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Relationships between microstructures and physical properties of white Apuan marbles: inferences on weathering durability

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ABSTRACT. — The different types of Apuan white marbles show varying degrees of durability when exposed to external weathering. Their durability has been related to different microstructures, linked to distinct tectonometamorphic histories within the Apuan Metamorphic Complex. This paper examines samples collected from several sectors of the Apuan Alps, the differing geological histories of which cause different marble microstructures. The samples are dominated by either granoblastic or xenoblastic microstructures. Chemical, mineralogical, petrographical and physical analyses were carried out. Microstructural characteristics were analysed by image analysis in order to quantify parameters such as grain diameter, diameter of equivalent circle, axial ratio, and roundness. These data are related to petrophysical parameters such as porosity, mesopore distribution, and saturation index. All the collected data indicate that the Apuan white marbles with low roundness (indicating granoblastic structure) are more susceptible to weathering than those with xenoblastic structure.

Furthermore, the different microstructural characteristics can be used to study the provenance of the white marbles used in the past in constructing and decorating historical buildings and monuments.

RIASSUNTO. — I marmi bianchi apuani mostrano una differente durabilità quando esposti all'esterno. La loro durabilità appare collegata alle differenti microstrutture dovute alla diversa evoluzione tettono-metamorfica cui è stato sottoposto il Complesso Metamorfico Apuano. In questo articolo sono stati studiati marmi provenienti da alcuni settori delle Alpi Apuane interessati da una diversa storia geologica e quindi con diverse microstrutture. Le principali microstrutture osservate sono quelle granoblastiche e xenoblastiche. Su questi diversi tipi di marmo sono stati determinati dati chimici, mineralogici, petrografici e fisici. Le caratteristiche microstrutturali sono state analizzate attraverso l'analisi di immagine e sono state definite mediante l'individuazione di parametri quantitativi quali il diametro granulare, il diametro del cerchio equivalente, il rapporto assiale e l'arrotondamento. I dati ottenuti sono stati correlati con i parametri petrofisici quali la porosità, la distribuzione dei mesopori, l'indice di saturazione. Tutti i dati raccolti indicano che i marmi apuani con basso valore di arrotondamento, indicativi di struttura granoblastica, sono molto più suscettibili all'alterazione di quelli xenoblastici. Le differenze microstrutturali possono essere, inoltre, utilizzate per l'identificazione dei marmi bianchi usati in passato in architettura e nei monumenti.

KEY WORDS: *Apuan marbles, microstructure, durability*

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INTRODUCTION

Apuan marbles have considerable importance in the cultural heritage of Italy and are architecturally valued all over the world, both in architecture as industrial stone and in artistic monuments. These marbles generally have a homogeneous aspect and their identification is in many cases problematic especially when they are already in place in monuments and buildings. Despite their aspect, the durability of white Apuan marbles when exposed to weathering show great variability. It is well known that some marbles from various sectors of the Apuan Alps have relatively low durability in the open environment. The characterisation and identification of the provenance of Apuan white marbles is therefore of crucial importance for commercial purposes, determination of authenticity or when material for restorations is needed. The architectural buildings and artistic monuments which highlight the problems related to the use of different types of Apuan marbles are numerous, both in ancient architecture (e.g., Florence Cathedral, where the marbles covering the facade, built in second half of the 19th century, are badly affected by weathering, whereas those covering the sides, emplaced in the 14th century are well preserved (Fratini *et al.*, 1987)) like that of modern buildings (Finlandia House in Helsinki, Amoco Tower in Chicago, Arc de la Défence in Paris).

Current methods for the petrophysical characterisation of marbles are based on the observation of thin sections under a polarising microscope or on determination of physical properties such as density, porosity, imbibition coefficients, and saturation index.

In a recent paper, Barsottelli *et al.* (1998) studied the petrophysical characteristics of two types of marble from the Apuan Alps, and concluded that tectono-metamorphic evolution exerted primary control on the different macrostructural and microstructural characteristics of the two types. These authors recognised that the physical properties of marbles are closely linked to their microstructural features.

This paper describes a systematic petrophysical study on samples from Apuan sectors which underwent separate geological histories and therefore have different microstructural characters.

The main purposes of this paper are:

– to correlate microstructures, quantitatively defined, with other physical properties such as density, porosity, imbibition coefficients, and saturation index, for information on the durability of the various types of white Apuan marbles;

– to define quantitatively the microstructural features of the marbles by image analyses;

– to define the microstructures and other physical characteristics of the various types of white marbles for more data useful in identifying the marbles used in the past for constructing and decorating historical buildings and monuments.

