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Procedia Structural Integrity 33 (2021) 966–981

Structural Integrity Procedia

www.elsevier.com/locate/procedia

IGF26 – 26th International Conference on Fracture and Structural Integrity

Influence of rock inclusion composition on the fracture response of cement-based composite specimens

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Abstract

This paper concerns the results of research into the influence of the composition of rock inclusions on the fracture response of cement-based composite specimens. Specially designed specimens of the nominal dimensions 40 × 40 × 160 mm with inclusions in the shape of prisms with nominal dimensions of 8 × 8 × 40 mm were provided with an initial central edge notch with a depth of 12 mm. These specimens, which were made of fine-grained cement-based composite with different types of rock inclusion – amphibolite, basalt, granite, and marble – were tested in the three-point bending configuration. Fracture surfaces were examined via scanning electron microscopy and local response in the vicinity of rock inclusions was characterized via the nanoindentation technique. The aim of this paper is to analyse the influence of the chemical/petrographic composition of rock inclusions on the effective mechanical fracture parameters of cement-based composites, as well as on the microstructural mechanical parameters of the interfacial transition zone. The results of this research indicate the significant dependence of the effective fracture parameters on the petrographic and related chemical composition of the rock inclusions.

Peer-review under responsibility of the scientific committee of the IGF ExCo

Keywords: Cement-based composite; Force–displacement diagram; Fracture test; Inclusion; Mechanical fracture parameters; Rocks.

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Peer-review under responsibility of the scientific committee of the IGF ExCo
10.1016/j.prostr.2021.10.107

1. Introduction

Cement-based composites, with concrete being the main representative of such composites, are widely used building materials (Aïtcin, 1998), (Neville, 2011). Concrete structures such as highway bridges, tunnels, dams, etc. are important parts of the infrastructure which should serve for many generations after their construction. In many cases, such structures show nonlinear, or more precisely, quasi-brittle behaviour – the ability to carry load continues even after the deviation from the linear branch of the force–displacement diagram until the peak point, after which a decrease in loading force follows until failure occurs, which is a phenomenon known as tensile softening (Karihaloo, 1995). The reason for this behaviour is, apart from strong heterogeneity, the existence of internal defects (pores, cracks, transition zones, etc.) or material discontinuities (e.g. inclusions), which work as obstacles to or promoters of crack propagation. Nevertheless, these discontinuities, which form stress concentrators that serve as potential weak elements in composites, are not given any consideration at all in the standards, e.g. EN 1992-1-1 (2004).

In this paper, material discontinuities are formed by rock inclusions placed in the middle of the test specimens. These specimens made of fine-grained cement-based composite with different types of rock inclusion – amphibolite, basalt, granite, and marble – were tested in the three-point bending configuration. The rock inclusions were made using a saw with a diamond blade. After the tests, the fracture surfaces were examined via scanning electron microscopy (SEM) and local response in the vicinity of the rock inclusions was characterized via the nanoindentation technique (Zacharda et al., 2018). Assuming that the test specimens were manufactured, compacted and tested in the same way and that the inclusions' surfaces had the same roughness, the only way to explain the deviation in overall fracture response should be, according to Randl (2013) and the fib Model Code for Concrete Structures 2010 (2013), chemical adhesion. The aim of this paper is to identify the influence of mineralogical composition of rock inclusions on the overall fracture response of the above-described cement-based composite specimens.

2. Theoretical background

2.1. Interface shear transfer

The bond mechanism is the interaction between reinforcement and concrete. The components of bond resistance are a combination of different mechanisms – chemical adhesion, friction, mechanical interlocking and the dowel action of reinforcement crossing the interface (Randl, 2013).

The dowel action of reinforcement crossing the interface is the result of the lateral displacement of the upper and lower reinforcement ends due to shear slip along the interface. The transfer of shear forces along the interface is thus provided by the bending, shear and axial stresses in reinforcement bars caused by this lateral displacement (Randl, 2013).

The mechanical interlocking resistance is the result of the forces acting perpendicular to the ribs of reinforcement. This resistance takes place in the case of excessive and irregular roughness when keying and undercutting effects occur. This effect will only take place if the aggregates/ribs protrude sufficiently from the surface (Randl, 2013).

The frictional resistance is the result of the compression forces perpendicular to the interface and also depends on the degree of interface roughness. In the fib Model Code for Concrete Structures 2010 (2013), there is a recommendation for the values of the coefficient of friction μ for a constant confining stress σ_c depending on whether the interface is smooth, rough or very rough. Several parameters are also described for the classification of the concrete surface roughness, such as mean roughness R_a and the mean peak-to-valley height R_z.

Although adhesive shear resistance is in the range of lower units of MPa for concrete grades ≤ C50/60, adhesive bonding can significantly affect overall shear resistance (Randl, 2013). Adhesive resistance is a result of chemical and physical bonding due to Van der Waals forces. For this effect to occur, the related slip at failure must be very small, otherwise the effect will vanish. Adhesive resistance strongly depends on the real surface of the contact area, and the quality, composition and properties (e.g. porosity) of concrete (Randl, 2013). It is connected to the formation of the interfacial transition zone – see the next section – at the aggregate/matrix or reinforcement/matrix interface, which is regarded as the weakest element of cement-based composites.

