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Microstructures associated with static and dynamic recrystallization of Carrara marble (Alpi Apuane, NW Tuscany, Italy)

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Abstract

The present contribution summarizes the first results of a study focusing on microstructures from Alpi Apuane marbles. Its aim is both an analysis of the evolution of the metamorphic complex recorded in marbles and the supply of basic material for process-oriented studies on calcite microstructures due to natural deformation. Quantitative analysis of the variations of statically recrystallized microstructures suggest a relationship with the peak metamorphic temperatures. Previously unrecognized post-thermal peak shear zones, showing overprint microstructures typical of grain-boundary migration and dynamic recrystallization, are described; they document the natural deformation of Carrara marble.

Introduction and geological overview

The Alpi Apuane marbles – and in particular the pure white variety called 'Carrara marble' – are well known all over the world. They have been quarried for more than twenty centuries since the Roman age and are still extensively used as building stone as well as for sculpturing. Carrara marbles are also well known in the geological literature; they were studied since the early seventies, when they became one of the 'classical' materials for experimental deformation tests (see, among others, Rutter 1972, 1995, Kern 1977, Casey et al. 1978, Spiers 1979, Schmid et al. 1980, Schmid et al. 1987, De Bresser 1991, Covey-Crump 1997, and references in these studies). No systematic microstructure- oriented study based on field data has been carried out to date, however.

The Alpi Apuane in NW Tuscany represent the largest tectonic window in the Northern Apennine fold and thrust belt and their marbles are metamorphosed carbonate-platform sediments forming part of a Mesozoic succession deposited on the former Italic-Adriatic continental margin. During the Tertiary, this continental margin was deformed in several phases and metamorphosed under greenschist-facies conditions (Carmignani & Kligfield 1990 and references therein). According to recent tectonic interpretations (Carmignani & Kligfield 1990), the Alpi Apuane region represents an example of a 'core-complex' structure formed during the post-collision extensional process beginning with the overthickening of the accretionary wedge during the Early Miocene and followed by large-scale extension related to the opening of the Tyrrhenian Sea.

As a result of this history, Carrara marbles are considered to be completely annealed, i.e., they were statically recrystallized during a thermal event postdating deformation. During annealing, the marbles acquired their typical 'foam structures' showing equant grain shapes, straight grain boundaries and coarse grain sizes. The main disadvantage of the pervasive annealing is that it resulted in the absence of dynamic microstructures, i.e., absence of any trace of the deformation mechanisms that were operational during the large-scale deformation of the Alpi Apuane. In other words: although substantial information exists on the deformational behaviour of Carrara marble under experimental conditions, practically nothing is known about its deformational behaviour under natural conditions. Shear zones have, however, recently been found in Carrara marble associated with microstructures clearly overprinting the equilibrated microstructures of the statically recrystallized marble (Molli unpublished data; Molli & Meccheri 1997). This allows unraveling of the post-thermal peak evolution in marbles, their deformational behaviour and the related calcite deformation microstructures. Both statically recrystallized (annealed) marble (i.e. the marble representing the 'starting' material) and dynamically recrystallized marble (i.e. the marble showing traces of active deformation mechanisms) were analyzed. Starting from detailed structural field work, optical microscopy was coupled with image analysis methods in order to obtain a quantitative description of the microstructures. Particular attention was paid to:

- the grain size and grain shape variations of the annealed marble;
- the dynamic overprint microstructures in post-thermal peak shear zones.

Samples

We collected samples of white marbles from different geometrical positions within the structural building of the Alpi Apuane. Two samples (A and C) were collected on the normal limb of the two major recumbent isoclinal folds/nappes, the Vinca-Forno and Monte Tambura structures. They represent the western and the central-eastern part of the metamorphic complex, respectively. Both sites of sampling are within 20 m of the contact with the underlying dolomitic-marble ('Marmi dolomitici Fm.' of Carmignani 1985); the sedimentary succession of both sections was dealt with by Carmignani (1985) and Coli & Fazzuoli (1992). This sampling method guarantees that microstructures of identical or nearly identical lithotypes can be compared. This is confirmed by the composition of the rocks (more than 99% of calcite, plus minor contributions of quartz, albite, white mica and/or opaque minerals) and by the chemical analyses of major and minor elements (Molli & Giorgetti unpublished). Calcite/dolomite thermometry (Molli et al. 1999) indicates temperatures of approximately 430 °C in the area of sample A and 380 °C in the area of sample C.