GEOLOGICAL AND PETROGRAPHIC DESCRIPTION OF APUAN MARBLES

Most Apuan marbles represent the deposits of a carbonate shelf which formed during Rhaetian-Liassic times. The sedimentation process may be subdivided into two main phases: a Noric-Rhaetian phase, corresponding to the hyperhaline shelf of the Grezzoni formation; and a second Rhaetian-Hettangian phase, producing a carbonate shelf which locally onlaps breccia deposits (Coli and Fazzuoli, 1992). The carbonate shelf was transformed into marbles during a regional metamorphic phase in the Oligo-Miocene. The corresponding unmetamorphosed carbonate succession is found in the Tuscany sedimentary series called «Calcare massiccio» (Carmignani *et al.*, 1993). Apuan marbles have long been petrographically and mineralogically studied (Zaccagna, 1932; Bonatti, 1938; Crisci *et al.*, 1975; Di Pisa *et al.*, 1985; Coli, 1989) and also used for theoretical studies on the natural and artificial deformation of the calcite crystals related to the existence of various microstructures (Schmid *et al.*, 1980; Schmid *et al.*, 1987; Ferril 1991; Burkhard, 1993; Covey-Crump, 1997). These differences have been related to static and dynamic recrystallization during the tectono-metamorphic evolution of the different sectors of the Apuan complex (Molli *et al.*, 1997; 2000). Starting from the Upper Oligocene, the Apuan succession was affected by two main deformation events: the first (D_1), dated at 27 Ma (Kligfield *et al.*, 1986), gave rise to thrusting and isoclinal folding, and resulted from collision between the Adria microplate and the European continental margin; the second event (D_2), occurring between 15 and 8 Ma, is characterised according to Carmignani and Kligfield (1990) by an extensional phase related to the opening of the Balearic and Corsica basin. The polyphasic and syn-tectonic metamorphic mark yields mineralogical associations related to the greenschist facies (Carmignani et *al.*, 1978), the stable association pyrophyllite+quartz (Franceschelli *et al.*, 1986, 1997) indicating temperatures between 300-450°C and pressures reaching 3-4 Kbar (Di Pisa *et al.*, 1985; Franceschelli *et al.*, 1997).

Marbles with granoblastic and xenoblastic structures were formed during the first tectonic phase. The first microstructure is mainly due to static recrystallisation and a «recovery» process, since the microstructure associated with the D_1 polyphasic deformation was wiped out by the effects of the thermal peak. In these samples, the grain boundaries are well defined, being straight or slightly curved, and with no optical evidence of crystal-plastic deformation. The xenoblastic microstructure is related to dynamic recrystallisation, the main features being variable grain sizes, evident grain shape orientation, and grain boundary shapes from slightly curved or embayed to lobate. These structures are related to a late stage of the D_1 event and located in the shear zone, in which earlier granoblastic structures were reworked. Marbles with polymodal grain size distribution, evidently preferred orientation of grain shape, and dynamic recrystallization by sub-grain rotation formed during the second tectonic phase (D_2). These structures, which are associated with narrow, millimetric to decimetric shear zones in the Carrara area and large-scale folds in the Arni area, are related to exhumation of the metamorphic core in retrograde metamorphic conditions (Molli and Heilbronner, 1999; Molli et *al.*, 2000).

SAMPLES AND EXPERIMENTAL TECHNIQUES

For this study, ten representative marble samples from the Carrara and Arni basins were selected (fig. 1). These two sectors have distinct microstructural characteristics due to the differing tectonic events of the Apuan evolution. The samples were collected from historical and recent quarries, and derive from the same stratigraphical level (Liassic).

Most of the samples (9) come from the Carrara basin (Fantiscritti, Miseglia, Colonnata), because this is the most important marble basin of the whole Apuan Alps.

Only one sample was chosen from the Arni basin, in order to compare its petrophysical features with those of samples from the Carrara district. In fact, the commercially named ordinary Carrara marble is often confused with others white marbles like that from Arni.