2.2. The Interfacial Transition Zone

The existence of the Interfacial Transition Zone (ITZ) between aggregate and cement paste was first described in the 1950s by Farran (1956). The ITZ is a region of about 50 μm in size, when using ordinary Portland cement (Scrivener et al., 2004). It should be noted that the ITZ is not a separate region, but the region of transition and it is difficult to determine the exact boundaries (De Rooij et al., 1998). On the surface of the aggregate grain, there is a thin coating of 1 μm in thickness, called "duplex film", which consist of a calcium hydroxide (CH) layer and a thin layer of short fibers of calcium-silica-hydrate (C-S-H) gel (Barnes et al., 1978). The remaining microstructure of the region is formed mainly by ettringite needles and portlandite plates, while the amount of unhydrated cement grains is reduced (Diamond et al., 1986).

The ITZ's significant feature is mainly its higher porosity compared to the bulk matrix (Scrivener et al., 2004). The local increase in porosity is in a good agreement with the lower values displayed by the mechanical fracture parameters of the ITZ, see e.g. Zacharda et al. (2018). These lower values are inevitably connected with the bond resistance.

3. Experimental part

3.1. Rocks

Four basic types of rocks were selected for the preparation of rock inclusions. Specifically, these were: (i) amphibolite from the former Rožná I uranium mine, (ii) olivine basalt from the Bílčice quarry, (iii) biotite granite from the Černá Voda-Nový lom quarry, and (iv) marble from the Horní Lipová-Mramorový vrch quarry (Fig. 1). These rocks were chosen deliberately, as they essentially represent the main raw materials used in the production of crushed aggregates in the Czech Republic. More than 200 deposits of crushed stone are currently quarried on the territory of the Czech Republic (Starý et al., 2020), of which about 23 % are granite deposits, approx. 12 % basalt deposits, around 7 % amphibolite deposits and approx. 2 % marbles. In terms of the total production of crushed aggregates in the Czech Republic, basaltic volcanites account for about 25 % and acidic plutonites such as granites about 20 % of the currently produced aggregates (Starý et al., opus cit.).

Fig. 1. Rock specimens used for inclusion preparation after fracture tests (in order from left to right): amphibolite, basalt, granite and marble.

Dark grey to black, coarse-grained amphibolite from the Rožná I mine (approximately 40 km NW from Brno) is mostly formed from amphibole (approx. 60–70 % of the rock volume), which varies in composition from tschermakite to magnesiohornblende (Bukovská et al., 2019). Other rock components consist of plagioclase, the basicity of which corresponds to andesine up to labradorite (approx. 20–30 vol. %) and rarely occurring quartz (up to 10 vol. %). Typical accessory minerals are represented by titanite, zircon and opaque phases, probably pyrite. The rock exhibits plane parallel structure and granonematoblastic texture.

Olivine basalt from the Bílčice quarry (approx. 40 km NE from Olomouc) typically exhibits massive to vesicular structure and porphyritic texture, with pilotaxitic texture of the rock matrix. Phenocrysts are predominantly formed by olivine (approx. 15–20 %), while other rock forming minerals are represented by pyroxene (in particular augite, approx. 37–45 %), calcium-rich plagioclase of labradorite composition (about 20–30 %) and magnetite (up to 20 %). The proportion of amorphous phase (basaltic glass) is up to 3 %. In addition to aggregate production, this basalt was also used in the past for mineral wool manufacturing (Slivka and Vavro, 1996).

The "light Silesian granite" from the Černá Voda-Nový lom quarry (approx. 10 km N from the city of Jeseník) is petrographically represented by light grey to grey, medium-grained biotite granite, which typically features holocrystalline, equigranular, hypautomorphic to panxenomorphic granitic texture and massive structure. Its mineral composition is relatively simple, with felsic rock components formed by quartz (approx. 30 %), K-feldspars (approx. 40 %) and plagioclase (approx. 25 %), while biotite (approx. 5 %) is the basic mafic mineral. Accessory minerals include zircon, titanite, apatite, magnetite and rare allanite (Malíková et al., 2019).

The marble quarried at the Horní Lipová-Mramorový vrch deposit (approx. 7 km W from the city of Jeseník) is a well-known building and decorative stone material often referred to as "dark Lipová marble". The rock typically has a light grey to dark grey colour, often with well-visible banding. It is almost entirely (often more than 90 %) composed of calcite, other minerals such as graphite, quartz, muscovite, and pyrite rarely occur, some of them even only as accessories. The rock exhibits massive to plane parallel structure and granoblastic texture.

The chemical composition of the rocks used for inclusion preparation was determined semiquantitatively using a XEPOS X-ray fluorescence (XRF) energy dispersive spectrometer (Spectro Analytical Instruments GmbH, Germany). The milled rock sample was mixed with wax and a tablet was moulded and then analysed in a protective atmosphere (He). The results of determining the chemical compositions of the rocks are shown in Table 1.

