Artificial microcracking of granites subjected to salt crystallization aging test

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Received: 21 August 2019 / Accepted: 25 June 2020 / Published online: 10 July 2020
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Abstract

Salt crystallization-induced decay of Vardavard granodiorite and Shirkouh monzogranite, two Iranian building stones, were assessed with two non-destructive methods: saturation-buoyancy technique and P- and S-wave velocity measurement. Moreover, polarized and fluorescence microscopy studies were used to evaluate the behavior of the studied stones at microscopic scale against a salt crystallization aging test. The aging test extended pre-existing microcracks and generated new ones. Intracrystalline microcracking was the most predominant microcrack type for both samples. Fine-grained Vardavard granodiorite experienced higher intercrystalline microcracking than coarse-grained Shirkouh monzogranite. The microcracking mechanism of feldspars substantially depends on their alteration degree and microstructural precursors. When a growing microcrack reaches a biotite, it propagates within the crystal if the growing microcrack coincides with the cleavage plane; otherwise, it propagates as an intercrystalline one. The increase in maximum microcrack length of the samples was higher than the increase in their mean microcrack length. Low-strength Vardavard granodiorite showed higher microcrack width after the aging test. Dry weight loss in low-strength Vardavard granodiorite was more pronounced than in high-strength Shirkouh monzogranite. Dry unit weight decreased at a higher rate than saturated unit weight with the increase of effective porosity. The reduction in ultrasonic wave velocities and the increment in effective porosity and water absorption were more pronounced for Vardavard granodiorite, indicating a higher degree of decay, i.e., higher microcrack generation, enlargement, and widening. Shirkouh monzogranite, which has large-sized crystals and pores, wider initial microcracks, high tensile strength, and low effective porosity and microcrack density, was more durable than Vardavard granodiorite.

Keywords Building stones · Microcracking · Salt crystallization · Non-destructive test · Durability

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Introduction

Granite is the most widely used intrusive igneous stone in construction with a broad range of different colors and compositional varieties. It has been quarried from ancient times to the present day for monuments and modern architecture purposes.

Durability of building stones plays a very important role in achieving sustainable construction. Granites are susceptible to weathering and exhibit signs of decay over time (Kim and Park 2003; Fort et al. 2013a; Heidari et al. 2013; Freire-Lista and Fort 2018). Dimension granites placed in buildings and monuments are prone to decay by salt crystallization (Chabas and Jeannette 2001; Begonha 2009). The salts contaminating building stones come from different sources, including seawater or groundwater, air pollutants, mortars, de-icing activities during wintertime, and animal droppings (Halsey et al. 1995; Gómez-Heras et al. 2004; López-Arce et al. 2009; Zalooli et al. 2019). Hydration and crystallization pressures inside stone pores are the two main mechanisms that explain stone deterioration by salt mechanical stress (Scherer 1999, 2004; Flatt 2002; Steiger 2005a, b). The crystallization of salts in a confined space (pore) occurs when a saline solution reaches supersaturation and the thermodynamic properties of the system are adequate for nucleation of the salt crystals (Gómez-Heras and Fort 2007; Flatt and Scherer 2008; Benavente et al. 2015). The growth of a salt crystal within the pore space of the stone will eventually lead to its microcracking and decay over time (Benavente et al. 2004, 2007; Angeli et al. 2007; Zalooli et al. 2017).

Accelerated salt crystallization test is a laboratory simulation of the natural conditions using extreme salt weathering. This is done to speed up the weathering process and to evaluate the durability of building stones. The mentioned test has been widely used due to its cost-effectiveness, technical simplicity, and similarity with the natural mechanical processes of stone decay (Positano et al. 2006). Despite the availability of different standards and guidelines such as EN-12370 (1999), RILEM (1980), and other recommendations, still there is no well-established testing and measurement protocol in this respect. One of the main disadvantages of the current standards is that the results obtained from the salt crystallization test, in terms of damage type and severity, are often different from those inferred from the field data. None of the current standard tests proposes an accurate method for monitoring decay progress during the aging test (Lubelli et al. 2014, 2018).

A number of researchers have attempted to investigate decay patterns of granitic stones induced by salt crystallization (Cardell et al. 2003; Sousa et al. 2017). Variations in physico-mechanical and surface properties of granitic stones due to salt crystallization test have been analyzed in the laboratory (Alonso et al. 2008; Silva and Simão 2009; López-Arce et al. 2010; Vázquez et al. 2013; Vázquez and Alonso 2015;

Momeni et al. 2015, 2017). The influence of some key parameters such as initial microcrack density and porosity (Sousa et al. 2005; Sousa 2014), pore size distribution (Rivas et al. 2010; Sousa et al. 2018), and initial physico-mechanical properties (Jamshidi et al. 2013) on durability of granitic stones against salt crystallization aging test has been investigated.

Non-destructive tests are gaining importance for the assessment of decay of building stones in recent years. Almost all non-destructive techniques are cheap and easy to conduct, and they yield reliable, detailed, and reproducible data of the stone decay (Hyun and Park 2011; Fais et al. 2017; İnce et al. 2018; Zhang et al. 2019). Granite, as a multimineralic rock, shows heterogeneity with respect to petrographic and physico-mechanical properties. The same specimen is measured prior to and during the aging test using the non-destructive tests (Siegesmund and Snethlage 2014). Ceryan et al. (2008) demonstrated that P wave velocity as a non-destructive test is the most reliable geotechnical measurement to evaluate the engineering characteristics of weathered granitic rocks.

Different microscopic approaches have been utilized to characterize decay in building stones, including optical, polarized and fluorescence microscopies, scanning electron microscopy (SEM), and confocal scanning laser microscopy (CSLM). These techniques provide clear evidence of the decay processes in the stones and the area where the decay is taking place (Benavente et al. 1999; Siedel and Siegesmund 2014; Zalooli et al. 2018a).

