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GENERATION OF MICROPARTICLES IN ROCK STRUCTURES

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Abstract

Regularity has been observed in the generation of microparticles during uniaxial deformation of rocks and building concretes with the following formula: a previously unknown phenomenon in solid non-regular structures has been experimentally established and investigated, which is expressed in emission of microparticles under the action of strong uniaxial pressures. The size and amount of the fine-dispersed microfractions depend on the substance's physical-mechanical properties and pressure values. The paper presents the first results of the observed phenomenon in rock structures – granite and limestone originating from some regions of Bulgaria. The effect motivates the option for using the emission process as a new sensor method for assessment of the stress condition of solid-state systems.

Key words: particles emission, uniaxial deformation, rock structures, integrated indicator for establishing the failure events, seismic prognoses

Introduction. In recent years, the study of micro- and nano-sized particles, including the creation of nanomaterials, has been a priority field of technological research. An important direction is the determination of the physical-mechanical condition of rocks and building materials as an indicator of pending pre-destruction and destruction processes in the Earth's crust. The emission of microparticles during destruction of rocks by explosion, as well as during their exploitation, the extraction of ores and minerals, and the cutting of underground galleries and tunnels affects negatively the ecological system [1]. A great number

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of techniques and instruments for investigation of deformation condition of rock massifs have been developed [2]. The improvement of the available approaches and technologies and the designing of new ones for diagnostics and control of the condition of the rock structures is of primary importance for the extraction industry, the urbanization utilization of underground spaces, including the metro, the non-destruction control in building to determine the fidelity of gross engineering facilities such as skyscrapers, bridges, viaducts, dam walls, concrete quality, and more [2,3]. There is still another important aspect of the stress structure condition, which is related to various natural catastrophes – displacement of tectonic plates and faults. The collected information about this process expands the database of potential causes and symptoms, through which the earthquake and volcanic activity forecasting models are developed and improved.

Recently, during the activity of devised sensor systems for investigation of the strength characteristics of rock and concrete structures, a new regularity has been experimentally observed. It is expressed in the emission of nano- and microparticles in the bulk and surface of rock samples under the impact of strong uniaxial deformations [4–7]. The size and amount of these dispersed microfractions depend on the physical-mechanical properties of the substance and the values of the pressure. The paper presents the first results from the new phenomenon in rock structures originating from some mountainous regions of Bulgaria.

Experimental methodology. The major objective of the investigation is to establish the regularity by a correct and reproducible experimental method. Therefore, the approach must prove the existence of an emission process of mineral microparticles during a strong uniaxial rocks deformation. The methodology of the investigation involves the following sequences: **1.** A cylindrical, parallelepiped or cube sample is cut-out from a rock structure with occasional configuration. **2.** Within the sample, a measurement space with fixed volume is formed or the test body is placed into an airproof closed box with regular geometric form. **3.** The input of the measurement volume is connected by air filter, and its output – to the input of an electronic laser counter of aerosol particles. **4.** The sample is placed between the plates of a hydraulic press, generating uniaxial pressure from initial (zero) value to destruction one. The measurement time for particle registration is 60 s. This time interval starts after the next step of increasing the pressure. All experiments are carried out according to this algorithm.

The new regularity is characterized by a peculiarity which has been unknown so far in other physical-mechanical processes. The uniaxial deformation of the rock body displays single-direction behaviour. Even where the pressure value is far from the destruction value for the specific type of rock, the generated particles are always in exhaustion regime. Reproduction with the same sample is impossible. However, the phenomenon may be reproduced with another probe formed out of the same rock structure. The methodology here described is principally correct. It places under the same conditions during the investigation the various types of rock samples which allow to make comparative analysis of the processes substantiating the new phenomenon.

