Preliminary results on linear expansion force change in freeze-thaw cycles as indicator of frost resistance

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Abstract: Frost resistance of rocks is important parameter in assessment of rock decay due to exogenous processes. Current testing methods rely on thermal loading of saturated samples in climatic chamber for extended period of time and consequent detection of drop in ultrasonic velocity speed or uniaxial compressive strength (UCS). Presented study aims at developing faster method of frost resistance detection by measuring linear expansion force generated during freeze-thaw cycling on fully saturated samples. Preliminary results on andesite samples taken from two sites in middle Slovakia suggest that higher volume of micropores influences frost resistance of samples, causing lower frost resistance to thermal loading of fully saturated samples. Further, cycle testing in modified thermodilatometer VLAP04 suggest the possibility of detection of higher amount of micropores by linear expansion force (LEFC) detection during initial cycles of testing, potentially allowing for faster detection of frost resistance in harder rocks compared to conventional methods using climatic chamber. Moreover, frost resistance parameter k_2 (STN 72 1800) appears to be not suitable for harder rocks due to high variance in UCS and destructive character of these tests.

Key words: andesite, linear expansion force change, freeze-thaw cycles, porosity, frost resistance, ultrasonic velocity, uniaxial compressive strength

1. INTRODUCTION

The formation of heat stress in hard rocks is caused by factors such as changes in temperature and humidity which can lead to the process of physical disintegration. The disintegration process is manifested by the enlargement of existing dislocations in the rock mass and disintegration of the rocks into larger or smaller fragments. Repetitive Freeze-Thaw cycles (F-T) in the surrounding environment play an important role in this process. Heat changes represent the primary impulse for dilation or contraction of hard rocks, while moisture multiplies it.

Bartlett (1832) belonged among the first academics dealing with fluctuation of heat followed by the formation of dislocations in hard rocks. Since then several other authors (Torraca & Weber, 1986; Weis, 1994; Widhalm et al., 1996; Leiss & Weiss, 2000; Weis, 2000; Zeisig et al., 2002; Weis et al., 2003, 1999; Scheffler and Normandin, 2004; Alnæs et al., 2004; Siegesmund et al., 2000a, 2000b, 2001, 2004; Ruedrich & Siegesmund, 2006) devoted their time to simulate cyclic temperature changes focusing on qualitative features of hard rocks in laboratory conditions. Buessem & Bush (1955) and Kingery (1955) focused on the meaning and role of heat stress in the deformation of hard rocks. In their opinion, the change of temperature doesn't cause any stress if the rock is homogeneous, isotropic and freely expandable in every direction. But in reality, such perfect conditions do not exist. The heat stress generates in hard rocks, which are subject to a series of temperature induced events, which may cause their deformation over time. The breakdown of hard rock at the moment of exposure is not visible but its causes can be exponential over a longer period of time. Research focusing on simulating natural heat conditions discovered that most cases occur in the range from –20 °C to +20 °C, with a temperature increase rate from 0.3 °C/min to 2 °C/min (Cooper & Simmons, 1977; Widhalm et al., 1996; Siegesmund et al., 2000a, 2000b; De Castro Lima et al., 2004; Salieri et al., 2005). Permanent deformation of the rock samples may even occur if they are exposed to daily and seasonal temperature changes, with permanent temperature changes occurring in various lithological types at different breakthrough temperatures (Battaglia et al., 1993). The problems of microscopic thermomechanical conditions of the rocks and the formation of thermal stress of the building stone were dealt with by Logan et al. (1993), Winkler (1996), Varga et al. (2004) Åkesson et al. (2006), Siegesmund et al. (2000b, 2007), Sippel et al. (2007), Grelk et al. (2007), Scheffzük et al. (2007) and Schouenborg et al. (2007). They concluded that one of the causes of thermal fatigue of treated rocks are cyclic temperature changes.

Porosity and its ability to absorb water are among the most important properties when studying the resistance of the rock material (Khan, 2007). Water is one of the major contributors involved in the weathering processes and is of a great importance for the long-term stability of rocks and rock masses. Based on the pore size, the presence of water in the pore system of the rock can be divided as follows: in micropores there is < 0.1 um of water condensation with a relative humidity less than 99 %, for pores of 0.1 mm there is > 1 um characteristic capillary saturation, and in macropores there is > 1 um convection in the rock pore system. The open rock porosity is a