Table 1. Chemical composition of rock inclusions determined using XRF spectrometry [%].

Inclusion	SiO_2	TiO_2	Al_2O_3	Fe_2O_3*	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	LOI	Σ
Amphibolite	44.80	0.88	15.25	12.08	0.22	9.56	12.47	1.57	1.05	0.05	1.57	99.50
Basalt	42.21	2.66	13.36	13.72	0.22	8.26	12.90	3.80	0.76	0.97	0.56	99.42
Granite	71.60	0.29	13.70	2.68	0.04	0.41	1.95	3.45	5.02	0.11	0.43	99.68
Marble	2.31	0.12	1.12	0.72	0.01	0.70	53.10	0.19	0.21	0.06	41.31	99.85

Explanations: * = iron in the form of Fe_2O_3, LOI = loss-on-ignition; the samples were burned in a muffle furnace for 3 hours at 1100ºC

3.2. Physicomechanical properties and fracture tests of rocks

The tested rocks were acquired in the form of blocks of irregular shape and with a side length of about 0.3–0.4 m. Cylindrical samples measuring 48 mm in diameter were subsequently drilled from the blocks under laboratory conditions. The ends of the drill cores were finally cut perpendicularly to their length, so that the L:D ratio (length-to-diameter ratio or slenderness ratio) of the prepared test specimens was about 2:0.7. Basic physical and mechanical characteristics were tested on dry specimens according to standard procedures represented by relevant European standards and suggested testing methods of the International Society for Rock Mechanics. The mechanical properties of the studied rocks were determined by computer-controlled mechanical presses: the FPZ 100 (VEB TIW Rauenstein Thüringer, Germany) and the ZWICK 1494 (Zwick/Roell, Germany).

In order to determine the fracture toughness and other important mechanical fracture properties of the input rocks, the three-point bending test was performed. For this test, long cylindrical specimens with a chevron (V-shaped) notch perpendicular to the specimen axis were used. A clip-on gauge type of extensometer was attached at the mouth of each chevron notch, allowing the relative crack face opening (*CMOD* – crack mouth opening displacement) to be measured. Cylindrical test specimens of 48 mm in diameter and about 190 mm in length were drilled from the rock blocks. A diamond blade was used to cut the chevron notches with an internal angle of 90° and a thickness of 1.5 mm perpendicular to the core body axis and positioned in the centre of each sample. After the chevron notches had been cut, the test specimens were dried to a constant weight. Fracture toughness was calculated from measured force F vs. *CMOD* diagrams obtained from a three-point bending test which was carried out at room temperature on an FPZ 100 power press with displacement control at a constant loading rate of 0.1 mm·min^{-1}. For more details about the methods employed in this test, see Vavro and Souček (2013) or Vavro et al. (2019).

The inclusions were made using a saw with a diamond blade to cut them from all above-mentioned rock types – amphibolite, basalt, granite, and marble.

3.3. Matrix material

The matrix of the test specimens was prepared from a fine-grained cement-based composite. The fresh mixture consisted of CEM I 42.5 R Portland cement (Mokrá cement plant, Czech Republic), ČSN EN 196-1 (2005) standard quartz sand with a maximum grain size of 2 mm, and water in the ratio 1:3:0.35 (cement:sand:water). To ensure workability, a polycarboxylate-based high-range water-reducing admixture (Sika SVC 4035) was added in an amount of 1 % by cement mass. The properties of the fresh composites were determined in accordance with ČSN EN 1015-3 (2000) and ČSN EN 1015-6 (1999). Workability of mixture was 140 mm and bulk density was established as 2.280 g/cm3. For more details, see Vyhlídal et al. (2019).

3.4. Specimens

The specimens with nominal dimensions of 40 × 40 × 160 mm containing an internal inclusion with nominal dimensions of 8 × 8 × 40 mm placed in the middle of the span above the initial notch were manufactured for the fracture tests, see Fig. 2. The only difference between the test sets was the type of rock inclusion – amphibolite, basalt, granite, or marble. Each test set contained three test specimens.

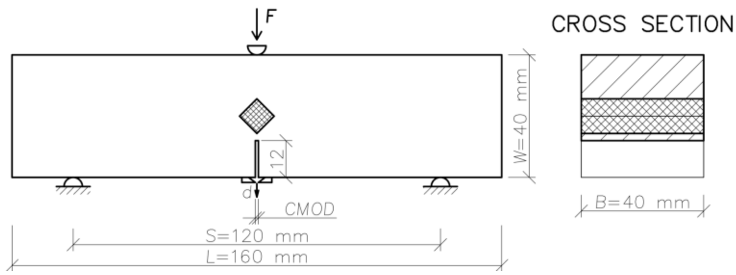

Fig. 2. Specimen geometry and fracture test configuration (Vyhlídal et al., 2019).

