

## Mineralogical and petrophysical characterisation of white Apuan marble

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**ABSTRACT.** — This study contributes to the knowledge of the compositional, mineralogical, microstructural and physical characteristics of the Apuan marbles, focusing the attention on the existing relationships among these different parameters. For this purpose samples of marble, primarily white, coming from different zones of the Apuan Metamorphic Complex, were investigated.

The studied samples show homogeneous chemical-mineralogical composition. They are characterised by high CaO content, low SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, MgO and Fe<sub>2</sub>O<sub>3</sub> amounts. On the other hand, it is clear that the microstructural characteristics vary not only among different basins (e.g., Carrara and Arni basins), but also within a same basin. This is the result of the complex tectono-metamorphic history suffered by the Apuan marble. Samples characterised by grain boundaries from straights to slightly lobates without shape orientation, up to terms characterised by strong orientation and grain boundaries from interlobates to strongly sutured were analysed.

Microstructural parameters were measured through image analysis methods in order to quantify the mean grain size and its distribution and to evaluate the presence of crystals shape orientation and the grain boundaries convolution. The different microstructural characteristics were connected with

the main physical characteristics of the stone material: total open porosity, mesoporosity, pore size distribution, imbibition coefficients, saturation indexes, trends of saturation curves obtained from total immersion and capillarity. It's evident that the microstructural characteristics strongly influence the petrophysical parameters of the examined white marbles.

**RIASSUNTO.** — Questo lavoro contribuisce alla conoscenza delle caratteristiche composizionali, mineralogiche, microstrutturali e fisiche dei marmi Apuani, focalizzando l'attenzione sulle relazioni esistenti tra i differenti parametri. A questo scopo sono stati esaminati campioni di marmo provenienti da differenti zone del Complesso Metamorfico Apuano.

I marmi studiati mostrano una composizione chimico-mineralogica omogenea, sono caratterizzati da elevato contenuto di CaO, basso contenuto in SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, MgO, Fe<sub>2</sub>O<sub>3</sub>. Risulta inoltre chiaro che le caratteristiche microstrutturali non variano solo tra diversi bacini estrattivi (ad esempio tra il bacino di Carrara e quello di Arni), ma anche all'interno dello stesso bacino. Questo è il risultato della complessa storia tettono-metamorfica che ha interessato i marmi apuani. Sono stati analizzati campioni caratterizzati da contatti tra i limiti dei grani da rettilinei a leggermente lobati, privi di orientazione di forma, e campioni caratterizzati da forte orientazione e contatti tra i granuli da interlobati a fortemente suturati.

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I parametri microstrutturali sono stati ottenuti attraverso metodi di analisi di immagine che hanno permesso di quantificare la dimensione media e la distribuzione dei granuli e di valutare la presenza di orientazione di forma ed il tipo di convoluzione tra i granuli. Le diverse caratteristiche microstrutturali sono state messe in relazione con le principali caratteristiche fisiche di un materiale lapideo: porosità, mesoporosità, distribuzione porosimetrica, indici di saturazione, andamento delle curve di saturazione ottenute per immersione totale e per capillarità. È risultato evidente che le caratteristiche microstrutturali influenzano fortemente i parametri petrofisici dei marmi bianchi esaminati.

KEY WORDS: *White Apuan Marbles, Microstructure, Petrophysical characteristics.*

#### INTRODUCTION

The use of marble in sculpture and architecture from the antiquity until today is linked to the history of the human civilisation. The marble is the preferred material for the realisation of anthropomorphous sculptures since ever considered an «humanising» material. In architecture the most ancient examples of the use of marble are represented by the Parthenon (440 B.C.) and the Palace of Mausolo in Alicarnasso (360 B.C.) (Gnoli, 1971). As regards decoration, the use of marble is already attested in Egypt, in the Ptolemaic age.

The first traces of workmanship of the Carrara marble are attested in the Roman Period, when surely the quarries in the slopes of Monte Sagro (i.e., Fantiscritti, Ravaccione and Colonnata; Rodolico, 1953) were used. Recently some authors using the  $^{14}\text{C}$  method for the datation of paleosols hypothesize a pre-Roman extraction of marble localized in the Carbonera quarry (Bruschi *et al.*, 2003).

The geographer Strabone is the first ancient writer talking with a certain precision of the Apuane Alps and the «Lunensis» quarries. In the I century B.C. he describes the Luni harbour, the mountains behind the city and the marbles drawn out. In this period the excavation of the lunensis marble became a

real state industry, particularly favored by the sea transport of blocks to Rome. With the decadence of the empire the quarry activity decays, even if the extraction continued for some centuries and, perhaps never stopped completely (Dolci, 1980).

The date of resumption of the extraction of the Carrara marble is not known precisely (Franzini, 1992). However in the XII century sculptors in Pisa, Lucca and Pistoia Tuscan towns perform their works exclusively using Carrara marble. The works are of small dimensions, often not well preserved. The most ancient medieval monument entirely built with marble stones is represented by the Carrara Cathedral (XII century). A real extractive industry was restored, however, only at the beginning of 1400 A.D.. New Carrara quarried marbles are used in Pisa for the first time in the structures of the monumental Cemetery (XIII sec.) (Franzini, 1993). A moment of intense use of the Carrara marble is the Baroque age. In the XVII century the excavation in Carrara is well developed because of the great use of marbles both for architectural and decorative purposes. For this reason the quarrymen start also to re-utilize ancient sites, now deserted.

An important contribution to the knowledge of the marble typologies used in antiquity is the first treatise on historical marmology of the modern period «*De Antiquis Marmoribus*», published in 1783 by the Neapolitan antiquary and poet Biagio Garofalo. Winckelmann (1717-1768 A.D.) was, however, the first archaeologist to pick up finds coming from the different quarries and to compare them with the material in the monuments.

As regards the actual data related to the extraction, there are around 200 quarries in activity with over 6÷7,000,000 annual tons of extracted material for ornamental uses. The prevailing export areas, for the Apuan-Versilia district, are the North America with 26.7%, the European Union with 23.8% and the Middle East with 14.7% (IMM data of the 1<sup>st</sup> semester 1999).

The most extracted typology is the White Carrara Marble. This variety is normally also

denominated White Ordinary. Nevertheless it is necessary to consider that in trade exchanges marbles with different mineralogical-petrographical characteristics, but with the same aspect, are often commercialized with the same name. This occurs frequently in the case of the white marbles. From the scientific point of view the archaeometric problem of determining the provenance of the marbles, above all the white ones is of great importance and motivated by the necessity to determine the provenance of many sculptural finds (i.e. Lazzarini, 2004; Attanasio, 2003; Attanasio *et al.*, 2000; Meloni *et al.*, 1995; Herz and Waelkens, 1986). This research is frequently accompanied by the necessity to determine the causes of decay of some marbles in artifacts.

Monuments and sculptures in marble are indeed often interested by strong decay phenomena such as development of patinas, crusts, decohesion, and bending of the slabs. The more recurrent decay phenomenon affecting the material exposed in the external environment is represented by the granular disgregation, able to turn the superficial layer of the marble artifact into an incoherent heap of granules. This process develops through the disjunction of the calcite grains, mainly when the marble is submitted to cyclic temperature variations. Apuan marbles, above all the white varieties, show a strong variability in the decay phenomena, particularly regarding the intensity of the granular breakup, which is independent from the macroscopic characteristics that often appear completely similar. The principal causes of this behavior seem to be referred to the petrographical and physical characteristics which are the main differences among the white Apuan marbles (Barsottelli *et al.*, 1998, Cantisani *et al.*, 2000).

The studies on the textural and microstructural characteristic of the Apuan marbles are numerous and have shown a constant evolution during the time. A first attempt to explain these differences was realised by Crisci *et al.*, (1975), who performed a comparative examination between the mineralogical and chemical composition, and

the data related to the texture and structure of marbles coming from different zones. These authors conclude that the development and the shape of the calcite grains is strongly conditioned by the presence of other mineralogical phases. The association with phyllosilicates, dolomite and opaque minerals, including also organic matter, would cause, according to these authors, a decrease of the calcite grain size and it would condition the development of the whole structure.