In the light of these considerations, the purpose of this study is to investigate salt crystallization-induced damage of two building granites: Vardavard granodiorite (Va-G) and Shirkouh monzogranite (Sh-M). The effect of initial physico-mechanical and petrographic characteristics, pore size distribution, and microcrack properties were taken into account in this study. Fluorescence-impregnated thin sections were applied to determine microcrack properties. The results from fluorescence microscopy technique were combined with those from mercury intrusion porosimetry test to provide complementary information about the microcracks. Non-destructive tests were used to obtain an accurate measure of the total damage induced by salt crystallization stress.

Materials and methods

Geological setting

Quarries of Va-G and Sh-M are located at 34°43'22" N, 48°20'01" E and 31°30'20" N, 53°58'45" E, respectively, in Alvand and Shirkouh mountains of Iran, to the southwest of Hamedan and Yazd cities, where examples of granite-built heritage can be found (e.g., Ganjnameh inscriptions and Bu-Ali Sina tomb) (Momeni et al. 2015).

Alvand batholith is one of the largest plutonic complexes in the Sanandaj–Sirjan metamorphic belt. It intrudes Jurassic sedimentary deposits and shows various petrographic facies including basic, intermediate, acidic, and ultramafic bodies.

All intrusions are generated during Jurassic–Cretaceous periods (Baharifar et al. 2004) (Fig. 1a).

The middle Jurassic Shirkouh batholith is located within the Central Iran zone. Shirkouh batholith exhibits three main petrographic facies as follows: granodiorite (northern part),

Fig. 1 Geological maps of the sampling areas. **a** Geological map of Alvand batholith (Momeni et al. 2015) and **b** Shirkouh batholith (simplified from Sheibi et al. 2012)

leucogranite (along the northwestern margin of the batholith), and monzogranite (Sheibi et al. 2012) (Fig. 1b).

Five fresh block specimens (each block had a size of approximately $30 \times 20 \times 20$ cm) without visible fractures were sampled. Cylindrical samples with a diameter of 54 mm (NX-size core) and length-to-diameter ratios of approximately 2.5, 1, and 0.5 were prepared by a laboratory water drilling machine. Sample ends were cut and polished parallel to each other and perpendicular to the sides using a low-speed diamond blade saw (120 rpm). These samples were used to determine the physico-mechanical properties of the stones before, during and after the accelerated weathering test.

Salt crystallization aging test

Sodium sulfate (Na_2SO_4) is frequently used to evaluate stone decay in laboratory-accelerated aging tests (Török and Přikryl 2010). The accelerated aging test was conducted for 30 cycles according to RILEM (1980). For one salt crystallization cycle, dry samples were totally immersed in a 14% Na_2SO_4 aqueous solution for 4 h, followed by drying at ambient temperature (20 °C) for 2 h. Samples were heated inside an oven at 100 °C for 16 h. Finally, they were air-cooled for 2 h. After every ten cycles, samples were washed with tap water of Tehran city to remove the salts fraction. The variation in physico-mechanical properties was measured after every ten cycles.

Petrography and chemical properties

Stone slices were cut from the fresh blocks and they were superficially impregnated using a fluorescence-dye epoxy resin under a vacuum pressure of 600 mmHg. Thin sections were made at low cutting speed (120 rpm). More than 40 images under a polarized microscope (PM) were captured, and these images were then stitched together using Photoshop software. Crystal size of the studied stones was measured using JMacrovision software. The boundaries of crystals were manually drawn. Two thin sections (one normal and another one parallel to the longitudinal axis of cylindrical samples) were examined to measure the Feret diameter and perimeter of each crystal. The Feret diameter is defined as the furthest distance between parallel tangents on the crystal (Ersoy and Waller 1995).

X-ray fluorescence (XRF) analyses were used for the determination of the elemental composition of fresh stones. Chips of each stone type were crushed into a fine powder and sieved through a sieve of 60 μm size. Oven-dried powders were examined in a Philips X'PERT MPD apparatus.

Physico-mechanical and pore properties

Eight cylindrical samples of each stone type with a length-to-diameter ratio of 2.5 were utilized to obtain physico-

mechanical properties before and after every ten cycles. Dry and saturated unit weights, water absorption by weight, and effective porosity of samples were measured using saturation buoyancy test according to ISRM (2007) (Fig. 2a).

P- and S-wave velocity measurements were carried out according to ISRM (2007) using a Pundit Lab ultrasonic tester. The lateral minimum dimension (perpendicular to the direction of propagation of the wave) must be at least 10 times the wavelength (ISRM 2007). Ultrasonic transducers, with a diameter of 36.77 and 44 mm and a frequency of 54 and 500 kHz, were used for the measurement of P- and S-wave velocities, respectively. The frequency of 54 kHz corresponds to a 6.4 mm wavelength. All samples had a diameter of 54 mm and a length of about 120 mm, which would be acceptable for ISRM standard. A gel-based couplant was applied between the transducers face and the stone surface to remove the air gap between transducers and specimen surface (Fig. 2b).

Uniaxial compressive strength (UCS) tests were conducted on four dry samples of each stone type with a height to diameter ratio of 2.5. Samples were loaded vertically with a constant load rate of 0.50–1.00 MPa/s. UCS tests were performed prior to accelerated weathering cycles (Fig. 2c).

To measure Brazilian tensile strength (BTS), three dry samples of each stone type with a thickness-to-diameter ratio of 0.5 were tested at a constant loading rate of 0.2 kN/s according to ISRM (2007) (Fig. 2d).

Mercury intrusion porosimetry (MIP) tests were applied for the determination of initial pore size distribution. A single prismatic oven-dried specimen with a length of 20 ± 2 mm and a diameter of 12 ± 2 mm was cut from each stone type. Samples were analyzed with a Micromeritics AutoPore IV 9520 porosimeter at maximum pressure of 414 MPa. Pore diameter higher than 5 μm was described as macroporosity, and pore diameter smaller than 5 μm was described as microporosity (Fig. 2e).