On the basis of new structural field data (Molli & Meccheri 1997, Molli & Meccheri in press), we have been able to analyze previously unrecognized shear zones (sample F) overprinting and deforming the main regional foliation and related structures. These shear zones, ranging in width from millimeters to decimeters, can be considered as post-main-phase deformation and therefore attributed to the D2 deformational event according to the Carmignani & Kligfield (1990) model. Calcite/dolomite analysis suggests a temperature of 350 °C for the development of these shear zones (Molli et al. 1999).

All samples under study were cut parallel to the stretching lineation, L, and normal to the foliation plane, S. These structural elements were easily recognizable in sample F from the shear zone. In the case of samples A and C, the attitude of nearby (a few meters) impure phyllosyllicate-rich or dolomite-rich marbles showing a well developed bedding-parallel foliation and associated stretching lineations (usually defined by calcite/quartz strain shadows around pyrite crystals or elongate minerals) was taken into account.

Methods of shape and grain-size measurement

For the microstructural analysis, ultra-thin sections (thickness less than 5 μm) were used. Optical photomicrographs were prepared from these thin sections. Transparent paper was placed over the enlarged cross-polarized-light micrographs. Pictures taken in more than one orientation allowed tracing of well defined grain boundaries. The resulting grain-boundary drawings were scanned on a flatbed scanner (300 dpi), postprocessed and saved as bitmaps.

Grain-size distributions (3-dimensional volumetric density histograms) were determined using the StripStar method (Heilbronner & Bruhn 1998). This program is based on the same theory that underlies the Schwartz-Saltykov method (Underwood 1970). The distribution h(R) of radii of spheres is derived from the distribution of cross-sectional circles, h(r). Since any h(R) distribution generates an h(r) distribution that trails off to zero, an h(r) distribution of which lower classes are empty cannot be generated from any real h(R). In practice, one will strive to prepare (i.e., measure) an h(r) distribution that has no empty classes and that produces the least number of antispheres, i.e., the negative occurences of the resulting h(R) distribution. Means for obtaining good h(R) distributions increase with sample size and/or with adjusting the class width (see Heilbronner & Bruhn 1998, for more details).

Using NIH Image (public domain software 1997), the pixel areas and the perimeters of the digitized grains were measured. The resulting files of the analyses were exported to a spread-sheet program (Kaleidagraph 1994). The sizes of cross-sectional areas of the grains were given in pixels, and had to be scaled and converted to μm^2. Subsequently, the diameter of a circle with the same surface area as the cross-sectional area was calculated. A minimum of 400 grains was evaluated for each sample. The data (20 classes) of the equivalent half diameter (radius) were used as imput for the StripStar program. The output is represented as a 3-D volumetric density histogram, a representation portraying the relative volume as a function of 3-D grain diameters.

The shape of the grains (or particle fabric) was analyzed using the PAROR method. This method (Panozzo 1983) is founded on the basic concept of the projections of the cross-sectional outlines of particles onto a reference axis for different angles of rotation.The total length of projection, $B(\alpha)$, depends on both size and shape of the particles, and on the distribution orientation function of the long axes. The preferred orientation of the grain boundaries (or the surface fabric) was analyzed with the SURFOR method (Panozzo 1984), which uses the projection diagrams for $A(\alpha)$ representing the total length of projected grain-boundary outlines onto a reference axis per angle of rotation. Using the ratios $A(\alpha)_{min}/A(\alpha)_{max}$ (SURFOR) or $B(\alpha)_{min}/B(\alpha)_{max}$ (PAROR), it is possible to derive a bulk anisotropy ratio of the analyzed fabric.

Finally, the corrugation or serratedness of the grain boundaries was quantified using the SHAPES program, which yields the PARIS factor (a measure for the convexity–concavity of shapes, Panozzo & Hurlimann (1983). This method uses the above defined projection functions $A(\alpha)$ and $B(\alpha)$, taking into account that in the cases of a fully convex grain-boundary outline $A(\alpha)$ and $B(\alpha)$ must coincide, whereas occurring differences can be used as indicators of corrugations of grain boundaries (see also Schmid et al. 1987).

To obtain the imput data necessary for the PAROR, SURFOR and SHAPES programs, either the original grain-boundary maps were digitized manually on a digitizing tablet (in the case of sample A and C) or the scanned bitmaps were used for further digital processing (sample F). Using Photoshop 3.0.4 (1997), high-resolution bitmaps (1000–3000 dpi) were calculated; using a special version of NIH Image (Ime d'Ouline 1995), the grain-boundary vectors were calculated automatically from the image matrix.