Three-part polyethylene (PE) moulds were used to produce the test specimens. The rock inclusions were fixed in position in each part of the moulds before they were filled, see Fig. 3. The mixture was prepared under laboratory conditions using a hand-held paddle mixer. After pouring and compaction of the fresh mixture, the moulds were covered with a thin PE foil and stored under stable laboratory conditions with a temperature of (22 ± 2) °C for 3 days. After demoulding, the test specimens were cured in a water bath until they were tested. The initial notch was made just before the fracture tests using a saw with a diamond blade, the notch depth being approximately 1/3 of specimen depth.

Fig. 3. Moulds defining the final shape of the specimens with fixed rock inclusions.

3.5. Fracture tests of test specimens

To determine the influence of the ITZ on the fracture behaviour of fine-grained cement-based composite, fracture tests were conducted on the aforementioned specially designed specimens via three-point bending. The experiments were conducted using a very stiff LabTest 6-1000 multi-purpose mechanical testing machine (LaborTech Ltd., Czech Republic) with a load range of 0−1000 kN. The fracture tests were conducted under monotonic loading conditions with a constant displacement increment of 0.02 mm·min^{-1}. The load span was 120 mm.

During the experiment, besides the force (F), vertical mid-span displacement (d) and *CMOD* were continuously recorded. In order to measure *CMOD*, a strain gauge was fixed between steel blades, which were placed in close proximity to the notch. The mid-span displacement was measured using inductive sensors. As a result, both F−d and F−*CMOD* diagrams were obtained. An illustration of the three-point bending fracture test configuration is shown in Fig. 4.

Fig. 4. Three-point bending fracture test – LabTest testing machine (on the left); detail of measuring equipment (on the right).

3.6. Scanning electron microscopy measurements

After the fracture tests, the resulting fracture surfaces were examined via scanning electron microscopy at the AdMaS science centre, which is part of The Faculty of Civil Engineering at Brno University of Technology, and at the Institute of Theoretical and Applied Mechanics (ITAM) of the Czech Academy of Sciences. A TESCAN MIRA3 XMU scanning electron microscope with an environmental probe with 3D imaging was used.

In this paper, micrographs created by the detection of secondary electrons (SE) are presented, while micrographs created by the detection of backscattered electrons (BSE) are omitted. SE are created by inelastic scattering of the beam electrons, while BSE are created by elastic scattering and are in fact primary electrons returning after a Coulomb interaction. SE thus provide information about topography, while BSE, in contrast, provides information about composition depending on atomic number Z (Goldstein et al., 2018).

3.7. Nanoindentation measurements

Nanoindentation was applied in the vicinity of each inclusion (ITZ zone) to reveal any changes in micromechanical response (Němeček et al., 2013). A Hysitron TriboLab TI-700 nanohardness tester equipped with a Berkovich diamond tip was used. A load-controlled test with the trapezoidal loading function (linear loading for 1 s, holding for 20 s and 1 s unloading) to a maximum force of 2 mN was prescribed for each indent. A rectangular matrix of about 100−200 indents was positioned partly to the inclusion, ITZ and bulk material, see Fig. 5. The matrix contained several rows with an inter-indent separation of 2−3 μm.

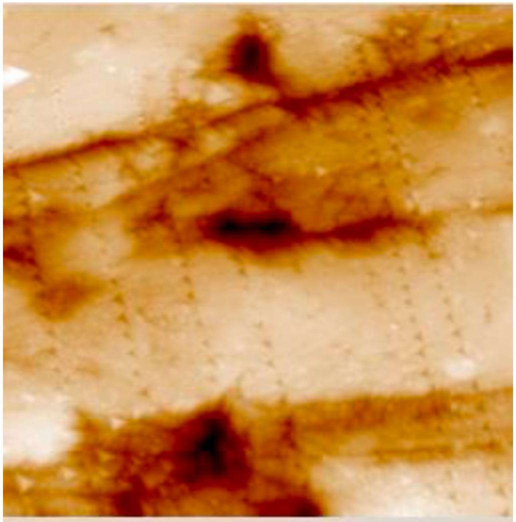

Fig. 5. Matrix of indents.

Young's modulus E and hardness H were estimated by Oliver and Pharr’s (1992) theory (assuming Poisson's ratio ν = 0.2) as

$$H = \frac{F}{A_c} \tag{1}$$

$$E = \frac{1}{1-\nu^2}\frac{S\sqrt{\pi}}{2\beta\sqrt{A_c}} \tag{2}$$

where F is the maximum indentation force, S and A_c are the contact stiffness and area, respectively, and β is the tip correction factor. During the holding period, time-dependent deformation is characterized with the creep indentation parameter, the CIT, as

$$CIT_{(P,t_1,t_2)} = \frac{h_2 - h_1}{h_1} \times 100 \tag{3}$$

which is defined as a relative change between indentation depths h_1 encountered at time t_1 and h_2 at time t_2, respectively (i. e. the *CIT* depends on the contact force *F* and the time of holding period). Creep was also described with the creep compliance function assuming step loading as:

$$J(t) = \frac{2h^2(t)}{\pi(1-\nu^2)F \tan\alpha} \tag{4}$$

where $h(t)$ is the depth of the indent at time t, F is the loading force and α is the angle between the surface and edge of the tip (for a Berkovich diamond tip $\alpha = 19.7°$). Although the assumption of step loading is not perfectly fulfilled, Eq. 4 gives a good estimate for the $J(t)$.