Microcracks characterization

Four thin sections of each stone type were examined under an Olympus Bx 51 polarized light microscope equipped with an Olympus ultraviolet lamp and attached to an Olympus DP (6 V/2.5 Å) digital camera running on (version 3.2) DP-Soft Olympus software (Fig. 2f). Applying an ultraviolet lamp, a petrographic microscope operates in fluorescence mode and fluorescent epoxy-filled microcracks are identified. More than 40 images both under the polarized microscope (PM) and the fluorescence microscope (FM) at the same magnification ($\times 40$) were photographed. PM and FM micrograph mosaics were made from the thin sections of each sample. The FM mosaic was laid over the same PM mosaic to generate a 1.5×2 cm grid. An area of about 12×16 mm of each thin section was divided into a grid of 4×4 mm squares. Microcracking was quantified by counting the number of

Fig. 2 Laboratory setups for determination of different physico-mechanical, petrographic, and microcrack properties. **a** Vacuum saturation equipment, **b** Pundit Lab ultrasonic tester, **c** uniaxial

compressive strength tester, **d** Brazilian tensile strength tester, **e** mercury intrusion porosimeter, and **f** polarized light microscope equipped with an Olympus ultraviolet lamp

microcracks that intersected with the sides of these squares (Fig. 3). Linear microcrack density (LMD) (Sousa et al. 2005) was measured as the number of microcracks per

Fig. 3 Thin section of Shirkouh monzogranite (Sh-M) after the 30th cycle. FM micromosaic of microphotographs overlaid on PM micromosaic of microphotographs and 16×12 mm rectangle divided into 4×4 mm squares

millimeter. Microcracks were analyzed before the aging test and at the end of every ten cycles.

Microcracks were manually traced on prepared micromosaics using JMacrovision software and their lengths were calculated by measuring the distance between their end points. When the micromosaics were magnified to measure the width of microcracks, they got pixelated and the image quality was decreased. To solve it, several FM images with a dimension of 1024×768 pixels were analyzed using JMacrovision software. The mean values of microcrack length and width as well as distribution of microcrack length and width were characterized prior to and after the aging test.

Results

Petrographic and chemical properties

Va-G is a inequigranular, fine- to medium-grained granodiorite consisting of plagioclase (Pl) ($\sim 45\%$ vol., $1.92 \pm$

0.71 mm), k-feldspar (Kfs) ($\sim 20\%$ vol., 1.86 ± 0.69 mm), quartz (Qz) ($\sim 25\%$ vol., 1.26 ± 0.58 mm), amphibole (Am) ($\sim 8.5\%$ vol., 0.29 ± 0.10 mm), and biotite (Bt) ($\sim 1.5\%$ vol., 0.66 ± 0.31 mm). Pl crystals are subidiomorphic and they are altered to sericite in the center (Fig. 4a, a' and a''). Kfs crystals are subidiomorphic–allotriomorphic with banded and patched perthites. They show a slight undulating extinction. The mean Feret diameter was 1.28 ± 0.78 mm. Crystals' perimeter varied from 0.98 to 6.06 mm with an average of 3.49 ± 1.70 mm.

Sh-M is a subidiomorphic, equigranular, medium- to coarse-grained monzogranite containing Qz ($\sim 50\%$ vol., 2.49 ± 0.91 mm), Pl ($\sim 25\%$ vol., 2.52 ± 1.08 mm), Kfs ($\sim 10\%$ vol., 1.93 ± 0.51 mm), and Bt ($7\text{--}15\%$ vol., 2.03 ± 1.01 mm) (Fig. 4b, b' and b''). Pl crystals are idiomorphic and zonal and exhibit albite twinning. Furthermore, some Pl crystals are altered. Qz crystals occur as anhedral ones, interstitial to other minerals, and heterometric. Bt crystals appear as isolated ones. This sample has coarser sized crystals than Va-G; its mean crystal Feret diameter was 2.02 ± 0.92 mm. The crystals' perimeter varied from 1.27 to 8.42 mm with an average of 5.95 ± 1.39 mm.

From the chemical analyses conducted on the powder samples, it was determined that Va-G has higher loss on ignition (LOI) in comparison with Sh-M. This might be due to the fact that Va-G has more light elements which are undetectable with this technique. SiO_2 is the major oxide of both samples,

and Sh-M possesses a higher amount of SiO_2 than Va-G. By contrast, Va-G exhibits a higher content of Al_2O_3 than Sh-M (Table 1).

Physico-mechanical properties

Mean initial values of physico-mechanical properties of the studied samples are listed in Table 2. Fresh Va-G exhibits lower unit weights, P wave velocity, and UCS than Sh-M. In contrast, Va-G shows higher effective porosity, water absorption, and S-wave velocity than Sh-M.

Initial pore size distribution of the studied samples is illustrated in Fig. 5. According to MIP results, initial porosities are 1.06 and 0.53% for Va-G and Sh-M, respectively. Mean pore diameters are 0.17 and 0.16 μm for Va-G and Sh-M, respectively. Although the samples have relatively identical mean pore size, micropores constitute 74.5 and 58% of total MIP for Va-G and Sh-M, respectively.