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decisive factor for detecting the amount of water absorbed in the pore system of rock. Litvan (1980) examined the effect of various open porosities of hard rocks in terms of resistance to weathering factors with hard rocks having an average porosity ranging from 1 μm to 5 μm being the least resistant. Cyclic freezing occurs at a temperature of about 0°C in the presence of water in the rock. However, many scientists point out that this phenomenon occurs at much lower temperatures (Morioka et al., 1973; Litvan, 1972; Bager & Sellevold, 1987; Banthia et al., 1989) because water in the macropores is frozen later than a free water. The connection is that the smaller the pore size, the lower the freezing temperature (Everett, 1961; Blachere & Young, 1972). Understanding the conditions of crystallization pressures in the rock is related to the spread of ice in the pores of the rock. The first mechanism was described by Powers (1949) who called his theory the theory of hydraulic pressure. According to this theory, frozen water increases its volume by 9%, which subsequently causes the hydraulic pressure buildup to form a closed pore system of the rock (Johannesson, 2010). Other authors studying the process of freezing water in pores were Vlahou & Worster (2010). On ideal models of spherical pores, they showed how crystallization pressure of ice develops. They discovered that low-permeable rocks, such as granites are more prone to cyclic freezing. The reason is the formation of tension between rock and ice (intersubject forces) in a form of a thin layer of water, which leads to a further, even minor, decrease in temperature, increasing the crystallization pressure of ice in the pores of the rock. Ruedrich & Siegesmund (2006) emphasized the importance of saturation in the damage caused by F–T for porous sandstones. Chen et al. (2004) agreed that the pore system of the rock must be saturated to a minimum of 70 % in order to show rock breakage during cyclic freezing. The resistance of porous media to F–T cycling depends on a complex set of material properties including mineralogical composition (Dunn & Hudec, 1966), pore characteristics of the rock (Everett & Haynes, 1965; McGreevy, 1982) and thus transfer properties (capillarity, permeability) (Prick, 1995) and the mechanical properties (tensile strength, anisotropy) (Nakamura et al., 1977). Work of Pasten et al. (2011) focused on the wedging and ratchetting mechanism of failure in rock blocks concluding that diurnal and annual temperature oscillations could initiate a plastic deformation in rock blocks with specific arrangement even without presence of water in pores. Following work of Šimková (2013) presented experimental study based on Pasten's analytical model showing the plastic deformation occurrence in dry and water saturated rock blocks. Šimková (2013) also studied relation between rock saturation and linear expansion force change (LEFC) during F-T cycling on sandstone samples. Benavente et al. (2013) studied rock resistance to F-T cycles and the change of rock properties during the weathering process. Six different lithological types of rock were subjected to thermal change from −8 °C to 20 °C concluding that the rocks with the highest open porosity values (>10 %) are the least durable. These rocks showed a non-linear decay pattern, with long periods of apparent stability followed by rapid and catastrophic decay. From the studied parameters spatial attenuation of ultrasonic waves revealed as the most sensitive parameter, detecting the critical decay threshold of rocks and their imminent breakdown.

Main objective of this research was to find more effective method of detection of decay prone rocks due to frost weathering. Current practice according to (STN 72 1800) is to subject rock samples to 25 F-T cycles and calculate reduction in uniaxial compressive strength. This method however, has several shortcomings. Time consumption and destructive character of the tests are the most significant. Two types of andesite rocks with expected difference in frost resistance (Holzer et al., 2009) were tested in modified thermodilatometer VLAP 04. Fully saturated samples were tested in climatic chamber of the thermodilatometer cycling the temperature from +20 to −15 °C (average rate of cooling 0,1 °C/min) while measuring linear expansion force change (LEFC) in unconfined conditions generated by the temperature changes and subsequent ice crystallization in the pores of saturated samples. After each test a P-wave velocity, porosity, degree of saturation was evaluated. Mercury porosimetry was later used to determine the pore size distribution on studied rock samples.

2. MATERIAL AND METHODS

Samples from two types of pyroxene rich andesitic rocks were taken from quarries in Babiná Hanišberg (BH) and Hontianske Trsťany (HT) - Fig. 1. Both sites belong to the complex of Neogene volcanic rocks located in the central Slovakia. Selected sites were supposed to be of different resistance to F-T cycles based on the data (Holzer et al., 2009) tested according to national standard (STN 72 1800). This Standard defines a parameter of frost resistance k2, as the ratio between uniaxial compressive strength of sample subjected to F-T 25 cycles and a UCS of dry sample not subjected to F-T cycling. European standard STN EN 12371 defines frost resistance as the percentage change in apparent volume (DVb) at n cycles, or the percentage decrease in dynamic elastic modulus (Young´s modulus). In this study we adopted modified procedure taking into account different temperature change ratios in above mentioned Standards as well as physical and mechanical parameters measured during the tests.

Twelve (HT) and seventeen (BH) cylindrical specimens of diameter 35 mm and height 50 mm were owen dried at 105 °C for 24 hr, weighted and longitudinal ultrasonic velocity at 55 kHz was measured using direct transmission method. Ultrasonic velocities were tested by high performance ultrasonic velocity tester C373N from Matest. Physical properties as apparent density (ρ_d), real density (ρ_s) and total porosity (n) of specimens were determined according to standard (STN EN 1936) and degree of saturation (S_r) was calculated after specimens saturation in water for all specimens (Tab. 1).

Specimens from both sites were divided into three groups based on the testing program. Nine specimens in group [I] were subjected to shock F-T testing cycles, group [II] consisting of 11 specimens were tested in modified thermodilatometer VLAP04 with lower temperature change ratio and 9 specimens in group [0] were not subjected to F-T cycling.