Petrophysical methods

For microstructural observation, 2-5 μm ultrathin sections were used, in order to observe grain boundaries and recrystallisation bands. In normal thin sections (30 μm), the extremely high birefringence of calcite gives rise to interference colour fringes along grain and twin boundaries, hindering observation of grain geometry (Vernon, 1981). Our ultrathin sections were cut parallel to the principal planes of the finite strain ellipsoid (*xz* and *xy* sections identified during sampling) and photographed with an optical microscope. Scanning of microphotographs quantified the most important microstructural features with the help of NIH Image 1.6 software. The following parameters were recorded: surface area (A), perimeter (P), long and short axes (L,

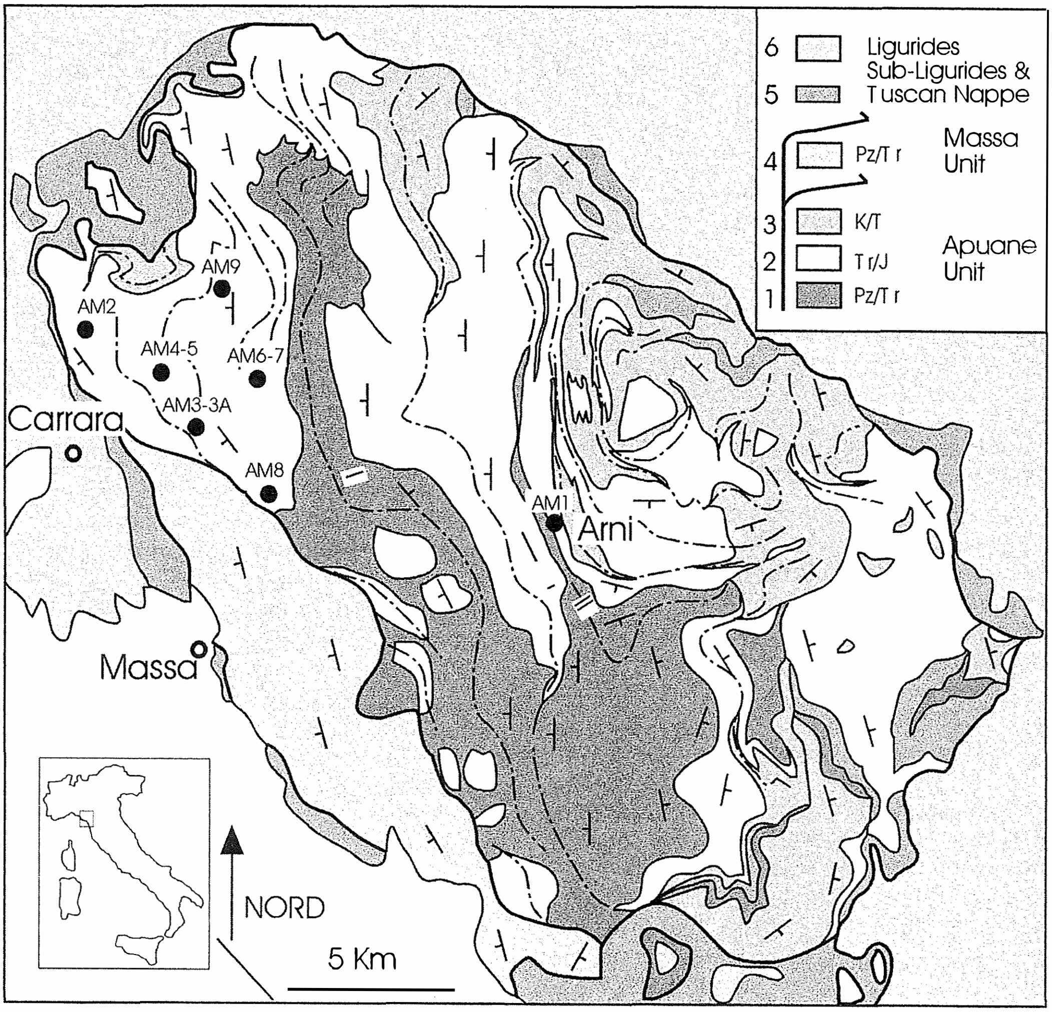
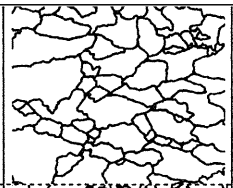
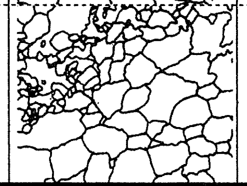
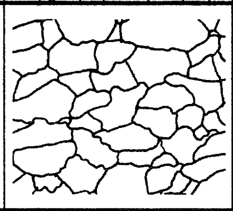
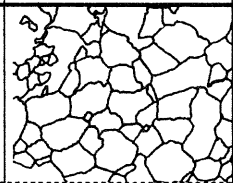
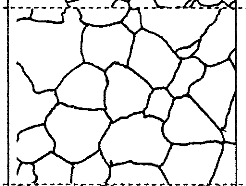
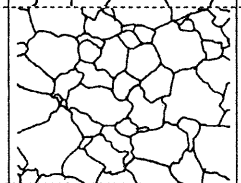
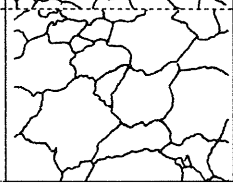