Di Pisa *et al.*, (1985) evaluated the temperature of the metamorphic peak of Apuan rocks applying the calcite/dolomite geothermometer on marble samples suggesting that the granulometric variations in marbles are related to the metamorphic conditions realised. In particular they found that the increase of the crystal size is related to an increase of temperature of the metamorphic peak proceeding westwards.

Other researches (e.g., Covey-Crump, 1992, 1994, 1996; Schmid *et al.*, 1980), studying the deformation of calcite crystals both in natural and artificial conditions, obtained important information for the interpretation of the microstructures observed in the Apuan marbles. These studies were addressed, particularly, to the possibility of connecting the rheologic behaviour of calcite and the microfabric development to the conditions of crystallization and deformation of natural calcite.

In the past ten years results were reached in the study of the microfabric, which allowed to draw reliable models for the interpretation of the different microstructures present in the Apuan Metamorphic Complex. Our research will refer to the model proposed by Molli *et al.*, (2000), described in the following section.

The aim of this research is to characterise some white Apuan marbles and evaluate the influence of the microstructural parameters on the physical characteristics of the material and finally on its durability. The final purpose is to find out guidelines useful to define the criteria for the prevision of the durability of the material exposed to weathering in order to

determine the most suitable use of the different marbles. To this purpose predominantly white marbles with similar aesthetic characteristics coming from different geological structures or from different zones of the same structures have been considered and studied from the chemical- mineralogical, petrographical and physical point of view.

#### GEOLOGICAL SETTING

Several geological studies on Apuan marbles are nowadays available in scientific literature. They are considered the product of a middle grade dynamic thermic metamorphism of sedimentary carbonatic rocks formed in a shelf environment during Rhaetian-Liassic times. This type of sedimentation can be subdivided in two main phases: a Noric- Rhaetian phase corresponding to the hyperaline shelf of the Grezzoni formation, and a second Rhaetian-Hettangian, corresponding to the carbonatic shelf, which then produced the marbles. These two phases are separated by a sedimentation phase sets in the late Rhaetian, characterised by deep climatic mutations, representative of the passage from an environment of low depth to a notably more deep environment well individuated in the area of Orto di Donna-M.te Altissimo-M.te Corchia (Coli and Fazzuoli, 1992). Between late Rhaetian and Hettangian, the basis of the «marbles carbonatic shelf» develops covering the underlying breccias. Successively the sedimentation basin becomes deeper and differentiated. A fracture of the carbonatic shelf develops giving rise to isopic zones (Coli and Fazzuoli, 1992).

The Apuan marbles are involved in the main tectono-metamorphic events that interested the Massa and Apuane units. Starting from the Upper Oligocene, after the deposition of Pseudomacigno sandstones, two main polyphase tectono-metamorphic events can be recognised: the D1 event, dated with the K/Ar and the  $^{40}\text{Ar}/^{39}\text{Ar}$  methods at 27 My (Giglia and Radicati di Brozolo, 1970; Kligfield *et al.*, 1986) gave rise to kilometer-scale isoclinal

folds, antiformal stack development and shear zones formation, the second event D2, dated at 10-8 My deforms all earlier features, developing folds and shear zones (Carmignani *et al.*, 1980; Carmignani and Kligfield, 1990).

The interference between D1 and D2 events produces complex deformative structures, also inverting folds of kilometeric extension.

As regards the metamorphic degree, it is necessary to consider the presence, in the Apuan massif, of a metamorphic polyphasic and sintectonic mark with mineralogical associations typical of the chlorite-biotite zone of the green schists facies (Barberi and Giglia, 1965; Tucci, 1980). The occurrence of the stable association pyrophyllite+quartz (Franceschelli *et al.*, 1986, 1997) indicates temperatures of 300-400°C and pressures of 3-4 kbars. The application of the calcite-dolomite geothermometer allowed to obtain different temperatures in the various zones (Di Pisa *et al.*, 1985).

The marbles of the western zone display a wide range of  $\text{MgCO}_3$  that corresponds to temperatures among 420°C and 460°C, those of the eastern zone show temperatures among 310°C and 380°C, while those of the central zone introduce intermediary temperatures in comparison to the preceding ones. These variations point out an increase of the metamorphic degree inside the Apuan metamorphic complex proceeding from east toward west. The last studies related to the microstructural characteristics allow to individuate marbles produced according to processes of static recrystallisation, dynamic recrystallisation and rimobilisation during the last stadium of deformation (Molli *et al.*, 1997, Molli and Heilbronner, 1999, Molli *et al.*, 2000).

Marbles with equigranular polygonal microstructure (granoblastic or «foam» microstructure), and straight to slightly curved grain boundaries belong to the first group. Marbles with this microstructure were found in the Western part, Middle and Eastern zone of the Apuane, with a characteristic variation of the grain size from a zone to another (250-

300µm in the western side, 80-100µm in the eastern side). These marbles were produced during the thermal relaxation and heating realised after early D1 deformation.

Marbles with microstructure exhibiting strong shape preferred orientation, coarse grains with lobate/sutured boundaries, formed during the late stage of D1 event, are considered products of the dynamic recrystallization. Also microstructures formed during the D2 event with shape preferred orientation, polymodal grain size distribution, smaller grain size (the crystals that are the relicts of the preceding microstructure show a mean grain size between 150 and 200µm, while those formed during the recrystallisation have dimensions of 20-50µm) and rotation of the subgrains are referred to dynamic events. The D2 event is, in fact, associated to an exhumation process in condition of retrograde metamorphism. During this event in the highest Apuan Alps levels, shear zones, millimetric to decimetric thick, develop (Carrara area), whereas in the lowest levels (Arni area) only foldings occur. The dynamic microstructures are related to high strain and high temperature. Grain boundary migration and recrystallisation can be considered predominant in the first one (D1 event), rotation recrystallisation and grain boundaries migration in the second one (D2 event).

The microstructure characterised by the consistent presence of twinnings has been interpreted as produced at low temperatures in the late stadium of the exhumation of the metamorphic complex. This deformation is found, with different intensity, in all the marble levels (Molli *et al.*, 2000).

#### SAMPLING AND ANALYTICAL METHODS

Samples were taken from Apuan quarries sited in the Carrara, and Arni basins, and in the Renara, Passo del Vestito and Isola Santa zones (Fig. 1). Sampling was performed to obtain white marbles with different microstructural characteristic and to evaluate the existing

relationships among microstructural characteristic and petrophysical parameters. We selected samples with different microfabric, belonging to different structures and located in different positions of the same geological structure, representative of the variability inside the Apuan Metamorphic Complex. From each site blocks of decimetric size were extracted in order to obtain samples useful for all the characterization tests.

All the quarries belonging to the Carrara basin are located inside a great natural amphitheater, defined from west to east, by the southern slope of the Uccelliera Mountain (1.246m), the Borla Mountain (1.469m), the Sagro Mountain (1.749m), the Cima di Gioia (810m) and the Brugiana Mountain (960m).

This wide area develops for more than 2,000 hectares and is morphologically divided in three valleys separated one another by the slopes of the Maggiore Mountain (1.396m), namely the valleys of Miseglia, Torano and Colonnata. These valleys correspond to the main marble extractive basins (Bradley, 1997). Inside these valleys there are numerous quarries used since antiquity for the extraction of white marbles. The samples M.T.1, M.T.2, M.T.3, M.T.4, M.T.5, M.T.6, M.T.14 have been taken in different positions of the Carrara sincline.

The samples coming from Arni, Passo del Vestito and Isola Santa were selected for the particular metamorphic history they underwent during the geologic evolution of the Apuan Metamorphic Complex.

The localization of the quarries is reported in figure 1, the number of blocks taken from each quarry and the indication of the orientation of each block respect to the foliation, useful to cut oriented samples to be used in some tests, are reported in the Table 1.