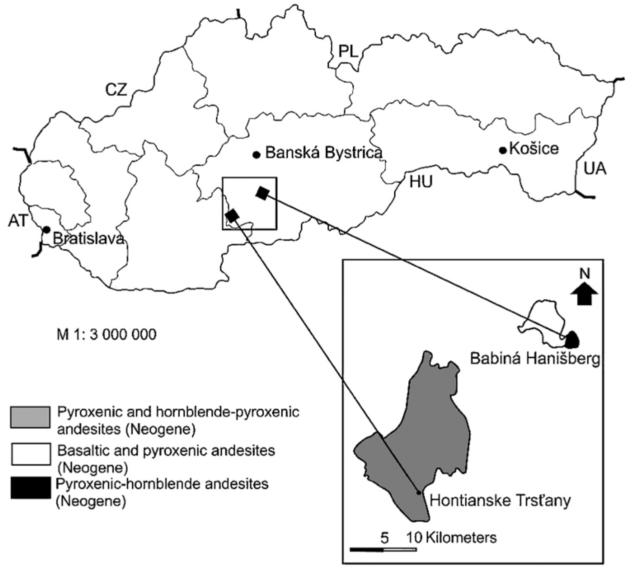

Fig. 1. Study sites locations and geological settings

Specimens in groups [I] and [II] were saturated for 48 hours using the forced water absorption method following the STN-EN 1936 standard.

Saturated specimens in group [I] were subjected to F-T cycles from room temperature +20 °C to –20 °C, they were freezing in air for 2 hours and thawed submerged in water for 2 hours. This process of accelerated F-T cycles can be considered as freezing by thermal shock due to rapid change in temperature (Fig. 2). After 25 F-T cycles specimens in group [I] were dried at 105 °C for 24 hours, weighted and longitudinal ultrasonic velocity after F-T cycling (V_{25}) was according STN EN 14579 (2010) determined.

Tab. 1. Physical characteristics of samples (AVG = average value, SD = standard deviation)

Location		ρ_d (g.cm^{-3})	ρ_{sat} (g.cm^{-3})	n (%)	S_r (%)
HT	AVG	2,54	2,57	3,53	78,25
	SD	0,02	0,02	0,78	17,84
BH	AVG	2,66	2,68	4,45	51,64
	SD	0,01	0,01	0,43	5,49

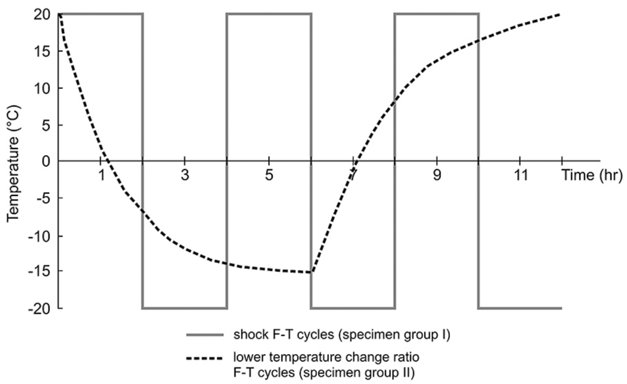

Fig. 2. Thermal loads applied during F-T cycling of specimens from group [I] and [II]

Specimens of group [II] were weighted after saturation, wrapped in thin PE foil to avoid loss of moisture and inserted to modified thermodilatometer VLAP04 (Fig. 3). Load cell sensor inserted in place of LVDT sensor allowed to record changes linear expansion force (LEFC) (Fig. 4) generated due to the dilation of specimen caused by temperature changes during F-T cycling. LEFC was measured by load cell with 0–20N range and linearity of ±0.25 % FSO from Omega.

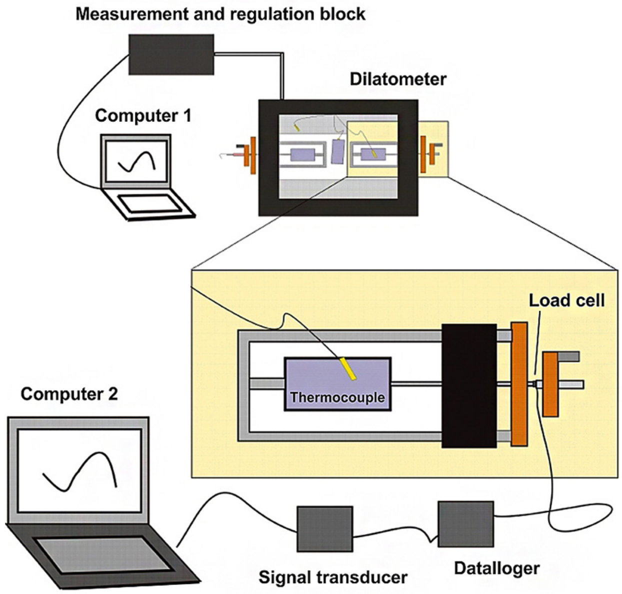

Fig. 3. Scheme of modified thermodilatometer VLAP04 (modified after Vlčko et al., 2005)