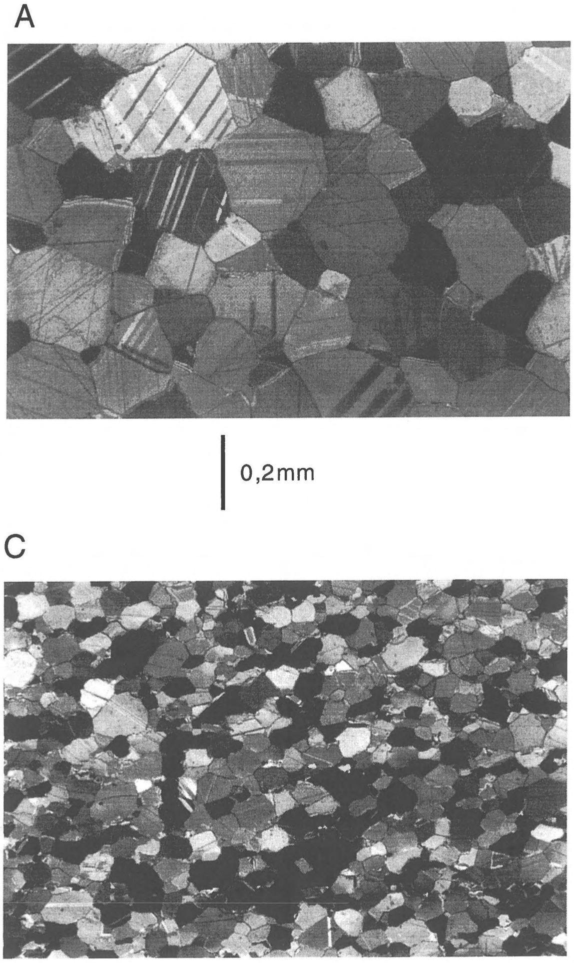

Figure 1. Micrographs of thin sections of statically recrystallized (i.e. annealed) Carrara marble. Scale bar applies to both micrograps; crossed polarizers. Sample A: normal limb of Vinca–Forno anticline, La Rocchetta quarry; Sample C: normal limb of Monte Tambura anticline, Passo Focolaccia quarry.

The microstructures of the statically recrystallized Carrara marble

So far, microstructural analyses of Carrara marble confirmed that this material has a regular granoblastic polygonal 'foam' structure, an average grain size of approximately 100–250 μm, grains of nearly isometric shape (no evident grain-shape orientation), and no crystallographic preferred orientation (cf. Ramez & Murrel 1964, Rutter 1972, Kern 1977, Casey et al. 1978, Schmid et al. 1980).