Variation of normalized physico-mechanical properties during the salt crystallization aging test are plotted in Fig. 6. After the aging test, Va-G had a steeper reduction in dry unit weights than Sh-M. A dry unit weight loss of 2.85% in Va-G and of 0.95% in Sh-M and a saturated unit weight reduction of 1.23% in Va-G and of 0.67% in Sh-M occurred after the aging test. The reduction in dry unit weight was more pronounced than the loss in saturated unit weight for the studied stones

Fig. 4 **a** Vardavard granodiorite (Va-G) polished surface **a'** and **a''** thin sections under polarized microscope. **b** Shirkouh monzogranite (Sh-M) polished surface. **b'** and **b''** thin sections under polarized microscope. Amphibole (Am), biotite (Bt), K-feldspar (Kfs), plagioclase (Pl), and quartz (Qz)

Table 1 Chemical composition (oxides content) of fresh samples obtained by XRF test

Sample	LOI (%)	SiO ₂ (%)	Al ₂ O ₃ (%)	K ₂ O (%)	CaO (%)	Na ₂ O (%)	Fe ₂ O ₃ (%)	MgO (%)	TiO ₂ (%)	P ₂ O ₅ (%)	Rb + Sr + Zr (%)
Va-G	0.76	68.44	15.16	4.54	3.63	2.68	2.92	1.01	0.43	0.26	0.17
Sh-M	0.38	70.89	14.48	4.7	2.4	3.57	2.3	0.6	0.23	0.18	0.27

LOI, loss on ignition; Va-G, Vardavard granodiorite; Sh-M, Shirkouh monzogranite

(Fig. 6a). Va-G had more significant dry weight loss than Sh-M. Va-G showed no remarkable variation in dry weight for the first 20 test cycles (a decrease of 0.39%), while the final reduction was found to be 5.5%. Sh-M showed 0.39% loss in dry weight after the aging test (Fig. 6a).

Effective porosity and water absorption of the studied stones rose as decay progressed. After the 20th cycle, an abrupt rise in these parameters occurred in both samples, particularly in Va-G. After the salt crystallization aging test, the increase in effective porosity and water absorption was higher in Va-G (a mean increase of 598% and 525%, respectively) than in Sh-M (a mean rise of 202% and 193%, respectively) (Fig. 6b).

Reduction in P- and S-waves velocities was higher for Va-G than for Sh-M after the aging test. P- and S-wave velocities were reduced by 75.64% and 71.36% for Va-G, 26.58% and 28.02% for Sh-M at the end of the 30th cycle (Fig. 6c).

Microcracks characterization

The results of the microcracks study are listed in Table 3 and illustrated in Figs. 7 and 8. The aging test induced intra-, inter- and trans-crystalline microcracks. Total LMD increased after the aging test. In Va-G, it increased from 1.97 N/mm (microcrack number/mm) in fresh samples to 4.85 N/mm in aged samples; in Sh-M, it rose from 1.07 before the aging test to 3.65 N/mm after the aging test.

In fresh Va-G, intracrystalline microcracks were the most frequent microcrack type. Intracrystalline LMD of Qz was the highest, followed by Kfs, Pl, Am, and Bt. Initial transcrystalline microcracks were infrequent. Kfs had the highest increase in intracrystalline microcracking due to the salt crystallization test, followed by Qz, Pl, Bt, and Am. The mean intracrystalline LMD increased until the 20th cycle when their LMD decreased slightly. Intercrystalline

microcracking was the most frequent microcracking type at the end of the aging test. The rise of intercrystalline microcracking in the minerals was in the following order: Qz > Kfs > Pl, > Am > Bt. Regarding transcrystalline microcracking, Qz experienced the highest, followed by Pl, Kfs, Am, and Bt. A steep rise in transcrystalline microcrack density occurred after the 20th cycle. However, the increase in transcrystalline microcracking was not as high as the increase in inter- and intra-crystalline microcracks (Table 3 and Fig. 8a).

In Sh-M, intracrystalline microcracks had the highest LMD than inter- and trans-crystalline microcracks prior to and after the aging test. Transcrystalline microcracks were the least common in fresh Sh-M. After the aging test, Pl had the highest rise in intracrystalline microcracking, followed by Qz, Bt, and Kfs. A steep rise in intracrystalline microcracking was recorded after the 10th cycle. The rise in intercrystalline microcracking affected the crystals in the following order: Qz, Pl, Bt, and Kfs. Fresh and weathered Sh-M had lower intercrystalline microcrack density than Va-G. Qz showed the highest transcrystalline microcracking after the aging test, followed by Pl, Bt, and Kfs (Table 3 and Fig. 8b). Aged Sh-M had lower transcrystalline microcrack density in comparison with aged Va-G (Fig. 8a, b).

In Va-G, the mean width of pre-existent microcracks was measured at $7.67 \pm 2.99 \mu\text{m}$, and after the aging test, it reached $23.96 \pm 25.14 \mu\text{m}$. Microcracks in fresh and weathered Sh-M had a mean width of 9.06 ± 4.02 and $16.69 \pm 18.56 \mu\text{m}$, respectively. In other words, the mean width of microcracks jumped to triple and double the initial values in Va-G and Sh-M, respectively. Initial microcracks with a width of less than $10 \mu\text{m}$ constituted 71% of microcracks in Va-G. More than 80% of microcracks had a width of more than $10 \mu\text{m}$ after the aging test. The majority of wide microcracks (more than $10 \mu\text{m}$) were $10\text{--}30 \mu\text{m}$ in width (Fig. 8c). In fresh Sh-

Table 2 Mean values and standard deviation of some physico-mechanical properties of fresh samples

Sample	γ_d (kN/m ³)	γ_{sat} (kN/m ³)	n_e (%)	W_a (%)	V_p (m/s)	V_s (m/s)	UCS (MPa)	BTS (MPa)
Va-G	26.28 ± 0.14	26.39 ± 0.11	1.03 ± 0.27	0.39 ± 0.10	4340 ± 124	2266 ± 73	124.25 ± 6.59	11.05 ± 2.28
Sh-M	26.75 ± 0.21	26.84 ± 0.21	0.83 ± 0.22	0.31 ± 0.08	5025 ± 85	2159 ± 19	145.80 ± 8.71	13.02 ± 2.69

Va-G, Vardavard granodiorite; Sh-M, Shirkouh monzogranite; γ_d , dry unit weight; γ_{sat} , saturated unit weight; n_e , effective porosity; W_a , water absorption by weight; V_p , P-wave velocity; V_s , S-wave velocity; UCS, uniaxial compressive strength; BTS, Brazilian tensile strength

Fig. 5 Initial pore-size distribution of Vardavard granodiorite (Va-G) and Shirkouh monzogranite (Sh-M)

M, microcracks with a width of less than 10 μm accounted for 59% of the total microcrack number. Although Va-G had the lower mean initial microcrack width in comparison with Sh-M, Va-G experienced more opening in microcracks after the aging test (Fig. 8d).