VLAP04 was calibrated using silica glass reference sample. F-T cycles were carried out in range from +20 °C to –15 °C with temperature change ratios (average cooling rate 0,09 °C/min ± 0,02 °C/min). Average cooling rate during cooling phase was from +20 °C to –5 °C 0,16 °C/min ± 0,02 °C/min and from –5 °C to –15 °C 0,04 °C/min ± 0,01°C/min. Average cooling rate during heating phase was from –15 °C to +5 °C 0,11 °C/min ± 0,05 °C/min and from +5 °C to +20 °C 0,04 °C/min ± 0,01 °C/min. Coolling rate was determined by thermodilatometer and we could not influence it. Degree of saturation of each specimen was checked after 5 F-T cycles by weighing and thermal cycling was resumed. After 25 F-T cycles specimens were weighted, dried at 105 °C for 24 hours and longitudinal ultrasonic velocity after F-T cycling (V_{25}) was determined. From P-wave velocities V_0 a V_{25} relative velocity RV was calculated as follows:

$$RV = x\ 100 \quad (1)$$

For all specimens from groups [I] and [II] uniaxial compressive strength after 25 F-T cycles (UCS_f) was determined. Uniaxial compressive strength of reference samples (UCS_0) (group [0]) not subjected to thermal cycling was determined following STN EN 1926 standard.

Parameter of frost resistance k_2 was calculated as follows:

$$k_2 = \quad (2)$$

This parameter differed from parameter k_2 calculated according to national standard STN 72 1800 only in specimen dimensions used during the tests, where national standard assumes cylindrical samples of 50 mm diameter and 50 mm height.

In order to assess the pore size distribution and volume of macropores and micropores two specimens from both studied sites were analyzed using mercury porosimetry in Quantachrome's Poremaster 60GT mercury intrusion porosimeter.

3. RESULTS

Typical record from one F-T cycle carried out in modified dilatometer VLAP04 on specimens from group [II] is shown in Fig. 4. Cycle is composed of two phases – cooling and heating, lasting approximately 6 hours each. First phase is characterized by contraction of saturated specimen (A-B), resulting in decrease in linear expansion force (LEFC is negative). LEFC is decreasing till reaching the point of phase transition C, where water in pores freezes and latent heat is released, demonstrated by slight increase in specimen temperature. This phase is characterized by sudden increase in LEFC generated during specimen expansion (B-C). Ice crystallization occurred in temperature range from −2,5 °C to −4,6 °C, as documented by several authors studying F-T behavior of porous media (Ruedrich & Siegesmund, 2006; Šimková, 2013; Ruedrich et al., 2010). Expansion of specimen continues (C-D) despite decreasing temperature due to continual ice formation in pores of rock specimen (Zheng et al., 2011). After reaching peak value D the expansion reverses and specimen starts to contract as all ice action in pores ceases and LEFC values continue to decline (D-E). F-T cycle continues into heating phase characterized by increasing LEFC values due to specimen expansion reaching point F. In this point ice thaws in the rock pores causing rapid contraction of the specimen manifested by rapid decrease in LEFC and subsequent latent heat absorption (F-G). Final phase of the F-T cycle is continual expansion of the specimen proportional to temperature increase (G-H).

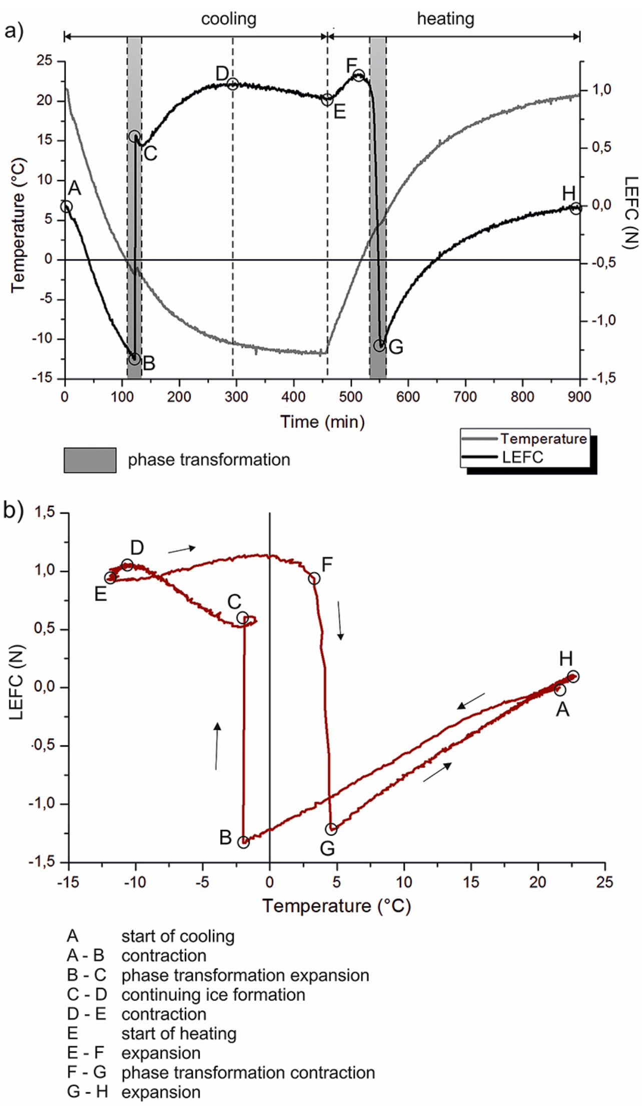

Fig. 4. Linear expansion force change path of typical F-T cycle applied to specimen from BH site describing phases with different behavior of specimen in time (a) and in relation to temperature load (b)