Experimental setup and its implementation. For the purpose of equivalent implementation of the described method, three configurations for measurement of the generated microparticles have been approbated. Accounting for the fact that a new phenomenon has been observed, which might arouse the interest of other researchers, we shall describe with the required details the experimental setup and used equipment. This will allow to reproduce our results, but what is more important – to deepen and develop them further in the research and application aspect.

1. The first setup includes the formation of structures with cylindrical shape, cut-out with diamond heads with diameter d: 40 mm, 60 mm and 80 mm, and height $h = 120$ mm. Laterally, in the middle of these samples, using Bernardo desktop driller, a cylindrical passage opening with diameters, accordingly, d: 4.0 mm, 6.0 mm, and 8.0 mm was drilled. An experimental session was purposefully conducted, whose methodological objective was to establish the impact of the cylindrical openings in the middle part of the samples on the strength characteristics of the structures. The ultimate strength was determined by the value of the structure's destruction deformation.

During these experiments, the ultimate strength (critical condition or destruction value) at uniaxial compression of various types of rocks with cylindrical form with and without a central passage hole was controlled. Destruction of the body with various types of rocks in two types of single type structures was observed. One specimen was without opening in the middle, the other sample was of the same material, but with a device for particle measurement (the hole) in the middle zone. It was established that, regardless of the nature of the rock probe, the diameter of the opening in the middle part within the range 4.0–8.0 mm has almost no effect on the strength properties. There were structures with which the available opening in the middle zone caused breaking of the specimens at pressure by about 5–7% lower that the pressure in structures without a hole. Such behaviour might be expected, since the opening should reduce the strength of the rock body. From the theoretical calculations in [8] it follows that, at ratio of the diameters of the probe itself and the middle zone of about 10/1, the strength of the sample will be reduced on average by no more than 5–8%. It has also been observed that, in some samples, regardless of the studied rock, the strength of the specimen with a cylindrical hole in it is higher. The reason for this is that, during the formation of the middle opening, additional distribution of the integral strength characteristics of the structure in the direction of their homogenization is achieved.

The side measurement opening was vacuumed on both sides by rubber stoppers with thin aluminium pipes passing through them, which were connected with the aerosol particles measurement system. With the device with cylindrical form, the metrology of the parameters and variables of the particles generation process results in technical difficulties, related mostly to the vacuuming of both side openings. At reaching pre-critical or critical values of the pressure for the respective sample body, the rubber stoppers fall-out of the openings. In addition, the breaking-off of the sample's surface during its deformation has significant effect on the particles emissions. In part of the experiments, the rubber stoppers were vacuum-fixed to the rock specimens by filling-in two-component Loctite epoxy glue, and in some cases – by the widely used Moment glue. It should be noted that satisfactory results were also achieved when fixing the rubber stoppers to the rock structure by the use of silicon. However, the drawback here is that you should wait quite some time until silicon polymerizes and hardens. Another problem is the measurement's accuracy, since the amount of released particles is restricted by the volume of the cylindrical hole in the body which is on average about $V \approx 1.70$–1.90 cm^3. A solution is proposed which increases by about 45% the amount of the released particle fraction, expressed in increasing the number of the cylindrical canals in the medium zone connected in parallel by hose [4] whose diameter is reduced by about 20%. In some cases, this approach deteriorates the strength characteristics of the specimen.

2. The second equipment used samples with parallelepiped form with the following dimensions: height $h = 80$ mm and equal side ribs $m = n = 50$ mm. The preparation of the specimens was made by a cutting device equipped with diamond disc. In the structures so formed, lateral cylindrical openings with diameter of 7 mm were made by a driller, which were closed on both sides by rubber stoppers, with aluminium pipes passing through them. Here again, similar behaviour with respect to the occurrence of the critical condition with and without hole in the middle zone was observed. The described metrological problems related with the correctness of the investigation of this device at reaching pre-critical condition, accompanied by falling-out of the rubber stoppers, remained unresolved.

In the described two types of systems, the cylindrical openings drilled in the middle zone of the sample bodies were flushed-out with water before the experiments to remove the fine dispersed particles, which were left during the openings' formation, after which they were dried.