Fig. 1 – Geological sketch map of Apuan Alps (modified after Carmignani & Kligfield, 1990). 1) Paleozoic to Triassic: metavolcanics (porphyroids), phyllites, quartzites and metaconglomerates; 2) Upper Triassic to Liassic carbonate platform deposits, mainly metadolostones, marbles and cherty metalimestones; 3) Cretaceous to Terziary phyllites and metasandstones; 4) Massa Unit; 5) breccias and cataclasites largely derived from Triassic evaporites (Calcare Cavernoso) base of Tuscan Nappe; 6) Tuscan Nappe, Subliguride and Liguride units; I) Vinca-Forno anticline; II) Tambura anticline. Circles and numbers refer to studied samples.

S). Average values were calculated by means of statistic software using about 350 grains for each ultrathin section. Diameter was evaluated by averaging long and short axes (S+L)/2, without applying any kind of stereological correction. Average area values were used to obtain the values of equivalent circle $D^*=(4A^*/\pi)^{-1/2}$. Roundness R_D ($R_D=P^2/4\pi A$) was calculated for each grain and indicated the shapes and grain contacts of the crystals: R_D values of 1.3 indicate the linear contacts and triple junctions typical of granoblastic

TABLE 1

Microstructural characteristics and location of the Apuan marble samples.

Sample	Location	Microstructure	Recrystallisation	S.P.O.	R_D
AM1	Arni	XENOBLASTIC 	Dynamic, syn D1, remobilization of grain boundaries after thermal peak	Present, related to main foliation	1.96 m 1.00 σ 5.52 χ
AM6 **AM7**	Colonnata		Dynamic, syn D_2, grain boundary migration and subgrain subdivision	Present only in finer granulometric classes	1.74 m 0.58 σ 3.28 χ
AM2	Fantiscritti	ORIENTED GRANOBLASTIC 	Static	Present, makes an angle with foliation	1.58 m 0.41 σ 2.79 χ
AM4 **AM5**	Miseglia		Static	Present in granulometric classes < 100 μm	1.62 m 0.57 σ 5.25 χ
AM3 **AM3A**	Fantiscritti	GRANOBLASTIC 	Static	Not significant	1.38 m 0.38 σ 2.70 χ
AM8	Colonnata		Static	Not significant	1.44 m 0.38 σ 1.66 χ
AM9	Fantiscritti		Static	Not significant	1.52 m 0.40 σ 0.50 χ

(*m* = mean; σ = standard deviation; χ = skewness) (S.P.O. = Shape Preferred Orientation).

polygonal structure; values greater than 1.3 indicate migration of crystal boundaries and the tendency of the crystals to have strongly sutured grain boundaries (xenoblastic structure) (Burkhard, 1990). The data were analysed with statistic software in order to obtain the mean, standard deviation and skewness of the above parameters. The following granulometric classes are shown in the histograms of diameter data: $0<a<50\mu m$; $50<b<100\mu m$; $100<c<150\mu m$; $150<d<200\mu m$; $200<e<250\mu m$; $250<f<300\mu m$; $300<g<350$ mm; $h>350$ µm.

The relative abundances of major and minor elements were determined by XRF (Franzini *et al.*, 1975). Mineralogical compositions were defined by XRD and by applying a gasometric method for quantitative determination of calcite (Leone *et al.*, 1988).

Samples were also studied using CCL (Cold Cathode Luminescence) 8200mk^3 operating at 25 KV accelerating voltage and 250 microamperes. Photomicrographs were made with a Nikon camera with an exposure time of 20 sec.

Physical parameters – density (γ), bulk density (γ_s), total open porosity (P), mesoporosity, weight imbibition coefficient (IC_w), volume imbibition coefficient (IC_v) and saturation index (SI) – were determined according to Barsottelli *et al.* (1998). For each marble type, test samples of $1.8 \times 1.8 \times 2$ cm were dried at 60°C and their dry weight (W*d*) was determined. Real volume V*r* (measured using a QuantaChrome helium picnometer) and bulk volume V*b* (measured using a Chandler Engineering mercury porosimeter) were also determined. Density (γ) was calculated as W*d*/V*r*, bulk density (γ_s) as Wd/Vd, and total open porosity as $P\%=100\cdot(Vb-Vr)/Vb$.