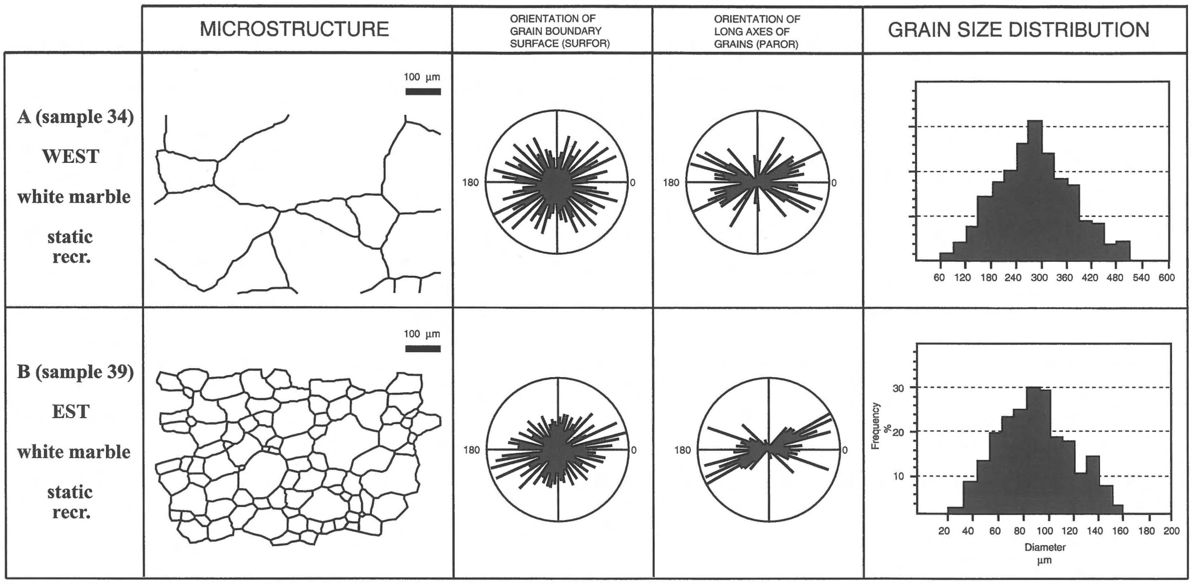

Figure 2. Shape and grain-size analyses of samples A and C. A representative detail of the microstructure (grain-boundary map), the rose diagrams of grain-boundary surface orientation (surface ODF), the rose diagrams of the long axes of the calcite grains (axes ODF), and the histogram of volumetric density versus 3-D radius (μm) are indicated for each sample. The reference axes for the SURFOR and PAROR diagrams are the foliation trace (O°/180° line) and its normal.

Although homogeneous and isotropic samples with the above mentioned features can be found in the various quarries, meso- and microstructural variability at a regional scale were noted for a long time (see, among others, Zaccagna 1932 and his previous papers, Bonatti 1938, Crisci et al. 1975, Di Sabatino et al. 1977, Di Pisa et al. 1985, Coli 1989). Our samples A and C are representative of such a variability. Both samples A and C (Figure 1) show an equigranular polygonal or granoblastic microstructure. Optical evidence of crystal-plastic deformation is almost completely absent and only represented by thin e-twins, which appear to be related to a late stage of low-temperature deformation. Straight to slightly curved grain boundaries are present in both samples. Moreover, they are characterized by very weak to completely absent crystallographic preferred orientation (as measured with the U-stage).

The microstructural analyses of samples A and C are presented in Figure 2. Both samples have a unimodal 3-D volumetric grain-size distribution with an average 3-D diameter of 90 μm in the East (sample C) to 300 μm in the West (sample A). The analysis of the preferred orientation of the grain-boundaries' surface (SURFOR) reveals a nearly random orientation distribution (surface ODF) for sample A and a more unimodal and asymmetrical surface orientation distribution for sample C (Figure 2). The slightly bimodal surface distribution that is discernible in the rose diagram of the surface ODF of sample A may reflect a weak grain-boundary alignment of rhombic symmetry, which has been noticed before for undeformed marble (Schmid et al. 1987). The asymmetric surface ODF of sample C shows a primary orientation at a low angle to the foliation trace.

The analysis of the preferred orientation of particle axes (PAROR) also shows that the microstructure in sample A (Figure 2(A)) tends to be more isotropic (showing a less preferred orientation of the axes in the rose diagram) than that of sample C. Both marbles appear to have a slightly bimodal distribution of long-axis orientation, with the first maximum being inclined at approx. 20–30° with respect to the foliation plane, and a second maximum at 20–30° in the opposite direction. On the whole, the western variety (sample A) has a more random orientation than the eastern one (sample C). Note that the symmetry/asymmetry of the surface ODFs and the preferred orientations of the particle axes of samples A and C coincide rather well. The bulk anisotropy ratio (derived from PAROR and SURFOR) of sample A is 0.85; the ratio of sample C is 0.77. The average aspect ratios (short/long axis) of the individual grains are of the order of 0.65 for both samples. The PARIS factor of microstructures is very low: 1.8% for sample A and 2.3% for sample C.

The microstructural features of samples A and C suggest static recrystallization or 'annealing'. The term 'annealing' is widely used in metallurgy, where it indicates the process of recovery and static recrystallization induced after the heating of previously deformed material. It is also used for microstructural interpretation in rocks (Vernon 1975, Schmid 1982, Shelley 1993, Passchier & Trouw 1996 and references therein). In natural rocks, the reorganization of a dynamic microfabric through recovery, recrystallization and grain-boundary area reduction (Urai et al. 1986, Passchier & Trouw 1996) is a common phenomenon reflecting that, in many geological settings, temperature instabilities do not decay as fast as geological strain rates. The main effect of 'natural annealing' is the reduction or complete elimination of crystal defects, the equilibration of grain boundaries, grain coarsening, an overall weakening, and/or complete destruction of the previously acquired preferred orientation (Turner & Weiss 1963, Tullis & Yund 1981, Schmid, 1982, Covey-Crump & Rutter 1989, Handy & Streit 1996). The microstructures of samples A and C can therefore be interpreted in terms of static recrystallization after deformation.