3. The third equipment by which these measurements were carried out overcomes the mentioned drawbacks. It features high sensitivity and enhanced measurement accuracy and is shown in Fig. 1. The device contains a rock specimen – granite, limestone, rhyolite, dolomite, syenite, marble, slate, or of building materials – mainly concretes, in the shape of a cylinder, a parallelepiped, or a cube. The rock (building) probe was placed inside an outer hollow cylinder, cube, or parallelepiped made of hard material (preferably plexiglass) without upper and lower bottom, whose height was smaller than the height of the specimen. The two bottom zones of the external body, whose size was greater than the size of the sample, were enveloped by flexible muffs, the other two ends of which were

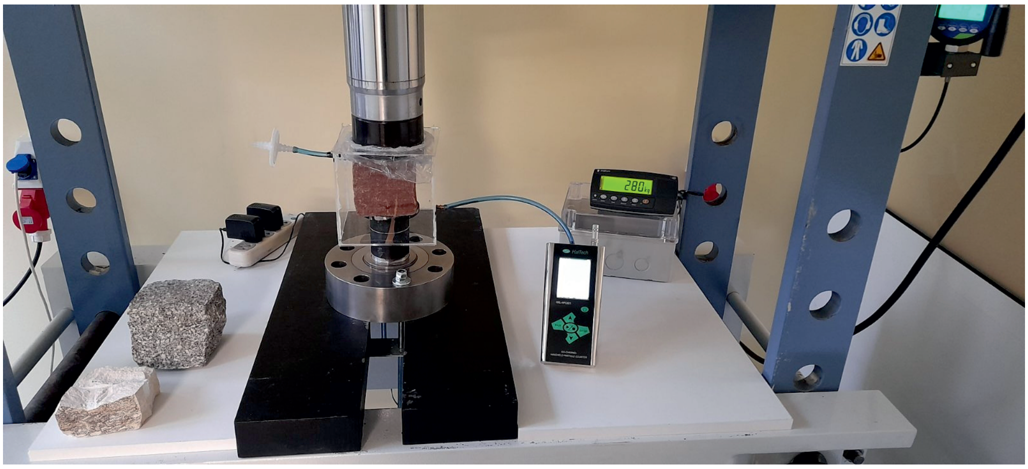

Fig. 1. System for investigating the emissions of fine dispersed mineral particles under uniaxial deformation of rock specimens

tightly fixed to the specimen by elastic rings. Close to the muffs fixed onto the external hollow space (cylinder, parallelepiped, cube), on its opposite sides, one opening each was formed to which flexible hose air-conductors were attached.

The other two ends were connected to the input of the laser particle counter and the air filter, accordingly. The specimen was placed between the two plates of the hydraulic press. The output information about the amount and size of the mineral fractions released by the deformed structure was registered on the display of the electronic counter or was fed-up to a computer system for processing and storage.

The advantages of this innovative device are as follows: **a**) high sensitivity throughout the entire experiment because of the increased size of the emitting surface; **b**) enlargement of measurement accuracy due to the drastically increased amount of generated microparticles passing through the counter; **c**) simplified construction, as a result of which there is no more need each time to drill a small-diameter opening in the rock specimens; **d**) the possibility for multiple use of the external hollow space with the fixed-onto-it two muffs, elastic rings and air conductors, thus expanding the device's use to include specimens of various size and configuration.

Experimental equipment. The experimental setup for investigating the generation of fine dispersed microparticles at uniaxial deformation of rock probes requires dedicated measurement equipment. The pressure is generated by a Bernardo BHP 200 hydraulic press featuring maximal pressure of up to 200 t. The pressure is regulated smoothly in ascending and descending mode, as well as in the mode of maintaining fixed static deformation value. The press is equipped with Sika electronic manometer with digital indication and accuracy of 0.1%. In addition, precise measurement equipment with Laumas tensosensor and accuracy of ±0.5%, provided with digital indicator and software, is applied. The innovative device is shown in Fig. 1.