Petrographic observations were performed using ultrathin sections (thickness between 5 and 2µm) in fact the high birifrangence of the calcite, which gives rise to interference colors fringes along the grain and twin boundaries in the sections 30 mm thick (Vernon, 1981). The use of ultrathin sections was very important

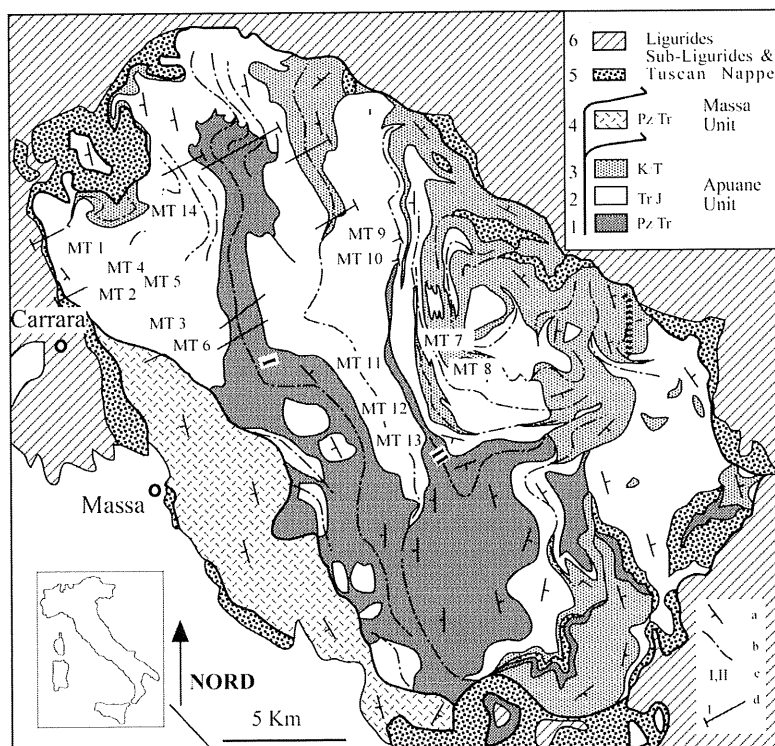


Fig. 1 – Localisation of samples in the geological map (modified after Carmignani and Kligfield) 1990.

also for the precise observation of the grain boundaries.

Two thin sections have been prepared for each sample, parallel (XY) and perpendicular (YZ) to the foliation plane, respectively.

In order to measure the mean grain size and its distribution, the Kaleidagraph method was used, whereas the grain shape orientation and the grain boundaries shape were quantified using the PAROR and SURFOR methods (Panozzo, 1983, 1984). Each grain boundary is digitised and consists of a short linear segment. The PAROR method defines the general orientation of a grain with respect to a reference line and it is based on the principle of the projection of the segments on a reference axis for different rotation angles. The total length of the B(a) projections depends upon the

dimensions and shape of the grain and the distribution of the orientations of the long axes. The preferential orientation of the grain boundaries was analysed with the SURFOR program, which uses projection diagrams of the A(a) value; it represents the cumulative orientation of the line segments of the projection of the grain boundaries with respect a reference line. Using the relationships  $A(a)_{\min}/A(a)_{\max}$  (SURFOR) and  $B(a)_{\min}/B(a)_{\max}$  (PAROR) it is possible to quantify the anisotropy of the analysed fabric. Therefore, the PAROR method allows to obtain the general orientation of the grains with respect to a reference axis, whereas the SURFOR method gives the orientation of the segments in which the grain boundary is decomposed. These data are represented

TABLE 1  
*Samples localization and sampling description.*

Name	Localization	Samples characteristics
<i>M.T. 1</i> (4 samples)	Ponte di Vara Fantiscritti basin	Samples oriented according to foliation
<i>M.T. 2</i> (3 samples)	Fantiscritti basin	Samples oriented according to foliation
<i>M.T. 3</i> (6 samples)	Canalgrande quarry Miseglia basin	Samples oriented according to foliation
<i>M.T. 4</i> (5 samples)	Fantiscritti basin	Not oriented
<i>M.T. 5</i> (8 samples)	Ortensia quarry, SE slope M.te Maggiore, Colonnata	Samples oriented according to foliation
<i>M.T. 6</i> (3 samples)	Gioia quarry Colonnata	Not oriented
<i>M.T. 7</i> (3 samples)	Arni	Samples oriented according to foliation
<i>M.T. 8</i> (4 samples)	Landi quarry Arni	Samples oriented according to foliation
<i>M.T. 9</i> (6 samples)	Renara	Samples oriented according to foliation
<i>M.T. 10</i> (4 samples)	Passo del Vestito	Samples oriented according to foliation
<i>M.T. 11</i> (5 samples)	Locanda La Romana	Samples oriented according to foliation
<i>M.T. 12</i> (3 samples)	Ex quarry Henraux	Samples oriented according to foliation
<i>M.T. 13</i> (4 samples)	Isola Santa	Samples oriented according to foliation
<i>M.T. 14</i> (3 samples)	Fossalunga quarry Fantiscritti basin	Samples oriented according to foliation

through the construction of Polar-Diagrams (Rose- Diagram) (Figg. 2a and b).

The convolution of the grain boundaries was determined using the SHAPES program (Panozzo and Hurlimann, 1983), which evaluates the Paris factor, able to synthesise the relationship concavity/convexity of the grain boundaries geometry (Fig. 3). A low PARIS factor points out straight grain boundaries and

its increase identifies a grain boundaries migration .

Major and minor elements composition was performed by XRF using an automatic Philips PW 1480 spectrometer and a correction for the matrix effect was realized according to the methods described by Franzini *et al.* (1975).

A gasometric method was used to determine CaCO<sub>3</sub> contents, measuring the relative content

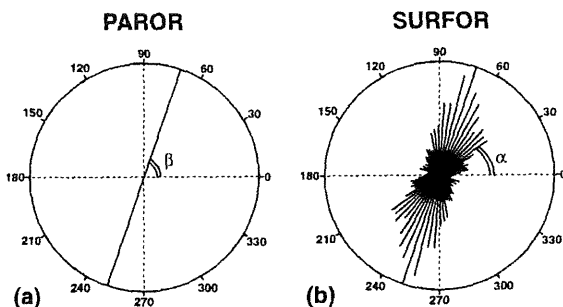


Fig. 2a,b – Results obtained by application of PAROR method (a) and SURFOR method (b) to a single grain.

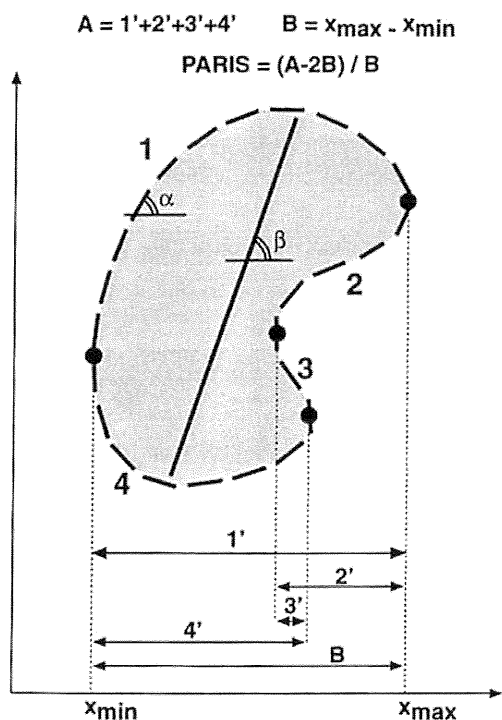


Fig. 3 – Projections utilised for the evaluation of Paris factor.

in CO<sub>2</sub>. This method can be utilised when the dolomite is absent or present in very low amount. The tests were realised using the methodology described by Leone *et al.* (1988). The measure related to the content of CaCO<sub>3</sub> in the sample, expressed as percentage in weight,

is taken making reference to a pure carbonatic standard used to built the calibration line and to turn the values of PCO<sub>2</sub> in the real contents in CaCO<sub>3</sub>.

Besides, the following petrophysical characteristics were measured: total open porosity P (determined through mercury intrusion porosimetry techniques and calculated as  $(V_a - V_r) / V_a * 100$ ; where  $V_a$  is the apparent volume of the test-samples measured with a mercury pycnometer, while  $V_r$  is the real volume, measured with a helium pycnometer); mesoporosity ( $0.0037\mu\text{m} < r < 150\mu\text{m}$ , determined with a mercury porosimeter); water imbibition coefficient [in weight (I.C.wt.%) and volume (I.C.vol.%)] obtained by absorption through total immersion and through capillarity; water saturation index (S.I.) which is the ratio I.C. vol P %. It indicates the amount of porosity occupied by water.