The differences between the two samples concern the grain size (300 μm in sample A, 90 μm in sample C), the preferred orientation of grain boundaries (a nearly random distribution in sample A; a more unimodal and asymmetrical distribution in sample C) and the preferred orientation of particle axes (more isotropic in sample A than in sample C). A slightly different bulk anisotropy ratio with a similar average aspect ratio (short/long axis) of the individual grains implies that the long axes of the grains are not perfectly aligned but show some degree of orientational scatter, more pronounced in sample A than in sample C. The PARIS factor, which is very low in both sample A and sample C, implies that – at the scale of the digitization – the grain boundaries are fully convex along most of the grain-boundary outlines, which in turn suggests that the grain boundaries are straight; a lower value can, however, be observed in sample A. All these data suggest that sample A suffered a more complete annealing process than sample B.

According to calcite/dolomite thermometry (Molli et al. 1999) the area of sample A shows a peak temperature of 430 °C, whereas it is 380 °C for sample C. Assuming that the measured peak temperatures were attained during the annealing process, it thus

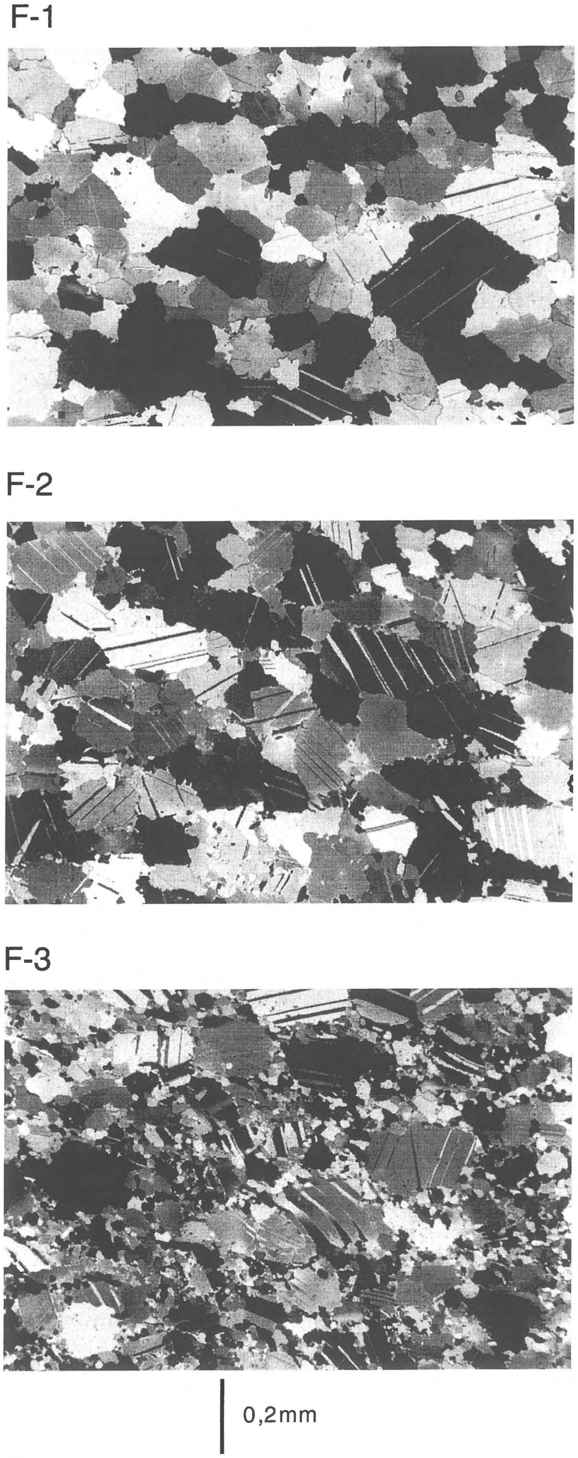

Figure 3. Micrographs of ultra-thin sections showing dynamic recrystallization overprinting of the annealed Carrara marble. Transition from weak (F-1) to strong (F-3) overprint. The long axes of the micrographs are parallel to the shear-zone boundary. The approximate distance between the microstructures is 10 mm in the direction normal to the shear-zone boundary. The scale applies to all micrographs; crossed polarizers.

appears that the efficiency of annealing as quantified in the various microstrutural parameters (e.g., the straightness of the grain boundaries) depends on the temperature.