4. Results

In this section, the results of fracture tests, nanoindentation measurements and SEM measurements are presented.

4.1. Physico-mechanical properties and fracture tests of rocks

Before fracture testing, some fundamental physical and mechanical rock properties were determined since these were assumed to influence the fracture mechanical behaviour of the studied rocks. Specifically, bulk density ρ, ultrasonic wave velocity v_P, water absorption capacity under atmospheric pressure w_{atm}, total porosity φ, and uniaxial compressive strength σ_c were determined on cylindrical specimens with an L:D ratio of 2 (48 mm in diameter, 96 mm high). Tensile splitting strength σ_t, determined by the Brazilian test, was measured on disc-like specimens with an L:D ratio of 0.7 (48 mm in diameter, 34 mm thick). Obtained results which represent the average value calculated from at least five individual measurements are shown in Table 2.

Table 2. Physical and mechanical properties of rocks.

Inclusion material	ρ [kg·m^{-3}]	v_P [km·s^{-1}]	w_{atm} [%]	φ [%]	σ_c [MPa]	σ_t [MPa]
Amphibolite	2990	6.68	0.13	0.81	193	13.5
Basalt	2970	5.49	1.16	3.44	232	12.3
Granite	2620	4.80	0.31	1.50	185	7.5
Marble	2710	4.92	0.17	0.69	107	8.9

As stated in Chapter 3.2., for the purpose of estimating fracture behaviour, the chevron bend (CB) test was performed and the mode I fracture toughness and other important mechanical fracture properties of the selected rocks were evaluated (see Table 3). Here, E_{agg} is the bending Young's modulus, ν_{agg} represents Poisson's ratio, $K_{Ic,\,agg}$ is the mode I stress intensity factor (fracture toughness), $G_{Ic,\,agg}$ is the mode I critical strain energy release rate, and $G_{F,\,agg}$ represents fracture energy.

Table 3. Mechanical fracture properties of rocks.

Inclusion material	E_{agg} [GPa]	ν_{agg} [–]	$K_{Ic,\,agg}$ [MPa·m$^{1/2}$]	$G_{Ic,\,agg}$ [J·m^{-2}]	$G_{F,\,agg}$ [J·m^{-2}]
Amphibolite	143.0	0.16	3.370	79.60	448.0
Basalt	87.8	0.15	2.250	57.40	339.0
Granite	59.6	0.18	1.260	26.70	189.4
Marble	108.1	0.20	1.850	31.60	249.2

4.2. Fracture tests of specimens

During the fracture tests, all possible crack propagation directions were observed, see Fig. 6. The crack propagation paths labelled a) and b) in Fig. 6 were observed only for the one specimen with a marble inclusion, while for the rest of the specimens with a marble inclusion, as well as for those with basalt inclusions, crack propagation path c) was observed. In the case of specimens with amphibolite or granite inclusion, the d) crack propagation path occurred. It is evident, that the crack propagation paths in the case a) and b) are caused by low hardness (3 on Mohs scale) and especially by perfect cleavage of calcite as a dominant rock-forming mineral of marble. Crack propagation path c) indicates a high degree of cohesion between rock inclusion and cement matrix and in the case of basalt it is probably due to the vesicular texture, i. e. the presence of pores on the surface of the inclusion. The pores increase the real surface of the contact area between cement matrix and aggregate inclusion which probably contributes to adhesive resistance improvement. Case d), found for amphibolite and granite, is then unfortunately probably due to the method of preparation of inclusions. Because of being sawn using a diamond blade, the inclusions have flat and smooth surfaces, which causes them to have lower cohesion with the cement matrix than there probably should be. Therefore, in the event of a future continuation of these experiments, it will be appropriate to consider another method of preparing the inclusions, for example by means of water jet cutting.

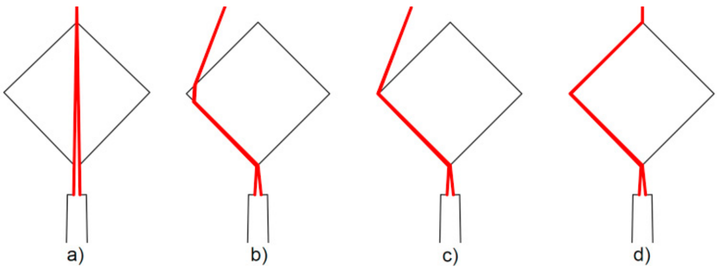

Fig. 6. Illustration of crack propagation paths.

Specimens after fracture testing can be seen with their crack propagation paths in Fig. 7. Please note that the specimens are labelled with the first three initial letters of the material from which the inclusions are made (e.g. AMP for amphibolite, BAS for basalt etc.). The specimens in the right side of Fig. 7 are reference specimens which were made only from fine-grained cement-based material (matrix) for the determination of the mechanical fracture properties of the matrix.

Fig. 7. Specimens after fracture testing (left), and selected details with inclusions: amphibolite, basalt, granite, marble.