The curves of water absorption through total immersion were obtained following the methodology described in the NORMAL Document 7/81 and were performed on test-samples of 2x1.8x1.8 cm dimension, whereas the capillary tests were performed on test-samples of 5x5 x2 cm dimension, modifying the times indicated in the NORMAL Document 11/85 in order to allow a better definition of the quantity of water absorbed by the samples during the first minutes.

For a better definition of the process of water absorption through total immersion a Mettler



TABLE 2  
Major elements composition (wt. %) obtained with XRF.  
(bdl=below detection limit)

	SiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	MnO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	LOI
M.T.1	1.61	0.08	0.66	0.28	0.10	0.04	54.1	0.02	0.21	bdl	42.9
M.T. 2	0.40	0.06	0.12	0.06	bdl	0.06	55.8	0.02	0.02	bdl	43.7
M.T. 3	1.00	0.13	0.32	0.16	bdl	0.16	55.2	0.02	0.06	bdl	43.3
A.M 4	0.41	0.04	0.12	0.07	bdl	0.06	55.6	0.02	0.02	bdl	43.8
M.T. 5	0.51	0.06	0.19	0.07	bdl	0.07	55.6	0.01	0.05	bdl	43.5
M.T. 6	1.23	0.17	0.37	0.19	0.24	0.21	54.6	0.12	0.07	bdl	42.9
M.T. 7	0.45	0.06	0.17	0.08	bdl	0.08	56.0	0.01	0.03	bdl	43.1
M.T. 8	1.73	0.20	0.61	0.39	bdl	0.32	54.6	bdl	0.10	bdl	42.9
M.T. 9	1.19	0.16	0.35	0.20	1.55	0.25	53.9	0.06	0.06	bdl	42.3
M.T. 10	0.90	0.15	0.28	0.15	0.45	0.17	55.0	0.21	0.05	bdl	43.0
M.T.11	2.51	0.36	0.80	0.39	bdl	0.46	54.5	0.11	0.15	0.05	42.8
M.T. 12	2.04	0.26	0.76	0.28	bdl	0.31	54.3	0.15	0.14	bdl	42.7
M.T. 13	1.53	0.10	0.85	0.28	bdl	0.08	54.1	0.02	0.02	bdl	42.9
M.T. 14	2.07	0.30	0.65	0.30	bdl	0.39	54.0	0.08	0.11	bdl	42.4

Toledo hydrostatic balance was used. This balance, generally utilised for the determination of the density of solid bodies, was used to record continuously the weight increase of marble samples dipped in distilled water.

## RESULTS

In table 2 the major element analyses of samples obtained by XRF are reported.

The results of the application of the gasometric method for the valuation of the CaCO<sub>3</sub> content are reported in Table 3. It is evident that the examined marbles can be defined as very pure calcite marbles (%CaCO<sub>3</sub>>95 wt.%)

With regard to the data related to the petrographic characterization a summarizing table is introduced (Table 4). For each site the dominant microstructure, the recrystallisation type, the presence of orientation inferred by the

TABLE 3  
CO<sub>2</sub> values obtained through gasometric method  
and CaCO<sub>3</sub> calculated as weight per cent.

	% CO <sub>2</sub>	% CaCO <sub>3</sub>
M.T.1	41.9	95.3
M.T. 2	44.2	100.0
M.T. 3	43.5	99.0
M.T. 4	42.6	97
M.T. 5	44.1	100.0
M.T. 6	43.3	98.5
M.T. 7	43.9	99.9
M.T. 8	48.4	100.0
M.T. 9	41.5	94.4
M.T. 10	44.8	100.0
M.T. 11	43.5	98.9
M.T. 12	44.3	100.0
M.T. 13	43.3	98.6
M.T. 14	44.3	100.0

TABLE 4  
Main microstructural parameters  
MGS = mean grain size

Dominant microfabric	Localization	Recrystallization type	MGS	Shape orientation	Paris %
M.T. 1 Granoblastic slightly oriented	Ponti di Vara	Static, almost complete recover	200 $\mu$ m	Present, small angle with the foliation	5-6
M.T. 2 Granoblastic, not oriented	Fantiscritti Basin	Static, complete recovery	235 $\mu$ m	Absent	3-4
M.T. 3 Granoblastic/eteroblastic*	Canalgrande Quarry, Miseglia Basin	Static	130 $\mu$ m	Present in the small grain sizes <100 mm	5-6
M.T. 4 Granoblastic	Fantiscritti Basin	Static	150 $\mu$ m	Absent	4-5
M.T. 5 Xenoblastic	Ortensia Quarry, Colonnata Basin	Dynamic, syn D <sub>2</sub> , grain boundaries migration and sub grain sub division	90 $\mu$ m	Present in the small grain size	8-9
M.T. 6 Granoblastic /xenoblastic*	Gioia Quarry, Colonnata Basin	Static	140 $\mu$ m	Absent	5-6
M.T. 7 Xenoblastic	Arni	Dynamic, syn D1, post thermal peak grain boundaries migration	180 $\mu$ m	Present, defines the main foliation	11-12
M.T. 8 Xenoblastic	Landi Quarry, Arni	Dynamic, syn D1, post thermal peak grain boundaries migration	90 $\mu$ m	Present in small grain size	8-9
M.T. 9 Granoblastic	Renara	Static	100 $\mu$ m	Absent	3-4
M.T. 10 Granoblastic/xenoblastic*	Passo del Vestito	Static	150 $\mu$ m	Absent	4-5
M.T. 11 Granoblastic	Locanda La Romana	Static	170 $\mu$ m	Present	5-6
M.T. 12 Granoblastic/xenoblastic*	Ex Quarry Henraux	Static	90 $\mu$ m	Present	8-9
M.T. 13 Granoblastic	Isola Santa	Static	150 $\mu$ m	Absent	2-3
M.T. 14 Xenoblastic	Fossalunga Quarry, Fantiscritti Basin	Static	200 $\mu$ m	Present	8-9

\*the prevalent microstructure is granoblastic but tardive mobilitation of grain boundaries determines a microstructure with intermedie characteristics

study of the Rose-Diagrams obtained from the values related to the PAROR and SURFOR parameters, the mean grain size and the PARIS factor are reported. The Fig. 4 shows the histograms of distribution of the granulometric classes.

The mean grain size, for the different kind of marbles ranges between 90µm (minimum mean grain size) and 235µm (maximum mean grain size), with PARIS factor varying from 3 (straight grain boundaries) to 12 (sutured grain boundaries). As regards the orientation, marbles without orientation and marbles characterised by strong shape orientation of calcitic grains were observed (Fig. 5a, b)

The Table 5 shows for each quarry zone, the mean values and the standard deviations (±) of the values of total open porosity, imbibition

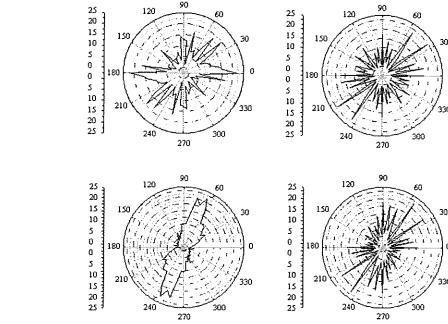


Fig. 5a,b – Rose diagrams obtained with the PAROR and SURFOR methods for an unoriented marble (a) and an oriented marble (b).

coefficient (wt.%, vol.%) and saturation index determined by absorption of water through total immersion.