Test samples were then dipped into deionised water to reach saturation, and their constant wet weight (W*w*) and adsorbed water (W*w*-W*d*) were determined. Weight imbibition coefficient IC*w* was calculated as $ICw\%=100\cdot(Ww-Wd)/Wd$. Volume imbibition coefficient ICv was calculated as $ICv\%=ICw\cdot\gamma_s$ Water saturation index SI was calculated as $SI\%=100\cdot ICv/P$.

Open porosity was subdivided into:

– macroporosity ($r>150\mu m$), absent in unweathered marbles;

– mesoporosity ($0.0037<r>150\mu m$), determined by a Thermo Quest mercury porosimeter;

– microporosity ($r<0.0037\mu m$), being as the difference between total open porosity and mesoporosity.

RESULTS

Microstructural data

Fig. 2 shows the three main textural types observed in this study. Figs. 2a and 2b show the xenoblastic structures of the sample from the Arni basin. The ultrathin section of sample AM1, observed in *xz* direction, (fig. 2a) shows: *i)* an evidently oriented grain shape; *ii)* small grain size (section is cut perpendicular to foliation plane); *iii)* sutured grain boundaries. The section AM1 *xy* (fig. 2b) shows: *i)* coarser grain size; *ii)* grain boundary shapes from slightly embayed to lobate. Fig. 2 also lists the values of the main microstructural parameters, including standard deviations and granulometric classes related to diameter D. In the histogram for AM1 *xz* (top), the predominance of a small grain size with unimodal distribution is observed, whereas that for section AM1 *xy* shows bimodal distribution.

Figs. 2c and 2d refer to the oriented sections of sample AM2 from the Fantiscritti basin. In these cases, unimodal distribution of grain size is evident.

Fig. 2e refers to sample AM9, which is an example of granoblastic polygonal structure. The histogram reveals the absence of any dominant granulometric class.

Table 1 lists the samples, their location in the Apuan Alps, a diagram of their microstructures, type of recrystallisation, presence of preferred orientation and roundness values. Data on microstructural features show that the Apuan marbles may be grouped into