To measure the emission of microparticles from the surface of the rock specimens at deformations, a new-generation electronic device of aerosol particles – Haltech Hal-HPC601 hand-held laser particle counter (laser spectrometer) is used. This instrument allows to determine the particles concentration within the range 0.3–10.0 µm. The operation of this apparatus is combined with an internal microprocessing unit (MCU), which allows measuring simultaneously six different particle sizes.

Results. The experiments were carried-out with parallelepiped specimens of limestone and granite, fixed in the system from Fig. 1 [7]. The two materials originated, as follows: the granite – from the village of Bogutevo, the Rhodopes, and the limestone – from the fault zone in Krupnik. Five identical rock probes were cut-out. The sample granite bodies with height $h = 80$ mm and equal lateral ribs sized 50 mm were subject to uniaxial deformation at constant loading velocity. By the use of the laser counter, the parameters of the emission processes of the specific type of rock were measured. Since the granite formation originated from a single specific area, the results obtained from the five specimens were averaged. This procedure was based on the fact that the obtained structure data did not vary by more than 6–8%. The strength of the samples at deformation was about 50 Mpa. The measurement of the number of particles and their disperse composition was carried out within the time interval 60 s and a constructively assumed parameter in the Haltech counter. Figure 2 and Figure 3 show the results of the experiments for limestone and granite, respectively. The tension σ in MPa, is on the x axis, and the generated number of particles in µm/cm^3 is displayed on the y axis.

The microparticles registered by the counter lie accordingly within the ranges: a) 0.3–0.5 µm; b) 0.5–5.0 µm, and c) above > 5.0 µm. The highest value of generated particles was observed in the first 8–12 s of increasing the load. As seen in Fig. 3, when the pressure nears 49 MPa, the number of granite-emitted particles in all three mentioned ranges increases faster. When the deformation reaches values of about 50 MPa, abrupt increase of microparticles emissions starts, corresponding to an occurring destructive process. At reaching a pressure close to the ultimate strength, exponential increase of the microparticles emission occurs. The behaviour and the plots obtained for limestone probes is similar (Fig. 2).

The results thus obtained prove undoubtedly the existence of the phenomenon – generation of nano- and micro-particles from rock structures sized between 0.3–5.0 µm at strong uniaxial deformations. With the increase of tension to a level preceding the specimens' destruction, the particle emission increases exponentially. The intensity of the emissions is determined by the mineral composition of the examined rocks and the degree of loading.

On the effect of microparticles generation in rock structures. The interpretation of the observed phenomenon should be focused primarily on the

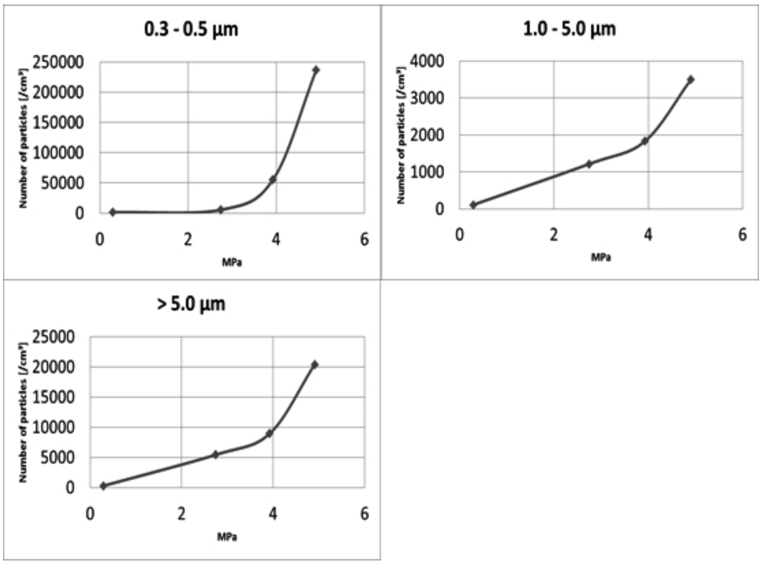

Fig. 2. Dependencies of emitted microparticles on uniaxial deformation for limestone in the ranges: 0.3–0.5 µm; 0.5–5.0 µm and > 5.0 µm