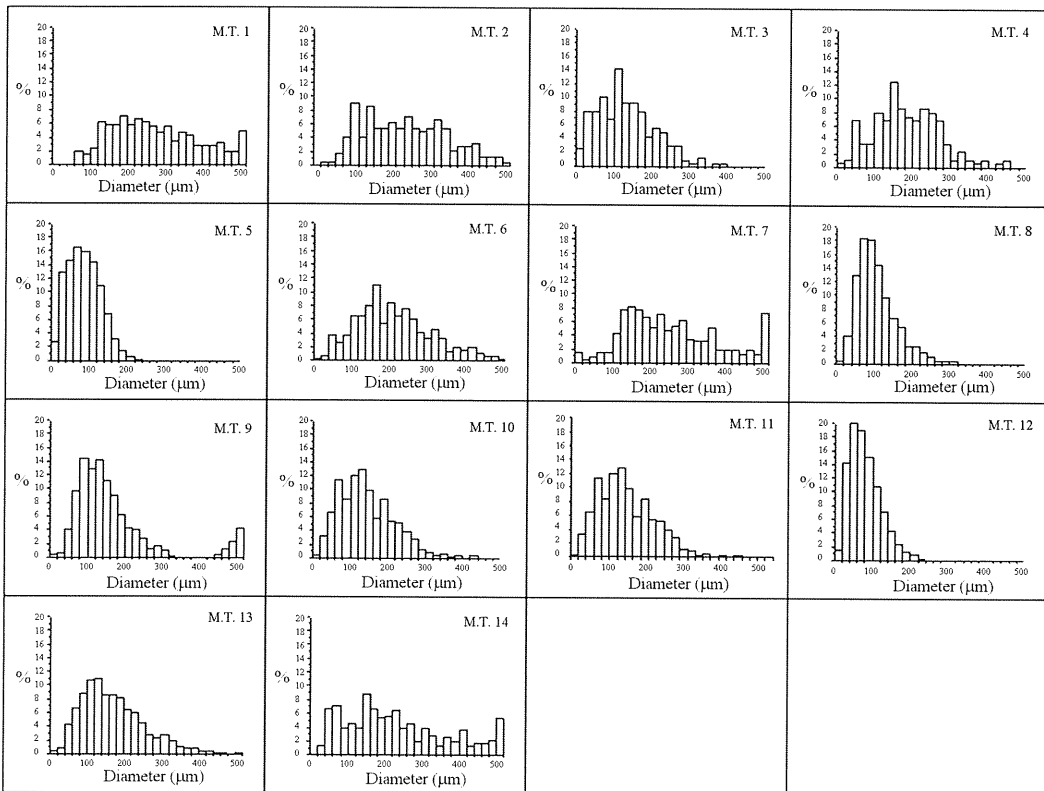


Fig. 4 – Grain size distribution obtained through image analysis.

TABLE 5

Physical parameters  $P$  = total open porosity (%);  $IC_{wt}$  = imbibition coefficient (in weight);  
 $IC_v$  = imbibition coefficient (in volume);  $SI$  = saturation index (%).

	P	$IC_w$	$IC_v$	SI
M.T.1	1.2±0.3	0.19±0.05	0.51±0.07	43±10
M.T. 2	1.7±0.2	0.37±0.04	0.99±0.04	59±6
M.T. 3	1.3±0.2	0.20±0.02	0.54±0.06	30±10
M.T. 4	1.5±0.3	0.27±0.03	0.72±0.03	45±7
M.T. 5	1.2±0.3	0.16±0.02	0.43±0.03	40±9
M.T. 6	1.3±0.2	0.23±0.01	0.62±0.03	50±10
M.T. 7	0.9±0.3	0.16±0.04	0.43±0.03	48±11
M.T. 8	1.2±0.4	0.23±0.07	0.62±0.03	52±7
M.T. 9	1.6±0.2	0.33±0.01	0.89±0.03	97±30
M.T. 10	1.1±0.2	0.38±0.07	1.02±0.04	91±6
M.T. 11	1.9±0.4	0.62±0.06	1.66±0.09	87±5
M.T. 12	0.7±0.2	0.21±0.05	0.57±0.04	82±20
M.T. 13	2.5±0.3	0.85±0.01	2.26±0.03	104±18
M.T. 14	0.7±0.2	0.16±0.03	0.43±0.07	61±19

With the aim to emphasise the differences among the marble samples, the porosity was divided in the following dimensional ranges: mesoporosity ( $0.0037 \mu\text{m} < r < 150\mu\text{m}$ ) measured with the mercury porosimeter; microporosity ( $r < 0.0037\mu\text{m}$ ) calculated as the difference between total open porosity and mesoporosity given the absence of macropores ( $r > 150\mu\text{m}$ ).

Table 6 shows the relationships between mesoporosity and microporosity. There are marbles where the mesoporosity prevails (e.g. M.T. 10, M.T. 11, M.T. 13) and marbles with a higher amount of micropores (M.T. 5, M.T. 1, M.T. 14). The figures 6a, b and 7 show the curves of water absorption obtained through total immersion, hydrostatic balance (time of absorption equal to 10 minutes) and through capillarity.

The figures 8a, b synthetize the main petrophysical characteristics for each marble typologies.

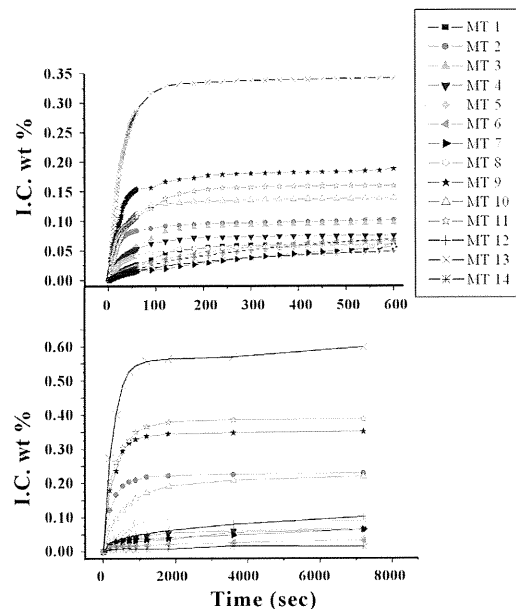


Fig. 6a,b – a) Water absorption trends obtained by hydrostatic balance (10 minutes) (I.C. wt%= imbibition coefficient in weight); b) Water absorption through total immersion.

TABLE 6  
Total open porosity decomposition

	Total open porosity	Meso-porosity	Micro-porosity	% mesoporosity	% microporosity
M.T.1	1.2	0.38	0.82	32	68
M.T. 2	1.7	0.75	0.95	44	56
M.T. 3	1.3	0.49	0.81	38	62
M.T. 4	1.5	0.65	0.85	43	57
M.T. 5	1.2	0.36	0.84	30	70
M.T. 6	1.3	0.49	0.81	38	62
M.T. 7	0.9	0.35	0.55	39	61
M.T. 8	1.2	0.53	0.67	44	56
M.T. 9	1.6	0.75	0.85	47	53
M.T. 10	1.1	0.80	0.20	73	27
M.T. 11	1.9	1.60	0.30	84	16
M.T. 12	0.7	0.41	0.29	59	41
M.T. 13	2.5	2.12	0.40	85	15
M.T. 14	0.7	0.26	0.44	37	63

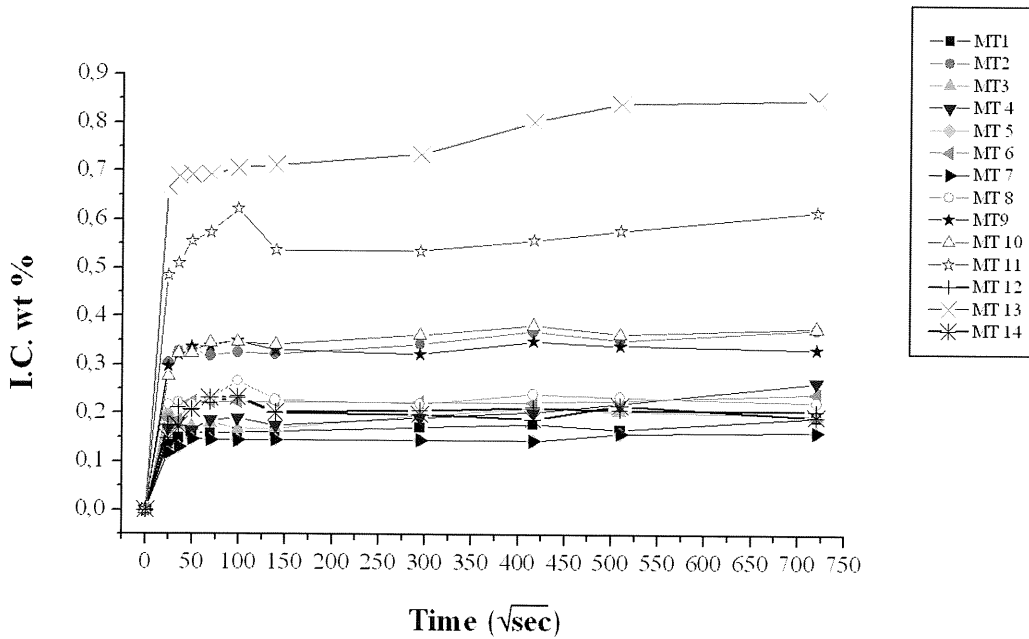


Fig. 7 – Water absorption trends obtained through capillarity tests.