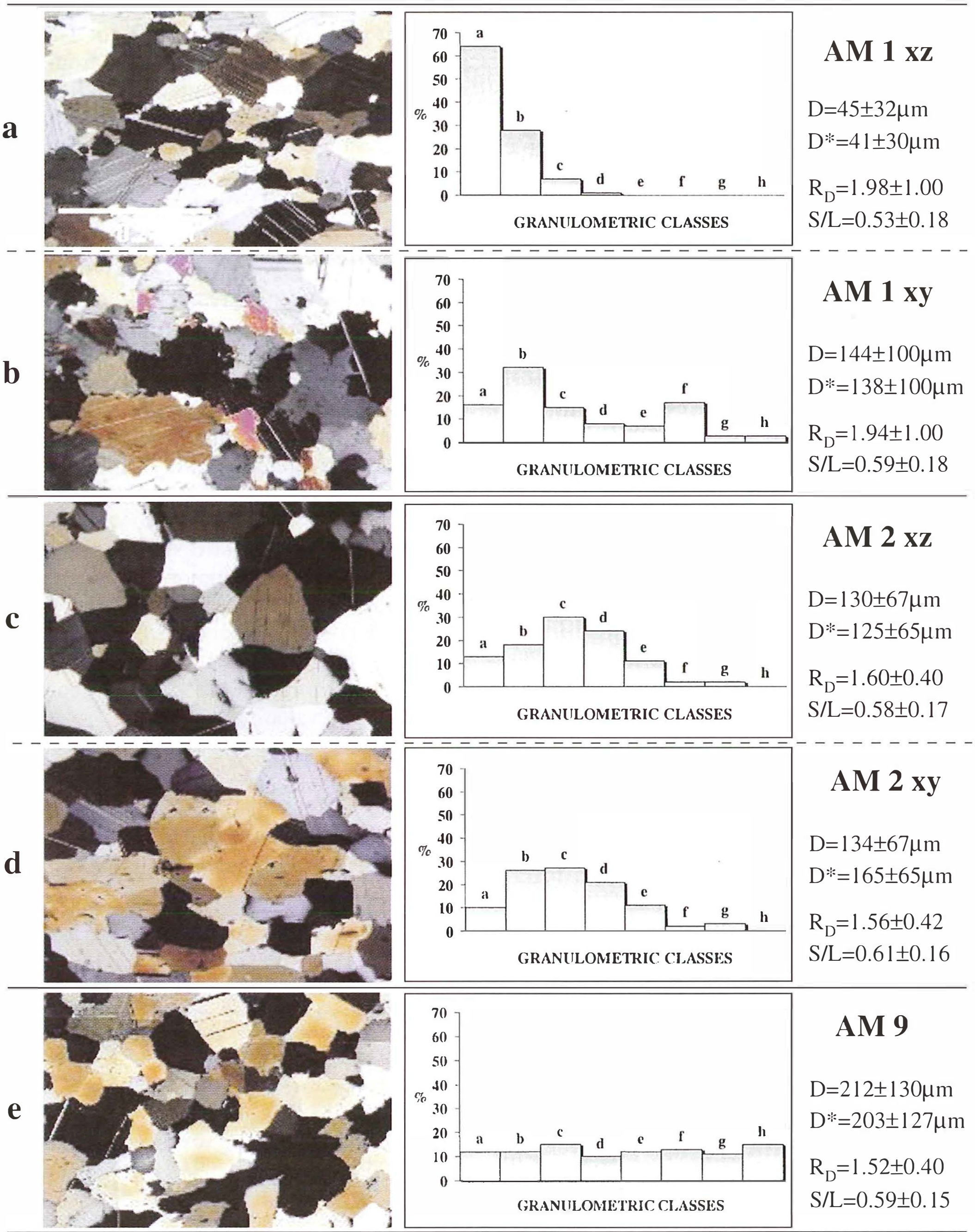


Fig. 2 – Micrographs, histograms of granulometric classes and main microstructural parameters for three representative studied samples: a and b (xenoblastic structure), c and d (granoblastic oriented structure), e (granoblastic polygonal structure). D = diameter; D*=diameter equivalent circle; R_D = roundness, S/L = axial ratio.

two main types of microstructure: polygonal granoblastic, forming during static recrystallisation and xenoblastic, forming during dynamic recrystallisation, both due to the different tectonometamorphic evolution of different sectors of the Apuan Alps. Sample AM2, however, has a structure with intermediate characteristics.

CHEMICAL AND MINERALOGICAL DATA

The samples are almost pure marbles containing mainly calcite, and they therefore have homogeneous chemical and mineralogical composition (Table 2). The presence of dolomite was determined qualitatively by X-ray diffraction. However, the small Mg contents in all samples indicate that this mineral is always present in a very low percentage. Diffractometric analyses carried out on insoluble residual deposits after acid attack (HCl 0,1N) reveal that accessory minerals are mainly quartz, micas, albite and pyrite.

The samples reveal different colours under cathodoluminescence (CL). Figs. 3a and 3b show photos of thin sections of samples AM1 and AM9 (chosen as representative of the different CL). All samples from the Carrara district show blue luminescence; that from Arni reveals orange luminescence. The different colours under CL suggest that the marbles have a distinct ratio between activator and quencher elements in the calcite structure (Marshall, 1988).

Physical properties

Measurements of physical properties are listed in Table 3. Total open porosity gives almost identical values for all examined samples. Significant differences are shown by mesopororosity, correlated with the different microstructures. The sample with polygonal granoblastic structure (AM9) shows almost unimodal distribution of mesopores, shifted towards larger pore diameters, whereas the sample with xenoblastic structure (AM1) shows bimodal distribution of mesopores (figs. 4a and 4b).

These data confirm that the different types of marbles have distinct SI and that the granoblastic samples (AM9, AM3) generally have higher SI values.

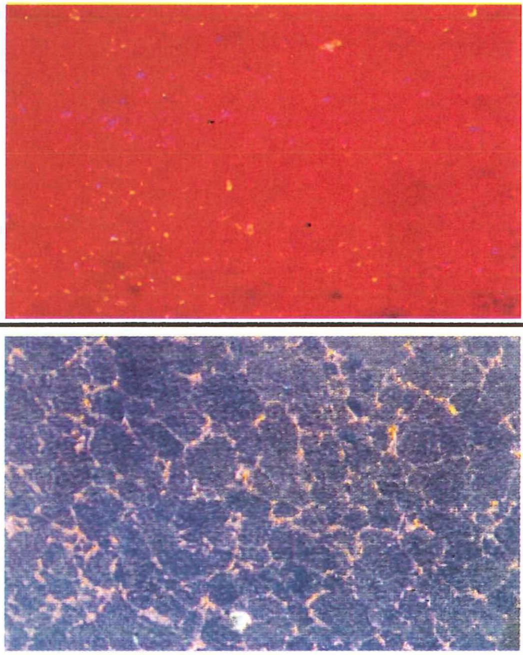
TABLE 2

Chemical and mineralogical analyses (Weight %). All elements determined by XRF; LOI by lost of ignition at 900°C; CO_2 by gasometric methodology; $CaCO_3$ calculated on basis of CO_2.