Fig. 5 shows records of first 5 F-T cycles carried out on specimens from both studied sites belonging to group [II]. There is substantial difference in the path of LEFC for BH, which is similar to typical F-T cycle presented in Fig. 4 and specimen from HT. The LEFC curve from HT resembles the behavior of dry specimen with the absence of phase transformation and

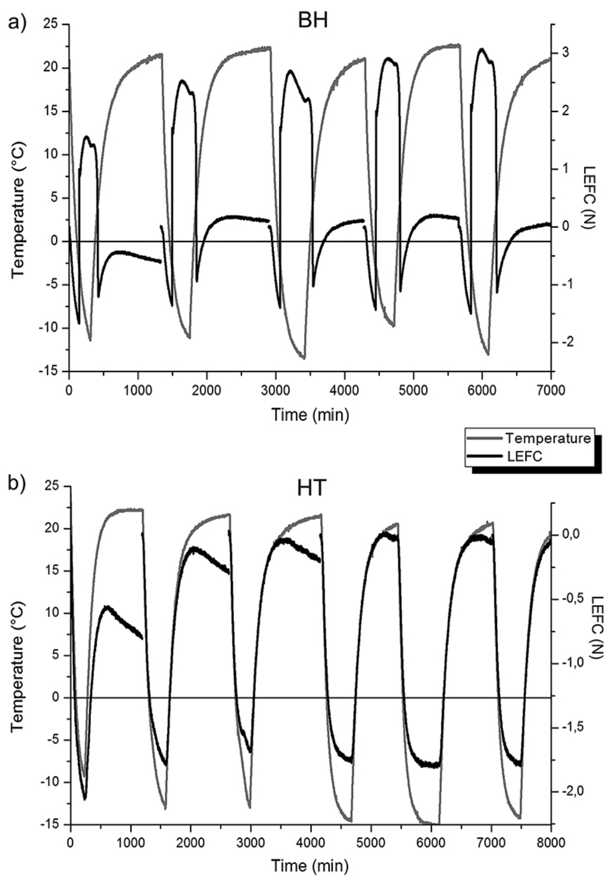

Fig. 5. LEFC curves recorded during first 5 F-T cycles for specimens from BH site (a) and HT site (b)

ice crystallization in the pores, following the curve of thermal change. In contrary the specimen form BH incorporates all phases shown in Fig. 4 including phase transformation and latent heat release.

Comparing the degree of saturation of specimens shows the HT specimens were able to saturate to an average value of S_r = 78 %, while the BH only to 51 %. Fig. 6 shows changes in group [II] specimen saturation during F-T cycling. Significant drop in saturation in case of HT specimens was caused by water condensation on the PE foil. This water was removed during specimen weighting after fifth F-T cycle and replaced by new PE foil.

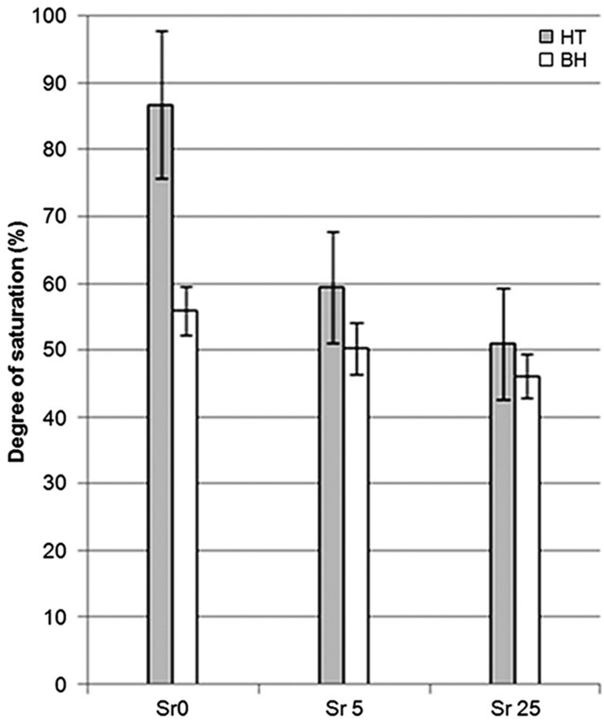

Fig. 6. **Changes in group [II] specimen saturation during F-T cycling, maximum saturation measured before F-T cycling (Sr_0) decreased due to water condensation on the PE foil after 5 F-T cycles (Sr_5) and after 25 F-T cycles (Sr_{25})**