The measured $F–d$ diagrams were used to estimate values for the maximal force F_{max}, Young's modulus of elasticity E, specific fracture energy G_F, fracture toughness K_{Ic} and effective fracture toughness $K_{Ic,e}$. Young's modulus of elasticity E was estimated from the first, almost linear part of these diagrams – see Karihaloo (1995). Specific fracture energy G_F was calculated using the work-of-fracture method. It represents the energy necessary for the creation of a unit area of a crack (RILEM, 1985). Fracture toughness K_{Ic} was estimated from F_{max} according to Karihaloo (1995). It represents a linear elastic brittle material's resistance to crack propagation. In contrast, the effective fracture toughness $K_{Ic,e}$ was determined based on the Effective Crack Model (Karihaloo, 1995), in which the difference between the initial tangent stiffness and the secant stiffness of the specimen at peak load F_{max} is considered. The determined mechanical fracture parameters can be seen in Table 4.

Table 4. Mechanical fracture parameters (Vyhlídal et al., 2019).

Inclusion material	F_{max} [kN]	E [GPa]	G_F [J·m^{-2}]	K_{Ic} [MPa·m$^{1/2}$]	$K_{Ic,e}$ [MPa·m$^{1/2}$]
Amphibolite	0.53	37.7	30.5	0.295	0.40
Basalt	0.79	42.1	42.0	0.443	0.74
Granite	0.83	46.5	42.4	0.462	0.67
Marble	0.83	39.8	57.7	0.462	0.97

4.3. Nanoindentation measurements

The results for E, H, CIT and $J(t)$ were evaluated with regard to their dependence on distance from an inclusion. E, H exhibit a gradual increase with distance, defining a weaker ITZ around the rock inclusion in the region of 0–20 μm. The region is characterized by a lower modulus and a lower hardness compared to the bulk for all specimens, as already detected in (Zacharda et al., 2018, see Table 5). Slightly lower E and H values among the specimens can be found for specimens with amphibolite inclusions. The CIT parameter in the ITZ around inclusions is always higher due to the higher creep encountered in this zone. The highest amount of creep and the highest CIT and $J(t)$ are exhibited by the specimens with amphibolite inclusions, especially in the ITZ of these specimens. Microstructurally, the ITZ can be described as having a higher porosity around the aggregate (Scrivener et al., 2004). Consistently, the evolution of Young's modulus and hardness has a negative correlation with porosity, while CIT and the amount of creep scales with porosity.

To quantify the influence of micromechanical parameters measured by nanoindentation, the mean hardness (H_{50}) and average creep compliance $J_{50}(t)$ values were calculated over an ITZ region of 50 μm, while the mean Young's modulus values were calculated over ITZ regions of 20 μm ($E_{mic,20}$) and 50 μm ($E_{mic,50}$) due to the higher values of porosity in the first 20 μm of the ITZ; see (Bourdette et al., 1995) or (Scrivener et al., 1987).

Table 5. Results of nanoindentation measurements (Zacharda et al., 2018).

Inclusion material	$E_{mic,20}$ [GPa]	$E_{mic,50}$ [GPa]	H_{50} [GPa]	$J_{50}(t)$ [GPa^{-1}]
Amphibolite	23.2	25.8	0.75	0.188
Basalt	32.8	36.1	1.32	0.053
Granite	34.2	37.9	2.12	0.045
Marble	34.4	34.5	1.33	0.063

4.4. Microstructure of the ITZ

In the following four figures, the micrographs display the hardened cement that adhered to the inclusions after the mechanical tests; the microstructures of the specimens with amphibolite, basalt and granite are very similar in character. The specimens with marble inclusions are different; there are large portlandite crystals in many places, and the CSH and CAH phases have a less porous structure.

Given that the used inclusions do not contain components that tend to react with hardened cement, such as pozzolans, or form new phases in the ITZ, the adhesion probably results from the surface roughness of the inclusions. This property of the inclusions and the adhesion of the hardened cement to their surface have not yet been measured; they will be the subject of further research.

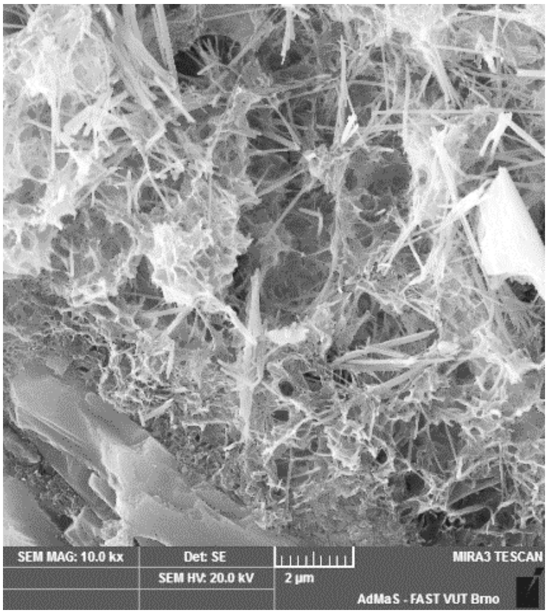

Fig. 8. Microstructure of the amphibolite–matrix interface characterized by SEM via the detection of secondary electrons.