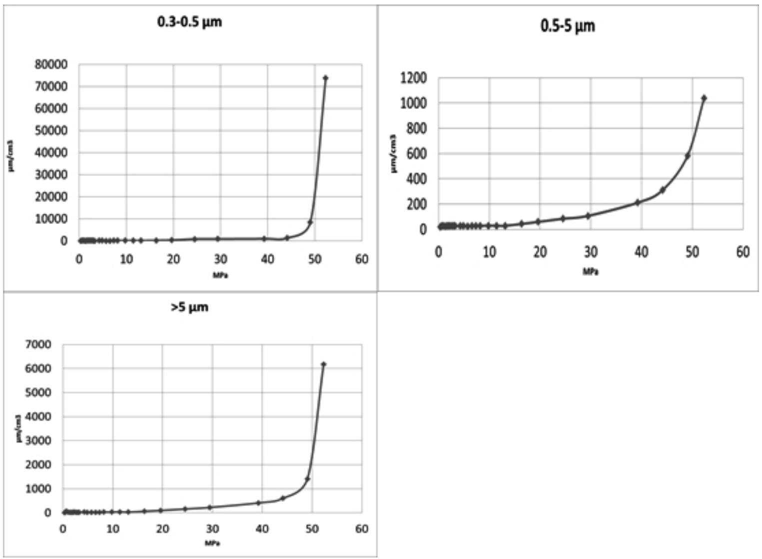

Fig. 3. Intensity of particles emission on uniaxial pressure for granite samples in the ranges: 0.3–0.5 µm; 0.5–5.0 µm and > 5.0 µm

fact that rocks, regardless of their origin, are unordered systems. Therefore, they differ substantially from the regular crystal lattices in metals. So far, no quantum-mechanical description of such complex formations is available, although the processes in this case are of quantum nature, surely. At a high uniaxial pressure, the links between the cluster formations, molecules and atoms in rock structures are deformed. The change in the location of the electrostatic connections is irreversible, in contrast to the situation with metals. This process transforms and modifies the position between the positive and negative charges. If the "elastic" reflexive behaviour at pressure with metals results from the fixed and regular location of the atoms in the crystal lattice, with rocks it is impossible to restore the condition upon deformation. The nano- and/or microcracks somewhere in the bulk or on the surface of probe form a "case" of electrically charged clusters. The released energy polarizes additionally the neighbouring molecule groups, whereas the interaction is probably achieved by Van der Waals exchange forces. The higher the pressure, the greater this interaction is. The ultimate result is generation of particles with relevant spectral composition. Initially, they leave the near-surface areas of the specimen. The plots for the respective rock sample in the three particle ranges (Fig. 2, 3), should differ due to features of the atom exchange interaction forces in the structures as observed experimentally. At reaching subcritical condition, clusters in the form of particles may be also emitted by the bulk. This model explains the availability of the exhaustion effect.

Conclusion. The key question here is the practical usefulness of the new regularity. Based on it, innovative sensor technologies for early warning, registration of pre-average and average conditions through permanent monitoring of the quantity of generated microparticles are being developed. The phenomenon serves as an innovative instrumental method for assessment of the stress level of mountainous massifs, underground and building equipment, and last, but not least, the future possibility for forecasting of catastrophic processes. The non-complex measurement system is appropriated for on-line nature investigation of the stress condition of rock structures. The results are of particular importance in the field of seismology and volcanology.

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