The difference in porosity determined following the standard STN EN 1936 (Tab. 1) is less than 1 %. The ability of BH specimens to be saturated to only 51 % and despite this fact to exhibit LEFC curves showing ice crystallization where the HT samples saturated to 78 % behave as dry specimens might be attributed to the difference in pore sizes. Volume of macropores, defined as pores larger than 2,1 mm and less than 490 mm, is larger in HT than in BH specimens (Tab. 2), indicating that volume of micropores (< 2,1 mm) is crucial in the process of water freezing inside the pores, resulting in corresponding LEFC curve shape. This points to a difference between HT specimens, where water freezes mainly in macropores (88,4 %) allowing super-cooled water free movement without the change in linear expansion of the specimen due to phase transition. Average volume of micropores in case of HT specimens was 11,6 %. LEFC curve seems to be following the temperature change path. Average volume of micropores in case of BH specimens is 24,7 %, which is more than twice the volume of micropores in the HT case. This is probably the main reason for different LEFC curve shape present in specimens from this site. Suggested method of LEFC monitoring during F-T cycling might be promising in determination of micropore volume in the rock specimen as an important factor in the rock frost resistance. This argument has to be supported by further experiments on lager number of samples from different lithological types of rocks. Distribution of pore radii in specimens from two studied sites is shown in Fig. 7.

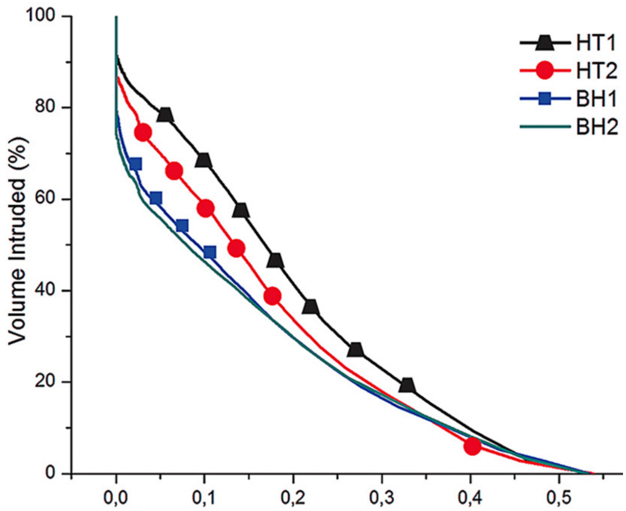

Fig. 7. **Pore size distribution in rock specimens taken from HT and BH sites**

Fig. 8 shows differences in average LEFC during various phases of F-T cycles mainly related to phase linear expansion force in BH specimens.

Parameter B-C represents change in linear expansion force during the phase transition (Fig. 4) and parameter B-D adds following continual ice formation phase. Parameter F-G represents decrease in LEFC during the thawing phase of ice in the pores of rock specimen during F-T cycle.

Results of ultrasonic wave velocity measurements before and after F-T cycling as well as uniaxial compressive test results are presented in the Fig. 9. Specimens of group [I] subjected to thermal shock cycling show decrease in longitudinal P-wave velocity by 2 %. UCS values for the same group of specimens are in range from 82,21 to 150,89 MPa for BH and from 66,78 to

Tab. 2. **Mercury porosimetry results for specimens taken from HT and BH sites**

Location	Total pore volume (cc/g)	Volume of micropores (%)	Volume of macropores (%)	Specific pore surface (m^2/g)	Median radius of all pores (nm)
BH	0,034	24,68	75,32	2,55	0,086
HT	0,026	11,62	88,39	1,6	0,16

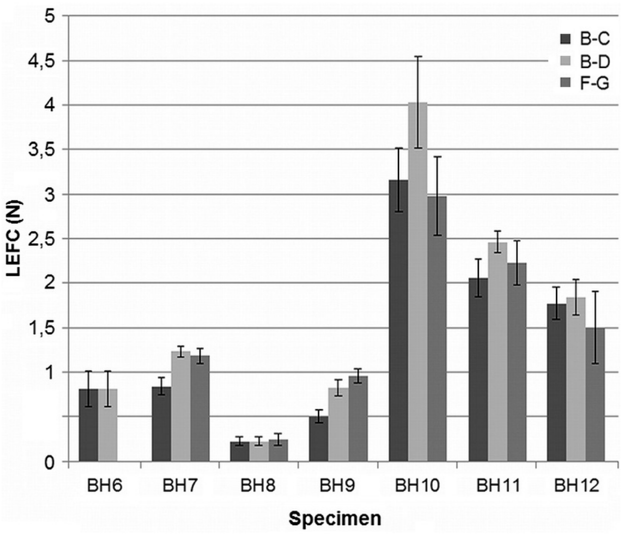

Fig. 8. Linear expansion force changes (LEFC) (N) recorded during first 5 cycles on BH specimens for various phases of F-T cycle. B-C – phase transformation expansion- freezing, B-D phase transformation and continuing expansion due to ice crystallization, F-G – phase transformation contraction, thawing)

127,21 MPa from HT. Specimens of group [II] tested in modified dilatometer VLAP04 with slower rate of temperature change exhibited average decrease in RV values of 8 % after 25 F-T cycles. UCS values range from 69,45 MPa to 161,22 MPa for site BH and from 117,45 MPa to 208,76 MPa in the case of site HT.