	Mesoporosity distribution*	Absorption through total immersion (balance 10 minutes)	Absorption through capillarity
M.T. 1 P % = $1.2 \pm 0.3$ M % = $0.38 \pm 0.05$			
M.T. 2 P % = $1.7 \pm 0.2$ M % = $0.75 \pm 0.03$			
M.T. 3 P % = $1.3 \pm 0.2$ M % = $0.49 \pm 0.04$			
M.T. 4 P % = $1.5 \pm 0.3$ M % = $0.65 \pm 0.05$			
M.T. 5 P % = $1.2 \pm 0.3$ M % = $0.36 \pm 0.06$			
M.T. 6 P % = $1.3 \pm 0.2$ M % = $0.49 \pm 0.05$			
M.T. 7 P % = $0.9 \pm 0.3$ M % = $0.35 \pm 0.06$			
M.T. 8 P % = $1.2 \pm 0.4$ M % = $0.53 \pm 0.02$			

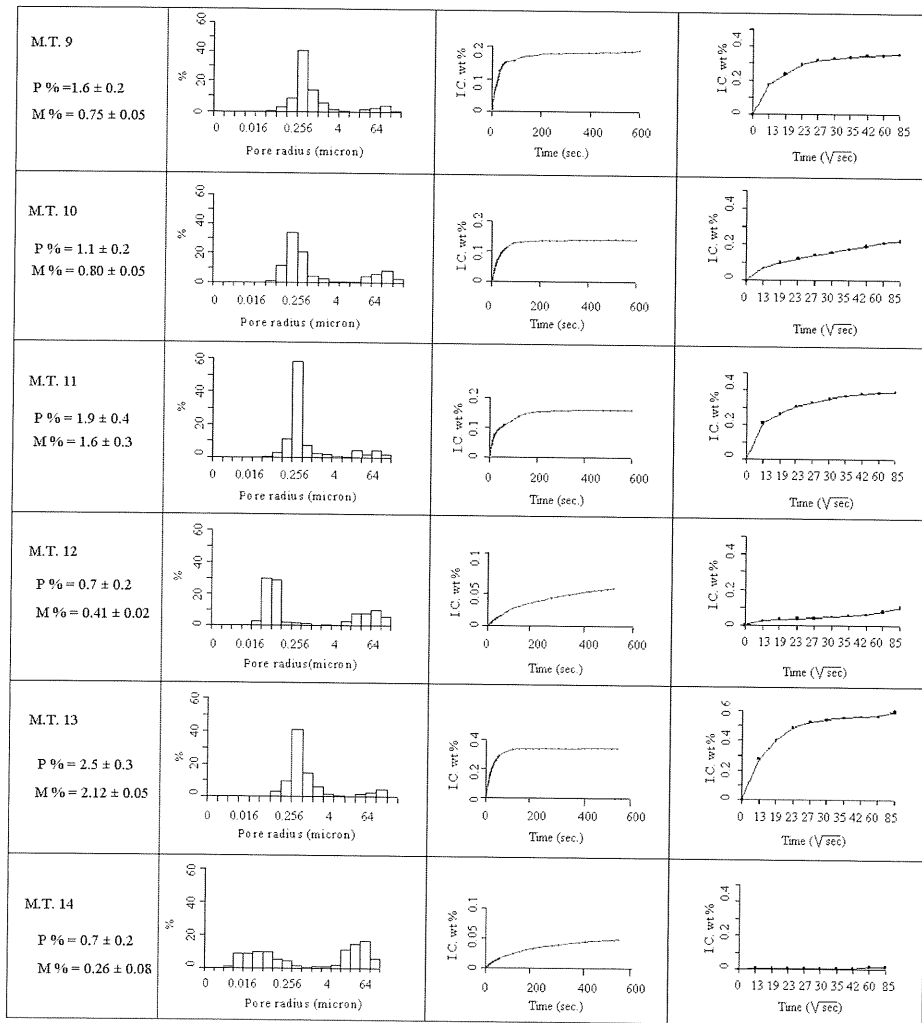


Fig. 8a, 8b – The main petrophysical characteristics of examined marbles (P%= total open porosity, M%= mesoporosity)

## DISCUSSION

From a compositional point of view the studied white marble are quite homogeneous with only small quantities of  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{MgO}$ ,  $\text{Fe}_2\text{O}_3$  (<5%) (cfr. Table 2), which are connected to the presence of silicatic minerals (quartz, feldspars, muscovite). Despite the presence of such impurities a distinction of the marbles on a compositional basis is not possible except for the Renara marble, which shows traces of dolomite.

The good agreement (within the error of measure) between the data related to the loss of volatile through calcination (LOI) and the values of  $\text{CO}_2$  obtained through the calcimetric test allows to exclude the significant presence of organic substance (cfr. Table 3).

Significant differences have been observed in the microfabric characteristics. The microstructures underline the passage from marbles representative of complete recrystallisation in the annealing phase, with absence of orientation, as shown by some samples coming from the basin of Fantiscritti, Renara and Isola Santa (typologies M.T. 2, M.T. 9, M.T. 13) to marbles with microstructures representative of dynamic recrystallisation, sin D2, with evidences of migration of the grain boundaries and incipient subgrains subdivision (Colonnata samples, typology M.T.5) up to marbles representative of dynamic recrystallisation, sin D1, with remobilisation of the grain boundaries post thermal peak (Arni samples, typology M.T. 7).

Differences have been found not only among different extractive basins (i.e., basin of Arni and basin of Carrara) (Fig. 9a), but also inside the same extractive basin. In the Fantiscritti basin, for instance, there are samples with granoblastic microfabric, with grain shape orientation (Fig. 9b) and samples with grain boundaries from lobate to sutured (Fig. 9c), which let us suppose to the existence of samples with intermedie characteristics between the two microstructures. This case is clear comparing some samples of Fantiscritti

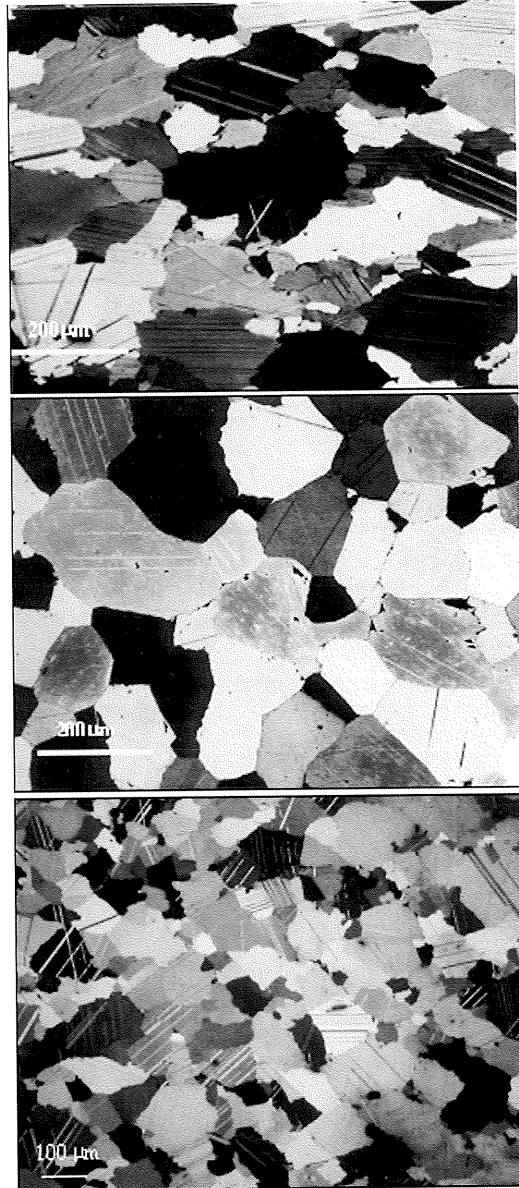


Fig. 9 – Ultrathin sections of Arni marble (M.T. 7 typology) (a), Fantiscritti marble (M.T. 2 typology) (b), Fantiscritti marble (M.T. 13 typology) (c)

(i.e. typology M.T. 2) and the samples of the Fossalunga quarry (typology M.T. 14).



