)*, National Technical Univ. of Athens.
- Binda, L. and Baronio, G. 1988. Survey of brick/binder adhesion in "powdered brick" mortars and plasters; *International Masonry J.*, 1988 Spring.
- Farran, J. 1956. Contribution minéralogique à l'étude de l'adhérence entre les constituants hydratés des ciments et les matériaux enrobés; *Revue des Matériaux de Construction et Travaux Publics*, (490-491), pp. 155-172.
- Katsaragakis, E. 1987 A new tensile test for concrete; *Materials & Structures*, 20, pp.120–125.
- Lyubimova, T.Yu. and Pinus, E.R., 1962. Crystallization structure in the contact zone between aggregate and cement in concrete; *Colloid J. USSR*, 24(5), pp. 491-498.
- Miltiadou, A. 1990. Contribution à l'étude des coulis hydrauliques pour la réparation et le renforcement des structures et des monuments historiques en maçonnerie, *Ph.D.Thesis*, ENPC Paris, p.353
- Tassios et. al, 1989. In-situ strength measurement of masonry mortars; *Structural Conservation of Stone Masonry*, International Technical Conference, ICCROM, Athens.
- Toumbakari E.-E., Van Gemert D., and Tassios T.P. 1999. Methodology for the design of injection grouts for consolidation of ancient masonry, *International RILEM Workshop on "Historic Mortars: Characteristics and Tests"*, P. J. M. Bartos, C.Groot, J.Hughes eds, Paisley, Scotland 1999, RILEM Publ. PRO12, pp.395-405.
- Toumbakari E.-E. 2002. Lime-pozzolan-cement grouts and their structural effects on composite masonry walls; *Doctor Thesis*, Katholieke Univ.Leuven.
- Toumbakari E.-E., Vintzileou, E., Pisano F., Van Gemert D.,2000. Development of a model wall for the experimental study of three-leaf masonry walls; *Proc.12<sup>th</sup> Intern. Brick/Block Masonry Conference*, vol.III, Adell J.-M. ed., pp. 1865-1875, Madrid.
- Toumbakari E.-E., Van Gemert D., Tassios T.P., Vintzileou E. 2004. Experimental investigation and analytical modeling of the effect of injection grouts on the structural behaviour of three-leaf masonry walls; *Proc. IV<sup>th</sup> Intern.Conf. on Structural Analysis of Historical Constructions*, Padova
- Vardoulakis, I. and Kourkoulis, S.K., 1997. Mechanical properties of Dionysos marble; Final Report of the Environment Project EV5V-CT93-0300 "Monuments under seismic action", Nat. Tech. Univ. of Athens, Athens.
- Vardoulakis, I., Stavropoulou, M., and Papadopoulos, Ch., 2000. Direct tension tests on Dionysos marble; EU DG XII SMT Programme No SMT4-CT96-2130, Final Report.
- Vintzileou E. 2001. The effect of deep rejoining on the compressive strength of brick masonry; *Masonry International*, 15, 1: pp. 8-12.
- Vintzileou E., Toumbakari E.-E. and Papadopoulos K., 2006. Dowel Action of Titanium Bars Connecting Marble Fragments, *Journal of The Masonry Society* (accepted for publication)