Ultrasonic wave velocity results correspond with results of several authors studying this parameter as potential measure of frost resistance of porous media (Ghobadi et al., 2016; Park et al., 2014; Molero et al., 2012; Martinéz-Martinéz et al., 2013). Surprisingly pronounced decrease in relative ultrasonic wave velocity in specimens subjected to gradual temperature changes during F-T cycling compared to specimens subjected to thermal shock cycling (Fig. 9). Small number of tested specimens might influence the results, however.

4. DISCUSSION

Parameter of frost resistance k_2 was studied by Holzer et al. (2009) at both sites HT and BH using the thermal shock cycling method. They determined the k_2 parameter values after 25 F-T cycles to be 0,88 and 0,35 for HT and BH sites respectively. This conclusion is in contradiction to results of this study where parameter k_2 of group [I] was after 25 F-T cycles 0,73 for site HT and exceeded 1,0 for site BH. For specimens from group [II] parameter k_2 exceeded the value of 1,0 for both studied sites (Fig. 10). These results show low reliability of parameter k_2 for hard rock specimens in both studies. Main drawbacks of this method of frost resistance determination are high variance in UCS values present in hard rocks such as andesites and more importantly the fact that different specimens are subjected to UCS testing due to destructive character of these tests. Therefore, it is suggested to use parameter k_2 only for soft rocks such as tuffs or low UCS sedimentary detritic rocks. Results are pointing towards high variability in UCS values of andesite rocks affected by presence of hidden discontinuities or alteration along internal cracks. From this point of view application of relative ultrasonic velocity ratio seems more advisable.

Decrease in RV values and more importantly lower values of BH specimens suggest good relation between LEFC data pointing at higher volume of micropores resulting in larger decrease in ultrasonic wave velocity after 25 F-T cycles compared to HT containing higher volume of macropores not prone to frost decay. This method might be less time consuming compared to other F-T cycling procedures applied by Martinéz-Martinéz et al. (2013), who used up to 96 F-T cycles, Ruedrich et al. (2010)

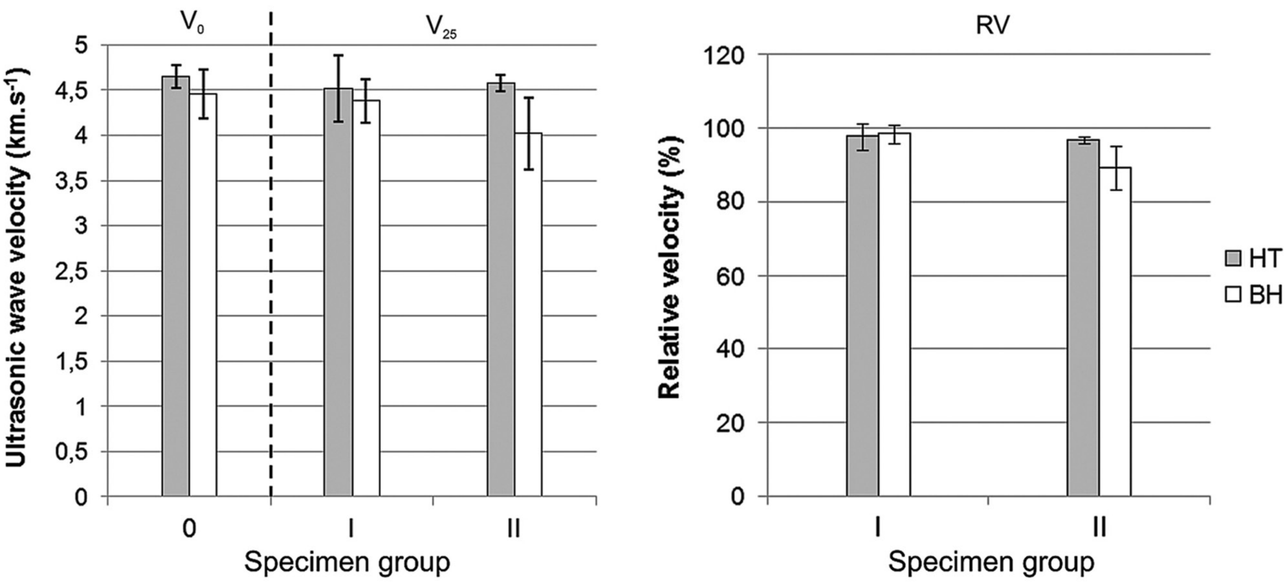

Fig. 9. The changes of ultrasonic wave velocity V and relative velocity RV for specimen group [I] subjected to thermal shock F-T cycles and specimen group [II] tested by continual thermal change cycling