The mean length of microcracks increased in both samples: in Va-G, it rose from 1.54 ± 1.18 in fresh samples to 2.28 ± 2.24 mm in aged specimens; in Sh-M, it increased from 2.35 ± 1.96 before to 2.70 ± 2.36 mm after the test. In Va-G, the maximum microcrack length jumped to triple the initial values (from 4.62 to 12.52 mm), while it rose from 6.79 to 8.84 mm in Sh-M. The mean length of intracrystalline microcracks decreased after aging test: in Va-G, from 0.65 ± 0.28 before to 0.56 ± 0.25 mm after the test; in Sh-M, it decreased from 0.98 ± 0.57 before to 0.89 ± 0.53 mm after the test. The mean length of intercrystalline microcracks rose in Va-G and Sh-M (from 2.06 ± 0.77 to 3.26 ± 0.45 mm and 3.90 ± 1.06 to $5.06 \pm$

Fig. 6 Physico-mechanical properties evolution of Vardavard granodiorite (Va-G) and Shirkouh monzogranite (Sh-M) with salt crystallization cycles. **a** Dry and saturated unit weights and dry weight loss (DWL), **b** effective porosity and water absorption, and **c** P- and S-waves velocities

Table 3 Mean LMD of total, inter-, intra-, and trans-crystalline microcracks before and during salt crystallization cycles by type of minerals affected

Sample	Mineral	Intracrystalline LMD (N/mm)				Intracrystalline LMD (N/mm)				Intracrystalline LMD (N/mm)				Total LMD (N/mm)			
		0	10	20	30	0	10	20	30	0	10	20	30	0	10	20	30
Va-G	Qz	0.42	0.53	0.73	0.56	0.3	0.37	0.48	0.51	0.03	0.07	0.1	0.35	0.75	0.97	1.31	1.42
	Pl	0.2	0.28	0.43	0.4	0.17	0.22	0.33	0.44	0.06	0.08	0.14	0.16	0.43	0.58	0.90	1.00
	Kfs	0.35	0.41	0.68	0.73	0.31	0.35	0.46	0.57	0.03	0.03	0.08	0.23	0.69	0.79	1.22	1.53
	Bt	0.01	0.09	0.16	0.17	0.03	0.07	0.12	0.19	0	0	0.04	0.07	0.04	0.16	0.32	0.43
	Am	0.03	0.06	0.1	0.16	0.03	0.09	0.17	0.22	0	0	0.02	0.09	0.06	0.15	0.29	0.47
	Total	1.01	1.37	2.1	2.02	0.84	1.1	1.56	1.93	0.12	0.18	0.38	0.9	1.98	2.65	4.04	4.85
Sh-M	Qz	0.31	0.34	0.59	0.89	0.12	0.16	0.3	0.4	0.03	0.07	0.1	0.17	0.46	0.57	0.99	1.25
	Pl	0.23	0.29	0.66	0.24	0.15	0.18	0.31	0.38	0	0	0.06	0.11	0.38	0.47	1.03	1.38
	Kfs	0.04	0.1	0.2	0.28	0.07	0.11	0.16	0.19	0	0	0	0.03	0.11	0.21	0.36	0.46
	Bt	0.04	0.08	0.15	2.09	0.08	0.14	0.16	0.22	0	0.01	0.03	0.06	0.12	0.23	0.34	0.56
	Total	0.62	0.81	1.6	0.68	0.42	0.59	0.93	1.19	0.03	0.08	0.19	0.37	1.07	1.48	2.72	3.65

Va-G, Vardavard granodiorite; *Sh-M*, Shirkouh monzogranite; *LMD*, linear microcrack density; *N/mm*, microcrack number per millimeter; *Qz*, quartz; *Pl*, plagioclase; *Kfs*, K-feldspar; *Bt*, biotite; *Am*, amphibole

1.46 mm, respectively). In *Va-G*, the mean length of transcrystalline microcracks jumped to double the initial values after the test (from 4.62 ± 0.27 to $8.05 \text{ mm} \pm 3.04$). In *Sh-M*, the mean length of transcrystalline microcracks rose from 5.02 ± 0.53 before to 7.42 ± 0.38 mm after the test (Fig. 8e, f).

Discussion

Effective porosity of granites is mainly due to microcracks. *Va-G* had higher initial effective porosity

because it had the greater initial intercrystalline microcrack density. Water absorption is strongly dependent on effective porosity (Zalooli et al. 2018b). Having higher effective porosity, fresh *Va-G* exhibited high water absorption. The initial effective porosity value obtained from the saturation buoyancy test was close to that obtained from the mercury porosimetry test for *Va-G*. This is an indicative of efficient transport of water between micropores and macropores (Rivas et al. 2010). The low UCS and BTS values of fresh *Va-G* was attributed to its higher effective porosity, higher

Fig. 7 Polarized petrographic micromosaics overlaid on fluorescence micromosaics; *Va-G* Vardavard granodiorite, *Sh-M* Shirkouh monzogranite, *Am* amphibole, *Bt* biotite, *Kfs* K-feldspar, *Pl* plagioclase, and *Q* quartz

Fig. 8 Microcrack measurement during the aging test. **a, b** Variation in linear microcrack density (LMD) of intra-, inter-, and trans-crystalline microcracks in Vardavard granodiorite (Va-G) and Shirkouh monzogranite (Sh-M); **c, d** distribution of microcracks width of Va-G

and Sh-M prior to and after the aging test and **e, f** distribution of microcracks length of Va-G and Sh-M pre- and post-aging test, respectively. Note that the length and width of microcracks were measured in millimeters (mm) and micrometers (μm), respectively

LMD, and lower Qz/Pl + Kfs ratio. Moreover, high content of easily cleavable crystals (Pl and Kfs) reduced the initial mechanical strength of Va-G.