Sample	AM1	AM2	AM3	AM3A	AM4	AM5	AM6	AM7	AM8	AM9
SiO_2	0.23	1.60	0.14	0.17	0.12	0.12	0.33	0.47	0.19	0.15
TiO_2	0.01	0.33	bdl	bdl	bdl	bdl	0.01	0.01	bdl	bdl
Al_2O_3	0.11	0.71	0.05	0.05	0.04	0.05	0.15	0.21	0.06	0.06
Fe_2O_3	0.05	0.24	0.03	0.03	0.02	0.02	0.04	0.04	0.02	0.02
MgO	0.95	1.25	1.22	0.95	1.16	0.98	1.05	1.69	1.21	0.71
MnO	0.01	0.01	0.01	bdl	bdl	bdl	bdl	0.01	0.01	0.01
CaO	55.44	53.02	55.48	55.00	55.50	55.44	54.89	54.45	55.58	55.31
Na_2O	0.03	0.02	0.03	0.01	0.03	0.03	0.02	0.03	0.03	0.03
K_2O	0.03	0.22	0.02	0.01	0.02	0.05	0.05	0.08	0.02	0.02
LOI	43.14	42.90	43.04	43.76	43.10	43.33	43.46	43.01	42.88	43.69
$CaCO_3$	99.9	95.3	98.2	97.0	98.8	99.0	100.4	97.8	98.5	100
CO_2	43.9	41.9	43.2	42.6	43.4	43.5	44.1	42.9	43.3	44.2

bdl:below detection limit.

a



AM 1 Arni basin

b

AM 9 Carrara basin

Fig. 3*a,b* – Microphotographs in cathodoluminescence.

TABLE 3

Physical parameters of Apuan marbles: γ=Density (g/cm³); γs=Bulk density (g/cm³); P=Total open porosity (%); ICw=Weight imbibition coefficient (Weight %); ICv = Volume imbibition coefficient (Volume %); SI=Saturation index (Weight %); Microporosity (r<0.0037μm) and Mesoporosity (0.0037μm<r<150μm).

Sample	AM1	AM2	AM3	AM3A	AM4	AM5	AM6	AM7	AM8	AM9
g	2.72	2.73	2.73	2.72	2.71	2.72	2.72	2.72	2.72	2.71
	± 0.00	±0.01	±0.01	±0.00	±0.00	±0.01	±0.00	±0.00	±0.00	±0.00
g_s	2.69	2.68	2.68	2.69	2.67	2.67	2.68	2.69	2.69	2.66
	±0.01	±0.01	±0.01	±0.01	±0.01	±0.01	±0.01	±0.01	±0.01	±0.01
P%	0.97	1.82	1.68	1.27	1.59	1.84	1.51	1.44	1.14	1.79
	±0.46	±0.33	±0.51	±0.17	±0.29	±0.40	±0.31	±0.45	±0.31	±0.53
IC*w*	0.07	0.13	0.09	0.10	0.07	0.13	0.06	0.07	0.05	0.24
	±0.01	±0.02	±0.01	±0.01	±0.02	±0.01	±0.01	±0.01	±0.01	±0.01
IC*v*	0.19	0.34	0.24	0.24	0.20	0.36	0.16	0.20	0.14	0.63
	±0.03	±0.07	±0.03	±0.03	±0.05	±0.03	±0.03	±0.04	±0.03	±0.04
SI	17	19	31	19	13	20	11	15	12	35
	±5	±5	±10	±2	±7	±8	±2	±6	±3	±7
Meso	0.35	0.23	0.44	0.45	0.24	0.81	0.28	0.36	0.39	0.75
Micro	0.62	1.59	1.24	0.82	1.35	1.03	1.23	1.08	0.75	1.04