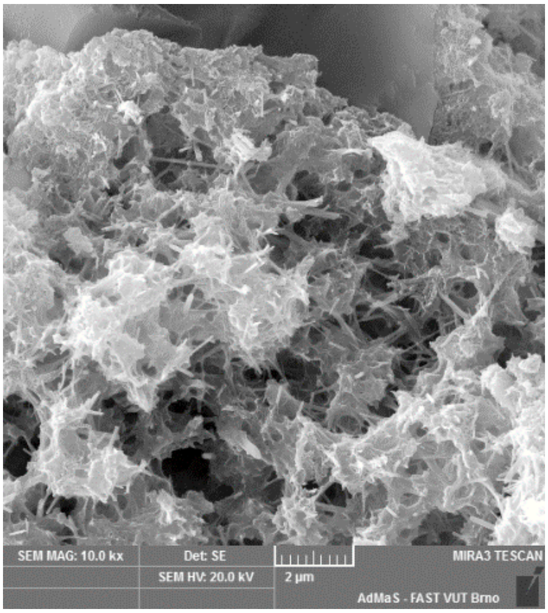

Fig. 9. Microstructure of the basalt–matrix interface characterized by SEM via the detection of secondary.

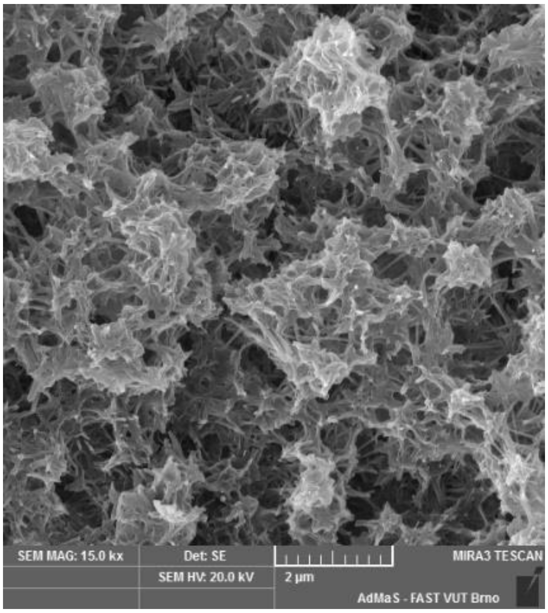

Fig. 10. Microstructure of the granite–matrix interface characterized by SEM via the detection of secondary electrons.

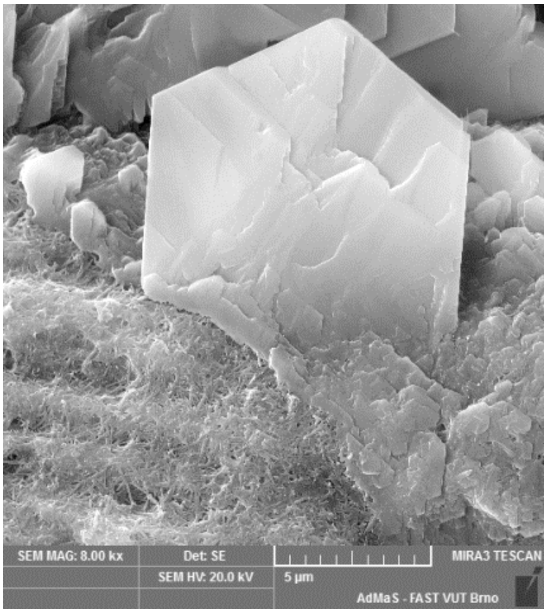

Fig. 11. Microstructure of the marble–matrix interface characterized by SEM via the detection of secondary electrons.

5. Discussion

In this section, the correlations between components will be described and discussed. The correlations will be divided according to their correlation coefficient value (dimensionless) into five groups – 0.00–0.30 weak, 0.31–0.70 moderate, 0.71–0.80 strong, 0.81–0.99 very strong and 1 for perfect – according to Kozak (2009), Akoglu (2018) and Ratner (2009). Negative correlations can be obtained by changing the sign to negative.

Before making these detailed correlations, it should be emphasized that the mechanical fracture parameters of the rocks under study correspond very closely with their basic physical and mechanical properties. It is obvious that high bulk density, low porosity and corresponding high strength properties are reflected, for example, in high rock fracture toughness values. As for the correlation between the indirect tensile strength measured using the Brazilian test and the mode I fracture toughness, the obtained results ranged within the relations published by Zhang (2002) and Vavro and Souček (2013), i. e. σ_t = approx. 4.0 to 6.0 K_{Ic}.

5.1. Influence of the chemical composition of rock on the overall mechanical fracture parameters of rock

The influence of the chemical composition of rock on the overall mechanical fracture parameters of rock is presented in Table 6. The correlation between the fracture toughness of rock $K_{Ic,agg}$ and the chemical composition is mainly weak or moderate. The correlation between the Poisson's ratio of a rock and its chemical composition is very strongly negative in the case of TiO_2, Al_2O_3, Fe_2O_3, MnO and MgO. The only one very strong positive correlation between E_{agg} and chemical composition was found for MgO. The authors are aware that correlations can be misleading. However, the deeper causes of chemical influence are still being researched, and thus only correlations are presented here.