Texture (crystal size and orientation) and mineralogical composition play an important role when it comes to stone weathering. Martins et al. (2016) expressed that coarse

granites are more prone to decay by the salt crystallization. López-Arce et al. (2010) mentioned that coarser biotite and feldspar crystals in granites allow greater salt solution penetration and crystallization within these minerals cleavage planes. In this study, fine-grained Va-G was more prone to salt-induced decay than coarse-grained Sh-M. This might be due to the fact that fine-grained Va-G had a higher number of intercrystalline microcracks (intercrystalline boundary) prior to and after the aging test. Emmanuel and Levenson (2014) postulated that a coarse-grained stone has a low number of crystal boundaries relative to a stone with small crystals. It seems that salt crystals deposit within intercrystalline boundaries in Va-G and cause grain detachment.

Both pre-existent and salt crystallization-induced microcracks of Qz generated in different directions due to the lack of cleavage or other weakness planes. In fresh samples, Qz had the highest LMD of intracrystalline microcracks. The higher LMD of intracrystalline microcrack within Qz crystals in fresh samples probably has a relation with thermal stresses produced during cooling. When a granitic magma cools down, Qz experiences more remarkable volume change (Nur and Simmons 1970). The higher number of intracrystalline microcracks within Qz crystals of the studied samples was attributed to the thermal stresses produced during magma cooling. After the salt crystallization aging test, the Qz had the highest number of transcrystalline microcracks among other minerals. Salt crystallization pressure coalesced pre-existing intracrystalline microcracks and linear density of transcrystalline microcracks remarkably grew in Qz crystals.

Almost all microcracks within Bt generated along the cleavage plane. In both samples, Bt dictated the path for microcrack propagation (Fig. 9a, b). The growing microcracks propagate within the Bt and act as transcrystalline ones if they coincide with the Bt cleavage plane (red arrows in Fig. 9b). This was more obvious in the larger-sized Bt of Sh-M due to its perfect cleavage planes. Microcracks terminated, deviated, or propagated as intercrystalline ones where they interacted with the parallel crystallographic c-axis of Bt but did not coincide with the cleavage plane (yellow arrows in Fig. 9a, b). When microcracks interacted with the oblique or normal c-axis of Bt, they propagated as intercrystalline ones (blue arrows in Fig. 8a, b). Vázquez et al. (2016) showed that Bt deviates microcracks. If microcracks interact with the perpendicular cleavage planes of Bt, they terminate or strongly deviate. According to our results, a growing microcrack propagates into a Bt when it coincides with the cleavage plane. Previous studies have shown that Bt tend to deform more than Qz, Pl, or Kfs since Bt has a lower elastic modulus and a higher surface energy in comparison with the more brittle Qz, Pl, and Kfs (Ündül et al. 2015; Freire-Lista et al. 2016).

Unaltered Pl and Kfs (Pl_u and Kfs_u) showed perfect cleavage in the (001) plane and good cleavage in the (010) plane (Fig. 9a, b). Microcracking of Pl and Kfs is strongly

influenced by their alteration degree, especially in Sh-M. Lower LMD was recorded within altered Pl and Kfs (Pl_a and Kfs_a) (Fig. 8a, b). Pre-existent intracrystalline microcracks were not observed in Pl_a and Kfs_a , whereas Pl_u and Kfs_u exhibited perfect cleavage in the (001) and (010) planes. Salt crystallization-induced stress caused the extending and widening of pre-existent cleavage planes in Pl_u and Kfs_u . Pl_a and Kfs_a were more resistant against salt crystallization and tended to be unaffected (compare the degree of microcracking within Pl_u and Kfs_u with Pl_a and Kfs_a , respectively in Fig. 9a, b). It seems that the stone resistance to brittle deformation can increase to some extent by alteration processes. Cleavage planes act as weakness surfaces and salt can precipitate within them to exert pressure. Alteration destroys cleavage planes of fresh samples; consequently, altered crystals do not have these weakness planes. Moreover, it is well documented that weathering reduces the deformation modulus of materials and modifies microcrack generation as a result of more plastic behavior of altered crystals (Freire-Lista and Fort 2017). The presence of soft and flexible clay minerals can increase the resistance of Pl and Kfs to brittle deformation. Pressure induced by salt crystallization generates zones of high stress on the boundaries between low-stiffness altered crystals and neighboring stiff ones, resulting in the formation intercrystalline microcracking.

Compositional zoning is another factor influencing the microcracking mechanism within zoned Pl (Pl_z) (Freire-Lista et al. 2015a). A concentric microcracking pattern was observed in Pl_z (red dashed lines in Fig. 9a, b).

Generally, the preferred orientation of crystals dictates the propagation path for a growing microcrack (Özvan et al. 2015; Martínez-Martínez et al. 2018). As shown in Fig. 9b, the crystallographic orientation of Bt, Pl, and Qz (dashed light green line) dictated the path for growing microcracks (dashed dark green line).

In Va-G, linear density of intracrystalline microcracks decreased slightly after the 20th cycle since transcrystalline microcracks formed by linking of intracrystalline microcracks. Although Sh-M had a lower number of initial intracrystalline microcracks than Va-G, this type of microcracks were the dominant microcracking type in Sh-M during the aging test. Having larger-sized crystals, Sh-M was more prone to intracrystalline microcracking. Previous studies have shown that the perfect cleavage planes in coarser-sized crystals (Pl, Kfs, and Bt) facilitated microcracking (Yilmaz et al. 2009). Fine-grained Va-G had a higher number of intercrystalline microcracks before and after the aging test than coarse-grained Sh-M. Crystal size mainly influences the ratio of inter- to intra-crystalline microcracks in granites (Sousa 2013; Ghasemi et al. 2020).