The value of total open porosity does not allow to characterise universally the different typologies of marble, but only a generally distinction is possible. Moreover data of table 6 show that there are samples where the mesoporosity prevails (e.g., M.T. 10; M.T. 11; M.T. 13) and samples characterised mostly by the microporosity (e.g., M.T. 1; M.T. 5; M.T. 6; M.T. 7). The curves related to the water absorption tests through total immersion (cfr. figure 6) show that this characteristic strongly influences the water absorption dynamics. In the samples characterised by a value of the mesoporosity that almost represents the whole of the total open porosity, the imbibition coefficient in weight and the saturation index are elevated. The samples coming from Isola Santa (M.T. 13), where all the pores fall within the dimensions of mesopores, show the maximum value of water absorption through total immersion. On the other hand, reduced values of water absorption through total immersion have been found in the marbles coming from Arni (M.T.7), Colonnata (M.T.5 and M.T. 6) and in some lithotypes of

Fantascritti (M.T.1; M.T. 14) characterized by a low ratio mesoporosity /total open porosity. (cfr Figg. 8a, b).

These data were confirmed by the curves obtained with the hydrostatic balance.

Another parameter that plays an important role in the dynamic of water absorption is the pore size distribution. We can observe that the samples with a higher capability of water absorption are mainly characterised by an unimodal pore size distribution with a peak between 0.25 and 0.5 µm (figure 10a M.T. 11). These samples are also characterised by a great speed of absorption. This type of pore size distribution can be found especially in marbles with straight grain boundaries. Marbles characterised by a low capability of water absorption are mainly characterised by a bimodal pore size distribution (figure 10b M.T. 5).

Meaningful data through the study of the processes of water absorption have been obtained by capillarity tests. In first place the amounts of absorbed water are fairly different from a type of marble to another one. Other interesting results come from the capillarity

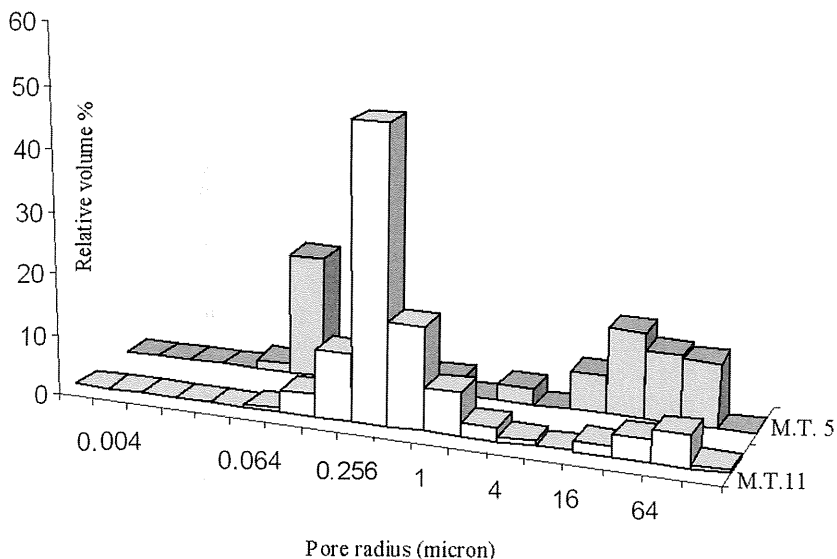


Fig. 10a,b – Pore size distribution of Locanda La Romana and Colonnata samples (M.T. 11 and M.T. 5 typology).

absorption tests performed on samples oriented respect to the foliation, which on its turn identify a possible grain shape orientation. (Fig. 11).

As expected, a greater rate of absorption in the directions parallel to the foliation is evident. In the case of samples without grain shape orientation [i.e., samples coming from Renara (M.T. 9) and from the Locanda La Romana (M.T. 11)] an increase of the rate of absorption has been evidenced in the directions perpendicular to the foliation, but that seems to be caused by the formation of evident macro cracking along the plans parallel to the foliation. In the case of the samples coming from Locanda La Romana, a macro cracking of the blocks during extraction from the front of the quarry probably due to the release of residual tectonic tensions, has also been recorded. Comparing the microstructural parameters with the physical characteristics we can suggest that the total open porosity is conditioned by the type of grain boundaries and by the grain size distribution: coarse grained marbles with straight grain boundaries show more elevated values of total open porosity. The total open porosity is maximum in samples with alternation of layers with different granulometric characteristic, it is minimum in smaller grained marbles with sutured grain boundaries.

The graph obtained reporting the total open porosity, mesoporosity and the mean grain size (Fig. 12a, b) does not reveal the existence of any clear trend. A possible relationship is, instead, evidenced between the Paris factor and the total porosity (Fig. 13a). The relationship becomes more evident in the graph of figure 13b where the mesoporosity is plotted against the Paris factor.

Figure 13c shows the relationship between the Paris factor and the weight imbibition coefficient obtained through total imbibition.

From these diagrams it is possible to infer that at a given degree of convolution of the grains (Paris factor) the dynamics of the water absorption is strongly controlled by the grain size distribution rather than by the mean grain size. This is highlighted when comparison between samples with the same mean grain size

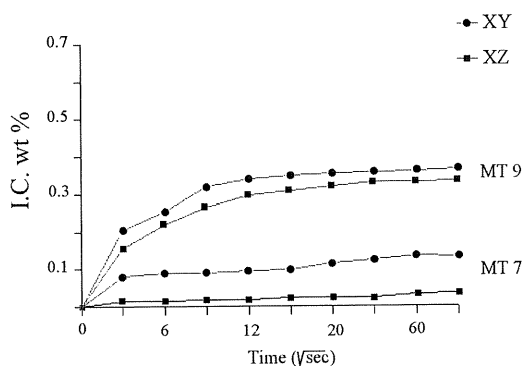


Fig. 11 – Capillarity water absorption for the Arni samples characterized by strong shape orientation (M.T. 7 typology) and for the Renara samples without shape orientation (M.T. 9 typology).

and different grain size distribution is made, alternatively. Therefore, given a same Paris factor, the presence of granulometric variations, distributed in layers, is the characteristic that potentially favors the water absorption.

#### SUMMARY AND CONCLUSION

Marble samples with different microstructure from different zones of the Apuan Alps and characterised by different tectono-metamorphic evolution were analyzed. This study highlighted the following aspects of the samples studied:

- the examined marbles show a notably homogeneous chemical-mineralogical composition: from the chemical point of view they are constituted by high content of  $\text{CaO}$ , smaller amounts of  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{MgO}$ ,  $\text{Fe}_2\text{O}_3$ . From the mineralogical point of view the analysed samples are calcite marbles; only some samples have shown the presence of traces of albite, quartz, micas and dolomite;
- the observed differences regard the microstructural characteristics referred to the different phases of the tectono-metamorphic events that have interested the Metamorphic Apuan Complex. The analysed samples show both no shape orientation, with straight to