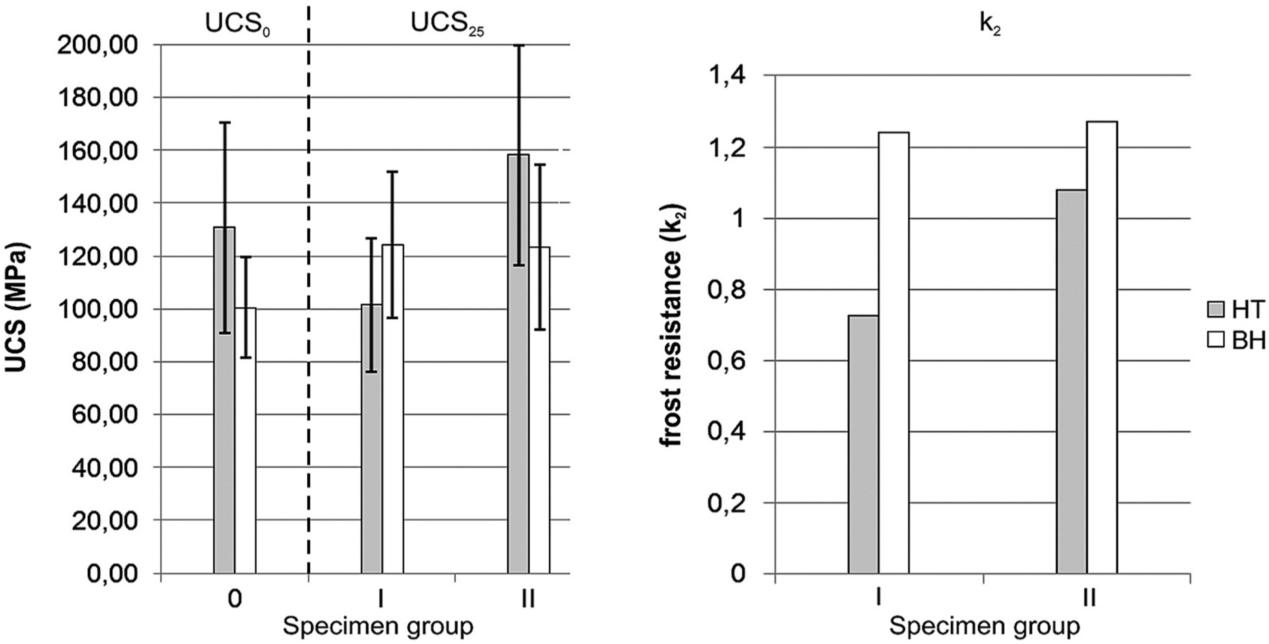

Fig. 10. Initial rock uniaxial compressive strengths UCS0 and UCS25 after 25 F-T cycles and frost resistance parameter k2 values for specimen group [I] subjected to thermal shock F-T cycles and specimen group [II] tested by continual thermal change cycling .

(1400 F-T cycles). On the contrary De Kock et al. (2015) were able to detect decay of tuff specimens during only one F-T cycle using micro CT scanning technique. Tuff, however, has far lower strength and higher porosity (10-times higher) compared to andesite.

5. CONCLUSIONS

Main objective of this study was to find more effective method of detection frost resistance of harder rocks as andesite. Current practice according to (STN 72 1800) is to subject rock samples to 25 F-T cycles and calculate reduction in uniaxial compressive strength. Other methods rely on testing samples in climatic chamber determining change in relative ultrasonic velocity after certain number of F-T cycles such as relative ultrasonic velocity RV. These methods require considerable time to carry out the tests. Two types of andesite rocks with expected difference in frost resistance (Holzer et al., 2009) were tested in modified thermodilatometer VLAP 04. Fully saturated samples were tested in climatic chamber of the thermodilatometer cycling the temperature from +23 to –13 °C (average rate of cooling 0,1 °C/min) while measuring linear expansion force change (LEFC) in unconfined conditions generated by the temperature changes and subsequent ice crystallization in the pores of saturated samples. After each test a P-wave velocity, porosity, degree of saturation was evaluated. Mercury porosimetry was later used to determine the pore size distribution on studied rock samples.

Based on the test results we can conclude following:

- relative ultrasonic velocity RV appears to be more reliable method of frost resistance determination in hard rocks as andesite compared to frost resistance parameter k_2

- more distinct decrease of RV in BH specimens measured after 25 F-T cycles compared to HT suggests that higher volume of micropores influences frost resistance of samples. Higher volume of micro pores is causing lower resistance to thermal loading of fully saturated samples

- results of F-T cycle testing in modified thermodilatometer VLAP04 suggest the possibility of detection of higher amount of micropores by LEFC detection during initial cycles of testing

- LEFC measurement could potentially be a faster method of determination of rock frost resistance compared to time-consuming F-T cycling (50-100 F-T cycles)

More tests have to be carried out in order to confirm this hypothesis.

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