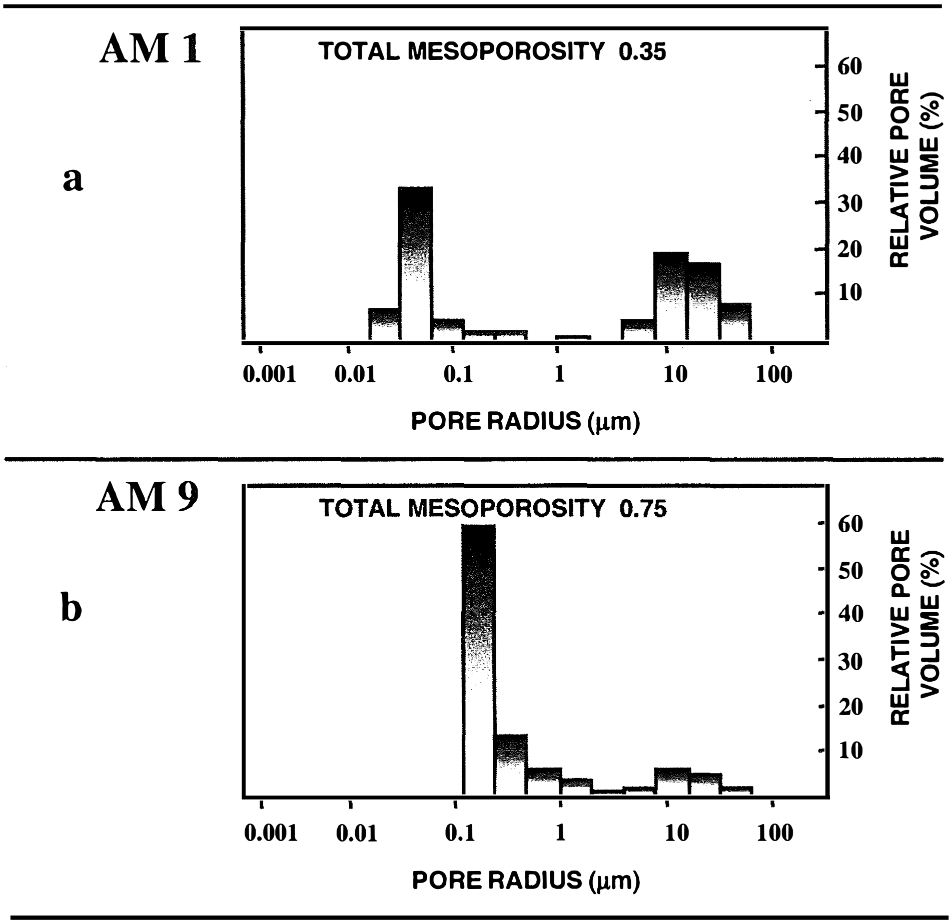


Fig. 4*a,b* – Mesopore distribution of Apuan marbles showing different microstructures: a (xenoblastic structure) and b (granoblastic polygonal structure).

CONCLUSION

The Apuan marbles examined here have homogeneous mineralogical and chemical composition, whereas the samples are markedly different from the petrographical and structural points of view. Their differing structural characteristics are due to the particular tectonic evolution of the Apuan Alps (Molli *et al.*, 2000). The obtained data may be used for identifying the provenance of the Apuan marbles used in many of the historical buildings and monuments representing the cultural heritage. A simple ultrathin section, obtained by applying a microdestructive methodology, can supply information about microstructural characteristics which, associated with cathodoluminescence colours, define the district of provenance of the sample. The various types of microstructures can be described by means of quantitative parameters such as grain diameter, diameter of equivalent circle and roundness, related to the petrophysical features of the marbles. The type of microstructure does affect the porosity of rocks: in particular, mesopore distribution seems to be linked to microstructure. The marbles with granoblastic structure show larger mesopore dimensions, whereas those with xenoblastic structure have smaller mesopores and bimodal mesopore distribution. Saturation index data can also be related to the various microstructures. As the durability of marbles depends on the amount and manner of water absorption, marbles with granoblastic structure and larger mesopores have low durability when placed in an external environment. Their behaviour with respect to thermal shocks is also related to these structural features (Boineau and Perrier, 1995). Among the studied samples xenoblastic marbles with higher roundness have better resistance to weathering. This is evident in Florence Cathedral, where marbles with granoblastic structure are badly affected by decay with respect to marbles with xenoblastic structure.

Cathodoluminescence analysis reveals red/orange or blue colours, although these results do not fit those of Barbin *et al.* (1992), which indicate that all the Apuan marbles are orange. The red/orange colour of the Arni sample compared with the blue of all the samples from Carrara may represent a further method for recognising the provenance of white Apuan marbles.

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