Intra- and inter-crystalline microcracks had a lower mean length in Va-G than in Sh-M before and after the test because Va-G had smaller crystals than Sh-M. Generally,

Fig. 9 PM micromosaics overlaid on FM micromosaics at the 20th salt crystallization cycle; **a** Vardavard granodiorite (Va-G) and **b** Shirkouh monzogranite (Sh-M). Red arrow, transcrystalline microcracks within biotite (Bt); blue arrow, the growing microcrack does not coincide with the cleavage plane of biotite (Bt); yellow arrow, microcrack interacts with

oblique or normal cleavage plane of biotite (Bt); green dashed line, orientation of microcracks parallel to c-axis of biotite (Bt) and plagioclase (Pl); Pl_a, altered plagioclase; Pl_u, unaltered plagioclase; Pl_z, zoned plagioclase; Kfs_a, altered K-feldspar; Kfs_u, unaltered K-feldspar

intracrystalline microcracks are smaller than a crystal diameter in length, while the length of intercrystalline ones is approximately equal to the crystal perimeter. Salt crystallization-induced intracrystalline microcracks were growing into one

other, generating a mesh of microcracks within the crystals and that was why the mean length of intracrystalline microcracks decreased after the aging test. Intercrystalline microcracks were incomplete in fresh samples and they

propagated due to salt crystallization pressure. After the aging test, the mean length values of intercrystalline microcracks were relatively similar to the mean values of the crystal's perimeter. The aging test led to an increase in mean total length of microcracks. The low increase in the mean total length of microcracks of Sh-M is explained by the prevalence of intracrystalline microcracks over other types of microcracks in this stone. The higher increase in maximum microcrack length in Va-G was due to generation of transcrystalline microcracks and also to the high intercrystalline microcrack number.

In Va-G, the width of microcracks increased considerably after the aging test. In other words, the widening of existing microcracks was more significant in Va-G than in Sh-M. It seems that salt crystals enter within intercrystalline microcracks and insert crystallization pressure against microcracks' walls in Va-G. This stone had lower mechanical strength, so salt crystallization pressure more easily overcame the stone strength and widened microcracks. The salt crystallization test induced greater increase in the mean width than in the mean length.

Micropores are important in solution absorption in the submerging-drying type of salt crystallization test (Rivas et al. 2010). Moreover, they determine the degree of decay of a stone subjected to salt crystallization cycles (Benavente et al. 2004). Assuming a uniform distribution of salt crystals in the pores, a higher amount of salt must precipitate in Sh-M with larger sized pores than in Va-G with smaller sized pores, to generate the same pressure. That means that, under the same conditions of a salt crystallization test, the loading surface (contact surface between salt crystal and pore walls) is larger in Va-G than in Sh-M, resulting in higher crystallization stress. Considering the high microporosity of Va-G, salt crystallization pressure was high enough to cause substantial microcracking and to widen pre-existent microcracks. Moreover, Va-G had higher effective porosity, and the salt crystallization pressure caused intense microcracking. Moreover, pores and microcracks add a dual property to the petrophysical characteristics of the stones, conferring them double porosity behavior. Fresh Va-G had narrower microcracks (lower width) than Sh-M. It seems that narrow microcracks are important in the aging test due to the effectiveness of salt crystallization pressure.

Effective porosity (the interconnected pore volume) of a stone is one of the main parameters that both controls and is influenced by weathering (Ballesteros et al. 2011). The so-called "weathering porosity" of building stones is a type of secondary porosity (Siegesmund and Dürst 2014). During the aging test, both stones showed an exponential increase in effective porosity with a parallel increase in the number of inter- and trans-crystalline microcracks. This is due to the fact that effective porosity is strongly influenced by the inter- and trans-crystalline microcracks. This is in good agreement by

Freire-Lista et al. (2015b). The interconnection between given microcrack (pore) networks is a function of microcrack density and microcrack length (Long and Witherspoon 1985). A direct relationship between effective porosity and microcrack density has been reported for granitic stones (Griffiths et al. 2017). Both stone showed an increase in LMD and length of microcracks as the number of salt crystallization cycles increased. Va-G underwent the greater increase in LMD after the aging test. That, and the fact that the mean microcrack length values were higher in weathered Va-G than in weathered Sh-M, can explain the higher values for effective porosity and therefore the greater decay in the former stone.

An increase in effective porosity resulted in a parallel increase in the water absorption during salt crystallization aging cycles. The water absorption ability of a stone is strongly dependent on its effective porosity, pore (microcrack) size distribution, and the mineralogical components (Siegesmund and Dürst 2014). Aging tests resulted in high effective porosity values combined with an effective interconnectivity between the microcracks, and consequently, water absorption rose in the samples.

Dry and saturated unit weights of the studied samples decreased after the aging test. Dry and saturated unit weights of granites with low porosity depend almost exclusively on chemical and mineralogical compositions of these rocks (Siegesmund and Dürst 2014). Salt crystallization causes the disintegration of rocks without significant chemical change (Alves et al. 2020). Va-G showed higher reduction in dry and saturated unit weights than Sh-M during the aging tests, particularly after the 20th cycle. The explanation for this finding is that the mean microcrack length and width, as well as LMD values, increased greater in Va-G than in Sh-M.