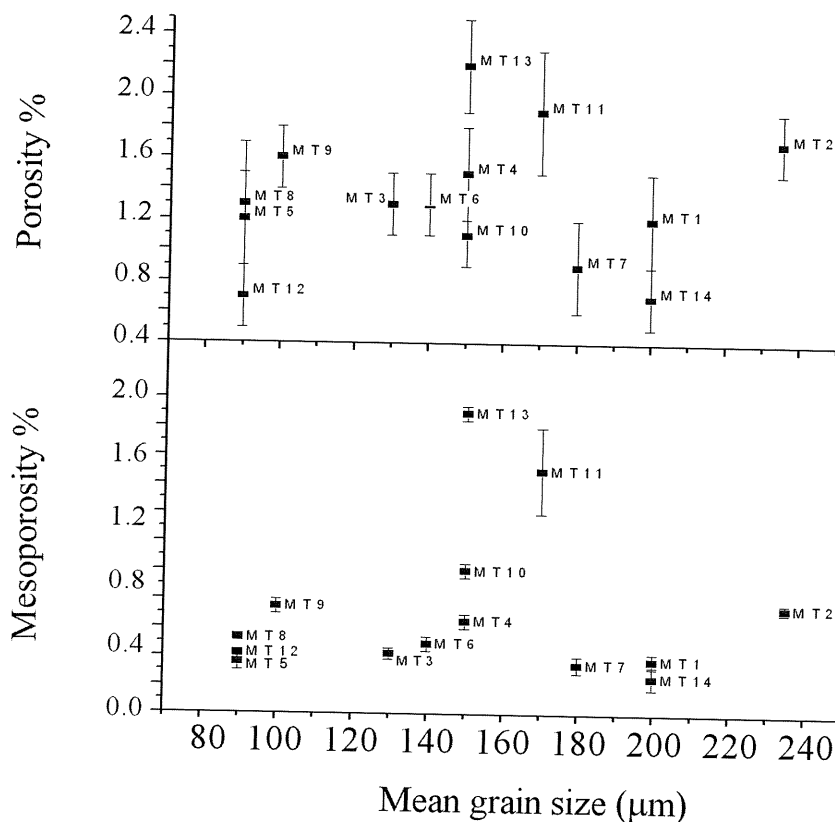


Fig. 12 – Relationship between total open porosity % and mean grain size (a) and relationship between mesoporosity % and mean grain size (b).

slightly lobates grain boundaries and marbles characterised by strong shape orientation and grain boundaries from interlobates to strongly sutured. The first typology can be found in the Western, Middle and Eastern zone of the Apuanes with a characteristic variation of the grain size from a zone to the other (250-300 µm in the western zones, 80-100 µm in the eastern zones). The second typology is attributed to processes of dynamic recrystallisation and it is represented both from marbles with relatively coarse grain size, sutured grain boundaries and strongly oriented shape, and from marbles with polymodal grain size distribution (the relict crystals of the previous microstructure have grain size between 150 and 200 µm, whereas

those new formed, due to the recrystallisation, have dimensions of 20-50 µm). The samples of the first typology come from Fantiscritti, Miseglia, Renara, Isola Santa, while examples of the different phenomena of dynamic recrystallisation can be considered the lithotypes of Colonnata and Arni;

– the Carrara basin has shown an evident variability: from terms characterised by inclusive mean grain size between 150 and 250 µm, without shape orientation or with weak orientation, Paris factor inclusive between 3 and 6 (Ponti di Vara in Fantiscritti; Canalgrande quarry in Miseglia) up to terms with grain size between 90 and 180 µm, strong shape orientation and values of the Paris factor

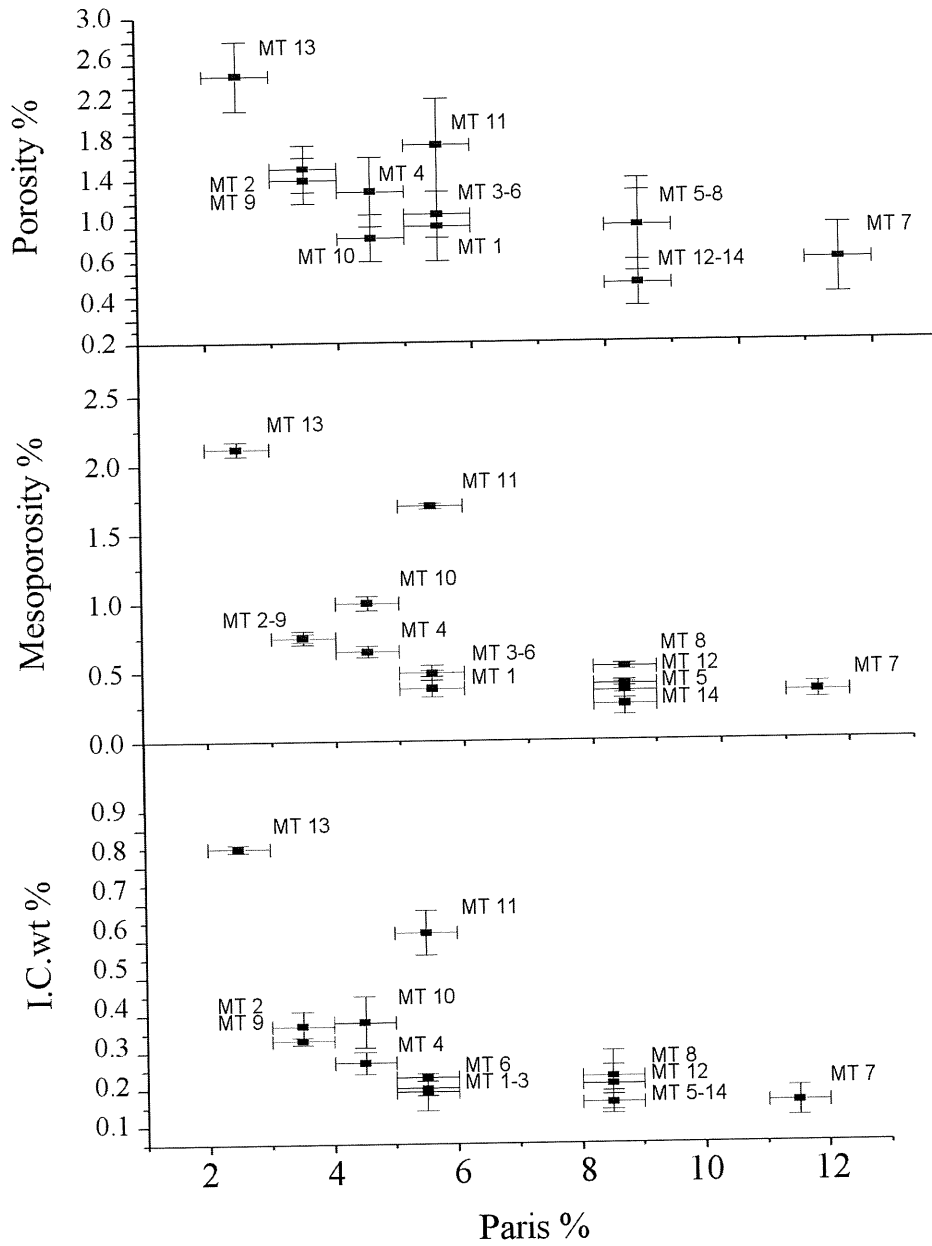


Fig. 13 – Relationship between Paris % and total open porosity % (a), relationship between Paris % and mesoporosity % (b), relationship between Paris % and I.C. wt % (c).

between 8 and 9 (Ortensia quarry in Colonnata, Fossalunga quarry in Fantiscritti).

Regarding the relationship between microstructural parameters and physical characteristics, the following conclusions can be drawn:

- marbles with xenoblastic microstructure given by predominantly sutured grain boundaries and strong shape orientation show low values of total open porosity while sensitively more elevated values have been evidenced in samples with typical polygonal granoblastic microstructure, without shape orientation;

- marbles in which the total open porosity falls within the range of mesoporosity show high values of the water imbibition coefficient and saturation index (i.e., Isola Santa samples);

- marbles characterised by prevailing micropores show low values of water imbibition coefficient and saturation indexes (i.e., samples from Arni, Colonnata and some samples from Fantiscritti);

- marbles characterised by grain shape preferred orientation show a higher water capillarity absorption in the direction parallel to the foliation;

- marbles with unimodal pore size distribution and a peak between 0.25 and 0.5  $\mu\text{m}$ , particularly evident in marbles with straight grain boundaries, are characterised by a high and quick water absorption;

- the presence of granulometric variation distributed in layers favors the water absorption.

In conclusion the grain size distribution, the presence/absence of orientation and the shape of grain boundaries affect the petrophysical characteristics.

When planning a restoration project or a new building with marble claddings or decoration, a systematic study of the microstructural characteristics of the marble material should be performed.

A further evolution of this work will be related to the artificial weathering of the examined marbles in order to evaluate the variation of the petrophysical characteristic of marbles.

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