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Role of grain boundaries and micro-defects on the mechanical response of a crystalline rock at multiscale

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ABSTRACT

The paper examines the cracks initiation, propagation and coalescence until failure in a crystalline rock under stress states involving rock volumes varying in size, from nano (Berkovich indentation in Continuous Stiffness Measurement mode) to micro (Vickers indentation, scratching) and macro (Rockwell indentation, Rock Impact Hardness Number) scales. The experimental work was performed on a calcitic marble characterized by two different textures (xenoblastic and granoblastic). The grain boundaries microcracks were deeply studied with Focused Ion Beam–Scanning Electron Microscopy technique, innovative approach in Rock Mechanics. The experimental results show that, at the three investigation scales, the microcracks give rise to the rock failure with fractures propagation mechanisms varying with the microstructure. In the granoblastic texture, fracturing is ruled by grain boundaries microcracks, which often appear almost open. Conversely, the fracture behavior of the xenoblastic marble seems dominated by the grains cleavage, even at the macroscale.

1. Introduction

The igneous, metamorphic and evaporitic rocks are aggregates of single or poly-mineral grains. They are affected by microdiscontinuities differing for their genesis, shape and size (i.e. intragranular, transgranular, intergranular and grain boundaries microcracks) [1,2].

The microcracks network can determine an anisotropic mechanical response of the rock, depending on the cracks statistically dominant orientation more than the grains iso-orientation [3–9]. More generally, each type of microdiscontinuites influences differently and always significantly the rock mechanical behavior (deformability and strength), since by cracks rock failure phenomena under stresses initiate [10–15]. Several experimental studies in literature [16–21] show that microstructures rule the cracks initiation, propagation and coalescence at the laboratory scale (macroscale). It seems also that some dynamic failure phenomena at the rock mass scale, i.e. spalling and rockburst, occurring in overstressed brittle hard rock under high overburdens, may be associated to the initiation, propagation and coalescence processes in the rock materials [22]. The deformation of rock specimens under uniaxial stresses is described by Bieniawski [23] who assumed an initial closure of microcracks followed by fracture initiation, stable and unstable fracture propagation and coalescence until failure. However, the validity of this model has been limited to qualitative observations with optical and scanning electron microscopy [12,13,24–26].

Therefore, in order to properly understand the above mentioned mechanisms, we performed a wide investigations campaign with a multidisciplinary and multiscale approach, from nano to macroscale, on a calcitic marble. We induced the neoformation of microcracks in different ways and studied the influence of preexisting microdiscontinuities on the crack initiation, growth and coalescence. In particular, several indentation and scratching techniques were applied on samples of different sizes with various loads on the indenter (from nano to macroscale) in order to obtain a broad framework of the rock fracturing mechanisms with changing both the induced stress states and the sample volume. Indeed, Berkovich nanoindentations were conducted inside the grains to examine, quantitatively and qualitatively, the interactions between the induced stress field and the intragranular cracks, while Vickers and Rockwell indentations and scratching, involving more grains and grains boundaries, were carried out to analyze the interactions between the indenter and the other types of cracks. Moreover, through Vickers indentations at various loads, the effect of the applied load on the marble hardness, in terms of indentation size effect (ISE), was quantified. The method [27,28] adopted for Vickers indentation data processing is introduced for the first time in the Rock Mechanics field and is illustrated in the paper.

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Finally, the microfracturing generated by scratching and indentations in the two textures was compared with the failure surfaces produced by the steel mass impacts in Rock Impact Hardness Number [29,30]. This test, although still not widespread, has been taken into consideration as well it highlights the relationship between the texture and the sample strength [21].

In conclusion, the experimentation, whose results are discussed in this paper, is innovative, since, despite the importance of studying the deformation and failure phenomena at the lowest scale for many engineering applications (drilling, blasting, sawing and comminution), the approach adopted and described in the present paper is very limited. Indeed, only recently, Bandini et al. [31] analyzed the effects of the intra-crystalline microcracks on the mechanical behavior of a marble at the nanoscale, by using nanoindentation in Continuous Stiffness Measurement (CSM) modality [32,33]. The CSM mode applied also in this study allowed us to examine the cracking mechanisms inside single grains of a marble during indentations, with a spatial resolution of a few nanometers and it has proven particularly suited to study the nanoscale failure mechanisms of crystalline rocks.

Table 1
Mechanical properties of the investigated marble (X=xenoblastic; G=granoblastic; ρ: density; n=total porosity; σ_c=UCS; $E_{t,50\%}$=tangent elastic modulus at 50% of UCS; σ_t=UTS; RIHN=Rock Impact Hardness Number; $V_{p,\mathrm{dry}}$ and $V_{p,sat}$=dry and saturated P-waves velocities; N=number of tests; m=mean value; ν=variation coefficient).