Both samples exhibited less tangible loss in dry weight up to 20 cycles; after that, the reduction in dry weight was much more pronounced for Va-G. Weight loss occurs when the stress induced by salt crystallization exceeds the cohesion threshold of the stone. When crystallization pressure is insufficient to reach the cohesion threshold, there is no grain loss, although other deterioration processes such as development and enlargement of microcracks may have occurred (Benavente et al. 2007; Zalooli et al. 2018a). Uniaxial compression strength and Brazilian tensile strength reflect these cohesive forces at a macroscopic scale. The uniaxial compression strength and Brazilian tensile strength values of Va-G are lower than those of Sh-M. Salt crystallization pressure widened microcracks more easily in low-strength Va-G than in high-strength Sh-M. Generally, mechanical strength of the stone decreases as LMD, the width, and length of microcracks increase. Granular disintegration was the most observable decay pattern in Va-G. This type of decay pattern occurs due to the widening of inter- and trans-crystalline microcracks. The higher reduction in dry

weight for Va-G was attributed to its higher intercrystalline microcracking and widening of microcracks, higher porosity, and microporosity as well as to its lower mechanical strength.

Wave velocity measurement is a non-destructive method and it is increasingly used in characterization of the degree of decay in stones (Fort et al. 2013b). It can be used as an indicative for the evaluation of initial mechanical strength and the salt weathering resistance of the stone (Basu and Aydin 2006; Mishra and Basu 2012; Jamshidi et al. 2017). Ultrasonic wave velocity is directly related to the mechanical strength of stone and it is inversely proportional to the decay of stone and consequently to its porosity. Changes in microstructure influenced P- and S-wave propagation. P- and S-wave velocities decreased exponentially with increasing number of salt crystallization cycles. The reduction in P- and S-wave velocities can be explained by the decrease in the cohesion and a corresponding decrease in the elastic coupling between the different crystals of the stones. A decrease in P- and S-wave velocities corresponds to an increase in the effective porosity, which in turn is a function of microcrack density and length. An inverse relationship between ultrasound velocity and LMD has been reported for granitic stones (Sousa et al. 2005; Vázquez et al. 2010). Increase of microcracks lengthened the propagation time of P- and S-wave to pass through the samples and thus reduced P- and S-wave velocities. The greater reduction in P- and S-wave velocities recorded for Va-G than Sh-M denotes a greater degree of deterioration, i.e., higher microcrack generation, enlargement, and widening in the former. This is consistent with the initially greater effective porosity, water absorption, and LMD values in Va-G and the steeper increase in these parameters after salt crystallization test.

Conclusions

The study involved an assessment of microcracking of Va-G and Sh-M against a sodium sulfate crystallization aging test. Based on the laboratory assessment performed in this study, the following conclusions arise:

- Crystal size is a key parameter controlling microcracking. Having larger sized crystals, Sh-M was more prone to intracrystalline microcracking. Fine-grained Va-G experienced higher intercrystalline microcracking than coarse-grained Sh-M.
- The mean length of intracrystalline microcracks decreased after the aging test because salt crystallization generated a mesh of microcracks within the crystals. The mean length of inter- and transcrystalline microcracks increased because of the propagation and coalescence of pre-existing ones. The increase in the mean width of microcracks was

higher than the increase in the mean length of microcracks for both samples.

- Intracrystalline microcracks within Pl, Kfs, and Bt are influenced by microstructures such as cleavage plane and compositional zonation. Moreover, chemical alteration of Pl and Kfs crystals modifies their microcracking. Intercrystalline microcracking was the most predominant microcrack type in Bt because this mineral has a lower elastic modulus and a higher surface energy than Qz and feldspar. The highest number of initial intracrystalline microcrack within Qz was attributed to the thermal stresses produced during magma solidification. Salt crystallization coalesced intracrystalline microcracks and LMD of transcrystalline microcracks remarkably grew.
- Porosity and pore size distribution, mechanical strength, and microcrack properties were the most important characteristics controlling the durability of the studied stones. Va-G experienced higher damage during the aging tests because it had the higher initial microporosity, intercrystalline microcrack density, effective porosity, and lower mechanical strength than Sh-M
- An increase in microcrack length and LMD yields an increase in degree of microcracks interconnectivity, thus increasing effective porosity and water absorption. Weathered Va-G had the higher effective porosity and water absorption than weathered Sh-M because the former one had the microcrack network with higher degree of interconnectivity.
- Dry and saturated unit weights evolution during the salt crystallization aging test affecting the studied samples was linked to effective porosity modifications.
- The reduction in dry weight was higher in Va-G because it had lower initial strength, smaller pore size, and higher intercrystalline microcrack number. Moreover the reduction in dry unit weight was more intense than the reduction in saturated unit weight in Va-G.
- Changes in microstructure influenced P- and S-wave velocities. Both stones undergo an exponentially growth in the effective porosity, water absorption, inter-, and transcrystalline microcrack number with a parallel decline in P- and S-wave velocities during the aging test. The greater reduction in P- and S-wave velocities recorded for Va-G than Sh-M denotes a greater degree of deterioration, i.e., higher microcrack generation, enlargement, and widening in the former.

Acknowledgments Authors are grateful to Pedro Vicente Lozano Carrasco, a laboratory technician of the Petrology and Geochemistry department of Complutense University of Madrid for his help in preparing fluorescence impregnated thin sections. Authors wish to thank Cristian Zapatero Martín, a member of the Petrophysical Laboratory of IGEO (CSIC, UCM) for doing MIP tests. We thank Alfonso Chao for English revision.

Funding information This study is financially supported by Tarbiat Modares University of Tehran (Iran), the Community of Madrid (Spain) under the GEOMATERIALS-2CM (S2013/MIT-2914) and TOP HERITAGE-CM (S2013/MIT-2914 and S2018/NMT-4372) programs and Fundação para a Ciência e a Tecnologia of Portugal (FCT) CEECIND/03568/2017. The Ministry of Science and Technology of Iran financed a six-month stay of the first author in Geosciences Institute IGEO (CSIC, UCM) of Madrid (Spain) to perform this research.

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