Table 6. Influence of the chemical composition of rock on the overall mechanical fracture parameters of rock: coefficients of correlation.

	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5
$K_{Ic,\,agg}$	–0.47	–0.03	–0.05	0.38	0.45	0.60	0.32	–0.64	–0.70	–0.28
ν_{agg}	–0.48	**–0.85**	**–0.82**	**–0.96**	**–0.95**	**–0.88**	0.69	–0.66	0.09	–0.67
E_{agg}	–0.14	0.30	0.34	0.72	0.78	**0.87**	–0.06	–0.25	–0.58	0.00
$G_{Ic,\,agg}$	0.06	0.48	0.54	**0.87**	**0.91**	**0.96**	–0.28	0.01	–0.47	0.19
$G_{F,\,agg}$	–0.08	0.43	0.42	**0.81**	**0.86**	**0.94**	–0.14	–0.12	–0.57	0.15

5.2. Influence of the chemical composition of rock on micromechanical parameters measured by nanoindentation

The influence of the chemical composition of rock on micromechanical parameters measured by nanoindentation is presented in Table 7. There is weak negative correlation between Young's modulus E_{mic} and minerals, except in the case of Na_2O, K_2O and P_2O_5, where there is weak to moderate positive correlation. Moderate to strong negative correlation occurred in the case of MgO and MnO. As regards hardness, we obtained moderate positive correlation in the case of SiO_2 and Na_2O. A strong to very strong positive correlation was found in the case of K_2O. The correlation between minerals and average creep compliance $J_{50}(t)$ is mainly moderate to weak.

More than the mineralogical composition of the inclusion, the properties of the ITZ will be influenced by the mineralogy of the newly formed phases at the aggregate-matrix interface, which was unfortunately not monitored.

Table 7. Influence of the chemical composition of rock on micromechanical parameters measured by nanoindentation: coefficients of correlation.

	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5
$E_{mic,20}$	–0.13	–0.08	–0.51	–0.60	–0.68	–0.76	0.28	0.18	0.25	0.24
$E_{mic,50}$	0.16	0.07	–0.23	–0.44	–0.53	–0.65	–0.02	0.46	0.44	0.36
H_{50}	0.46	–0.26	–0.03	–0.59	–0.64	–0.76	–0.26	0.47	**0.81**	–0.01
$J_{50}(t)$	0.00	–0.07	0.36	0.47	0.55	0.66	–0.12	–0.36	–0.31	–0.38

6. Conclusion

The chemical composition of rocks is correlated with their mechanical fracture parameters. The most important in terms of influence are Fe_2O_3, MnO and MgO. The other elements are primarily moderately correlated. These results correspond to reality – a higher content of Fe and Mg is typical for basic and ultrabasic rocks (basalt, amphibolite, gabbro, eclogites, etc.), which usually have very high strengths. Nevertheless, not only the chemical composition, but also the structure and texture of rock have an influence on mechanical fracture parameters.

The influence of the chemical composition of rock on micromechanical parameters shows mostly weak or moderate correlations, and thus it seems that it does not significantly affect the micromechanical parameters measured by nanoindentation, while the influence of the mechanical fracture properties of rock seems to be important. These results are remarkable and will push our future research in the direction of studying the processes of aggregate-matrix interface formation more deeply. Nevertheless, the properties of the ITZ will be influenced mainly by the mineralogy of the newly formed phases at the aggregate-matrix interface, which unfortunately was not monitored. These newly formed phases are the results of chemical and physical reactions between the minerals (e.g. plagioclase, quartz) and cement phases (alite, belite, etc.) of the rock. In order to describe the effect of the chemical composition of rocks on the mechanical properties of the produced concrete, several indexes have been established, see e.g. Lampropoulou et al. (2020). Nevertheless, these indexes are only applicable for a few types of rocks or for ordinary concretes.

Acknowledgements

Financial support provided by the Czech Science Foundation (GACR) under project No. 19-09491S (MUFRAS) and by Brno University of Technology under project No. FAST-J-21-7497 is gratefully acknowledged. Support from GACR 21-11965S is also acknowledged by the Czech Technical University in Prague (J. Němeček, nanoindentation of ITZ).

The authors would also like to thank the many kind colleagues who lent a helping hand, especially Petr Daněk and Patrik Bayer from Brno University of Technology's Institute of Building Testing and Institute of Chemistry, respectively for providing support for the performance of fracture tests and for scanning electron microscopy micrographs. Our thanks also go out to Alexandr Martaus from the Institute of Environmental Technology at VSB–Technical University of Ostrava, who kindly performed an analysis of rock chemical composition using X-ray fluorescence spectroscopy. These experimental results were accomplished using the Large Research Infrastructure ENREGAT supported by the Ministry of Education, Youth and Sports of the Czech Republic under project No. LM2018098.

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