		N	m	ν (%)
ρ [kg/m^3]	X	6	2661	0.2
	G	11	2663	0.2
n [%]	X	6	3.5	5.7
	G	11	3.6	4.4
σ_c [MPa]	X	4	94.3	6.9
	G	4	66.9	6.3
$E_{t,50\%}$ [GPa]	X	3	58.2	2.4
	G	3	57.4	4.3
σ_t [MPa]	X	3	8.1	7.5
	G	3	5.8	5.2
RIHN	X	36	31	12.5
	G	36	7	35.8
$V_{p,dry}$ [m/s]	X	6	5446	1.9
	G	11	2538	8.3
$V_{p,sat}$ [m/s]	X	6	6136	0.4
	G	11	5679	0.4

2. Rock under investigation

The marble under investigation (Tuscany, Italy) is made up of calcite grains whose sizes range from 0.1 to 0.3 mm. It was formed by contact metamorphism from the “massive Limestone” formation. In the metamorphic orebody two microstructures are recognized: xenoblastic (with well interlocked grains of irregular shape) and granoblastic (with regular grains and triple points). Such textures are characterized by a different content of preexisting microcracks, mainly extending along the grains contacts. Moreover, they show a different response to the same stress field at the laboratory scale [21] (Table 1), even if the rock is almost monomineralic, with rare dolomite grains in the xenoblastic marble, and the grain size does not vary meaningfully with microstructure. In particular, as highlighted in a previous work [21], the highest mechanical properties are found in the xenoblastic marble which has a uniaxial compressive strength (UCS) and a Brazilian strength 1.4 times higher than the granoblastic marble (Table 1). The stress versus axial strain curve (Fig. 1a) of the granoblastic texture shows a marked downward curvature at the beginning, indicating a more intense microcracking, while in the xenoblastic marble the behavior is immediately almost linearly elastic. With the progressive closure of open microcracks inside the sample, the tangent Young's modulus of the granoblastic specimens initially increases with the stress up to a value corresponding to the beginning of the linear portion of the stress–strain curve, where, after the microcracks closure, microcracks have no more influence on the mechanical behavior of the rock until crack initiation and propagation [23]. In this portion of the stress versus strain curve, the elastic modulus is almost the same in the two textures ($E_{t,50}$ in Table 1). Afterwards, with increasing the applied stress, the tangent modulus of both textures gradually decreases as a result of the extension and coalescence of microcracks [23].

3. Experimental methodology

The multiscale experimental methodology is schematized in Fig. 1. First, analyses of grain boundaries cracks in the two marbles were conducted with Focused Ion Beam–Scanning Electron Microscopy (FIB–SEM). A FIB equipment uses a 4 nm wide focused beam of gallium ions (Ga+), working at low beam currents (of the order of pÅ) for imaging and at high beam currents (of the order of nÅ up to the μÅ) for site-specific controlled milling. By controlling the location, beam size and current density of the ion beam, material can be selectively removed from sub-micron areas. In this way, micro-cross-sections can be realized at surface features and the sub-surface microstructure can be observed with high resolution. In most of the new generation FIB equipment, the ion beam column is coupled with an electron beam column (SEM) in the same microscope, providing imaging of the ion beam milling process during the milling process itself or after it. In this case, the instrument is called Dual-beam (or cross-beam) microscope. The capability of the high-resolution imaging using both secondary electron and secondary ion signals has made the FIB microscope a unique imaging tool. In this work, high-resolution SEM observations of the grain boundaries in cross-section by FIB were performed on the investigated marble. In all cases, a thin Pt layer was deposited on the surface prior to FIB milling (using the FIB gun itself): a preliminary high ion current milling (9 nÅ) is followed by a cleaning step of the section (0.9 nÅ) until the desired section was obtained.

In order to study the interaction between indentations and microdiscontinuities, indentation tests were carried out on samples of granoblastic and xenoblastic marbles. Berkovich nanoindentations in CSM modality were performed inside calcite grains composing the two textures (xenoblastic and granoblastic). The testing was carried out with Agilent G200 Nano Indenter, equipped with the three-side pyramidal Berkovich diamond indenter. The CSM mode [32,33] lies in superimposing a small sinusoidal oscillation on the primary loading signal and analyzing the resulting response of the system. In this way, the hardness and the elastic modulus of the material can be recorded as a continuous function of the surface penetration depth by continuously measuring the dynamic contact stiffness. In the experimentation, the CSM modality was applied with a frequency of 45 Hz and a displacement amplitude of 2 nm. In each test, the indenter was loaded at a strain rate of 0.05 s^{-1} until reaching a maximum indentation depth of 1600 nm and 2000 nm inside the calcite grains of the xenoblastic and granoblastic marble, respectively. After reaching the maximum indentation depth, the load was kept constant for 15 s before unloading. Sixteen nanoindentations on each sample were carried out, randomly distributed on the tested surface. Series of Vickers indentations were carried out by applying a maximum load in the range 50 mN–20 N (from nano to macroscale), according to the following sequence: 50 mN, 60 mN, 70 mN, 80 mN, 90 mN, 100 mN, 110 mN, 120 mN, 130 mN, 140 mN,