Recrystallization overprint

The most recent studies on the Alpi Apuane marbles (Di Pisa et al. 1985, Coli 1989) suggest that the end of the microstructural history of these rocks coincides with the thermal-peak effects that produced the above described microstructures. We have been able however, on the basis of our new structural field study, to analyze previously unrecognized shear zones overprinting the annealed Carrara marble.

In association with these shear zones, a gradual modification of the microfabric in the marble can be observed (Figures 3–4). The equigranular polygonal granoblastic protolith (mean grain diameter approx. 150 μm) shows evidence of intracrystalline deformation (mainly undulatory extinction) and deformation twins in the low-strain domain; grain-boundary migration can be also observed (Figure 3, F-1 and F-2). Towards the high-strain zone (F-3), the tortuosity of the grain boundaries, i.e. the PARIS factor, increases from approx. 10% (F-1 and F-2) to 23% (F-3). The large relict grains have broad, lenticular twins, some of which show evidence of twin-boundary migration. Bulging recrystallization and subgrain rotation appear to be present together. The wavelength of the grain-boundary bulges corresponds to the grain size of the newly formed recrystallized grains.

In the grain-size histograms (Figure 4), an increase of the grain-size fraction of dynamically recrystallized new grains with diameters in the 20–50 μm range can be observed from the low to the high strain zone (F-1 to F-3). The recrystallized grains have smooth grain boundaries, as evidenced by their low PARIS factor (i.e., 5%). The recrystallization occurred preferentially at the boundaries between the old grains; a progressive localization of recrystallized grains in submillimetric bands, subparallel to the shear-zone boundaries, can be observed. The newly recrystallized calcite shows an oblique shape preferred orientation (PAROR and SURFOR of F-3) (Figure 4) with respect to shear-zone boundaries.

These rock-types are currently being studied intensively (Oesterling et al. 1999) because they represent the first examples of the naturally deformed Carrara marble described in literature. Since a large data set of experimentally deformed Carrara marble exists, a thorough comparison of experimentally and naturally deformed materials will be possible.

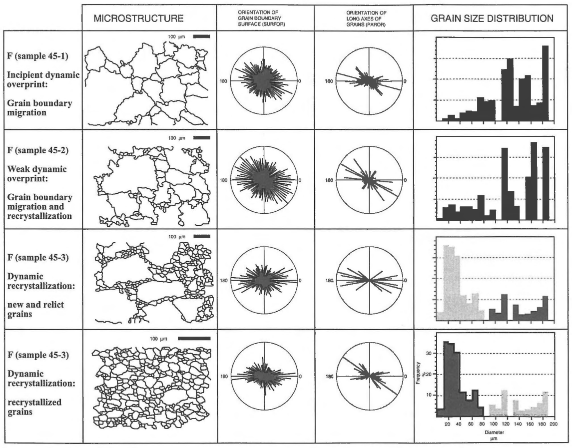

Figure 4. Shape and grain-size analysis of sample F. (F-1), (F-2) and (F-3) show increasing influence of the dynamic recrystallization overprint. A representative detail of the microstructure (grain-bounday map), the rose diagrams of grain-boundary surface orientation (surface ODF), the rose diagrams of the long axes of calcite grains (axes ODF), and the histogram of volumetric density versus 3-D radius (μm) are indicated for each sample. – For (F-3), the relict grains have been analyzed separately; the right and left parts of the grain-size histogram apply, respectively.

Conclusions

The present contribution describes quantitatively two examples of static recrystallized Carrara marble, the average grain diameter of which ranges from 90 μm to 300 μm. This grain-size variation is associated with a difference in the peak metamorphic temperature (calcite/dolomite geothermometry) from 380 °C to 430 °C. The marble in the western part of the study area is coarse grained and more isotropic than in the eastern part; its surface and particle fabric tend to be more random, and the grain boundaries thend to be straighter. This is attributed to static annealing and suggests a different thermal evolution of the western and central-eastern part of the Alpi Apuane metamorphic complex during and after the main phase of deformation, D1.

In post-thermal peak shear zones (D2), a gradually increasing overprint of annealed Carrara marble can be observed; it is described here for the first time. The wavelength of the migrating grain boundaries and the size of recrystallized grains are approximately equal. The 3-D grain-size histogram shows a gradual decrease of the original grain-size fractions in the 150–200 μm range and a gradual increase of the fractions in the 20–50 μm range. The microstructures are typical of grain-boundary migration and dynamic recrystallization.

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