Temperature effect on rock properties – an example of granite, andesite and sandstone

Pavol Rybár*[1]*, Štefan Nižník*[2]*, Ľubomír Štrba*[1]*, Marek Vojtko*[2]*, Mário Molokáč*[1]*,
***Ladislav Hvizdák*[1]** *and* ***Lucia Domaracká*[1]**

New method of drilling narrow vertical boreholes by controlling hydrogen combustion assumes that rocks melt and radial fractures are formed within the rock under high temperatures and pressures at the interaction of the rock and the flame. To verify the range of formed radial fractures in different rock types, it is needed to know in detail rock and mineral properties before and after melting process of the rock(s). Rocks extend due to the heat and contract with cold. As the rocks consist of minerals with different thermal expansion, the rock extension and contraction differs in different directions. Temperature changes cause the stress between mineral grains, resulting in microfracture formations. Rock samples of granite, andesite and sandstone were pounded into fraction < 10 µm, melted and cooled under specific conditions, and analysed using REM and EDAX. Cooled granite melt contains shear fractures evolved across the sample due to the tensile stress in the quartz grains occurring in pounded sample. No fractures were observed in cooled melt of andesite. Cooling of melted sandstone grains resulted in evolution of highly porous material with no microfractures.

Keywords: *rock properties, temperature impact, melting, cooling, fracture formation*

Introduction

The article presents results of a research,which was aimed at the study of structure changes within the rock samples under high temperatures exceeding rock melt temperature, and on the determination of the phase when microfractures originate during the melting-cooling process (Kudelas et al., 2011; Rybár et al., 2011). The study is connected to the Litho-Jet project that was focused on narrow vertical drilling controlled by the oxygen burning where originated melt penetrated into radial fractures located in the surrounding rock environment (Lazar et al., 1998; Peren, 2008; Rybár et al., 2011; 2012). Taking into account previous similar studies focused on rock melting and cooling, (e. g. Rowley, Neudecker, 1985; Kenedy, Spray, 1992; Valix, Cheung, 2002; Gahan et al., 2011; Guo et al., 2012; Singh et al., 2012; Farkašovský, Zacharov, 2013) or studies devoted to the influence of temperature on the rock properties (e.g. Molaro, Mckay, 2010; Nara et al., 2010; Savoye et al., 2011; Vishal et al., 2011; Bortóns et al., 2013) this paper brings completely new data resulting from a used research method.

Preparation of the rock samples and methods

Rock samples of granite (sample V2), andesite (sample V3) and sandstone (sample V5) were subjects of the study. Before the melting process, petrographic, mineralogical and chemical characteristics of each sample used in the study were studied. Detailed knowledge of these attributes is important for further research of the resulting melt. Rock slices were studied using optical microscope NIKON AZ 100 with motorized Z-axis control and proportion of mineral phases, their character and rock texture were determined.

Subsequently, the samples were pounded into powder (fraction <= 10 µm). Powder of the selected samples with a weight of 65 g had been placed in ceramic pot before they were melted in the air atmosphere. The furnace heated for 4 hours up to 1200 °C. These temperature conditions were held for 20 minutes. Then, the furnace was cooled for 8 hours and it was opened at the temperature of 250 °C. Finally, cooled samples were studied and described using raster electron microscope and energy dispersive X-ray analysis.

---

[1] *Dr.h.c. prof. Pavol Rybár, MSc., PhD., Ľubomír Štrba, MSc., PhD., Mário Molokáč, MSc., PhD., Ladislav Hvizdák, MSc., PhD., assoc. prof. Lucia Domaracká, MSc., PhD.*, Faculty of Mining, Ecology, Process Control and Geotechnology, Technical University of Košice, Letná 9, 042 00 Košice, Slovakia, pavol.rybar@tuke.sk , lubomir.strba@tuke.sk , mario.molokac@tuke.sk , ladislav.hvizdak@tuke.sk , lucia.domaracka@tuke.sk

[2] *assoc. prof. Štefan Nižník, MSc., PhD., Marek Vojtko, MSc., PhD.*, Faculty of Metallurgy, Technical University of Košice, Letná 9, 042 00 Košice, Slovakia, stefan.niznik@tuke.sk , marek.vojtko@tuke.sk

Results

Granite

Sample of muscovite-biotite granite used in this study comes from the area of St. Martin/Mühlkreis which lies in the northern part of Austria geologically belonging to the southern part of Bohemian Massif that mainly consists of different types of granite (e.g. Weinsberg granite, Mauthausner granite, Schremser granite, Eisgarner granite), gneiss and migmatite. Two main granite types are present in the area of the St. Martin/Mühlkreis village: Weinsberg and Schlieren granite of Variscan age (Büttner, 2007).

Macroscopic description: the rock has light grey colour, it results from the quartz and feldspar occurrence. Rock texture is fine grained to medium grained, phaneritic – granoblastic, without preferred orientation of mineral particles. Minerals in the rock are macroscopically visible, with an average size of more than 0,5 mm, locally 6-7 mm. The rock has no indications of weathering or alteration. Is is not disrupted by the dislocations of macroscopic size.

Microscopic description: the rock has holocrystalline, hipidiomorphic granular structure with idiomorphic but mainly hipidiomorpic crytals of biotite and plagioclases, and xenomorphic shaped quartz and K-feldspars.

Mineral composition: prevailing mineral components of the rock are feldspars. They are mainly represented by orthoclase in the form of bigger (up to 0,5 mm) poikilitic grey grains. Orthoclase is strongly perthitized what results into microperthite and checkerboard perthite formation. The mineral hems in idiomorphic grains of plagioclases and are often flawed. Albite part of the orthoclase is up to 9 mole. %. Plagioclase creates perfect idiomorphic to hipidiomorphic oscillatory zoned crystals and has albite – oligoclase composition. In the centre is oligoclase with share of anorthite up to 24 mole %. Albites can be present on the edges. In centers, the plagioclase is often sericitized and partly saussiritized creating tiny zoisites. Alteration principally follows the fractures. Quartz in form of big irregular xenomorphic crystals is present in lesser extent than feldspars. Occasionally, wormy quartz occurs in feldspars. The quartz is undulose what refers to the pressure processes. The group of fylosilicates is mostly represented by light brown, medium to small sized, partly chloritized biotite. It contains less MgO and more TiO_2 and FeO referring to S-type granites. Light mica – muscovite occurs only in association with biotite in the form of very fine (max. 0,1 mm) leaves. Sporadically, microscopically visible opaque minerals are present. It is probably ilmenite associating with biotite. Detected accessory minerals are zircon monazite.

Features after the melting and cooling process: The granite sample melted in standard furnace is characterized by inhomogeneous structure with several phases. There were present circular and oval interstices 50 to 150 μm long (Fig. 1). An abundance of fractures occurring in the matrix, which evolved in the melted material after its cooling, as well as in the boundary of matrix and non-matrix (crystals) material testifies the fragility of this sample. The occurrence of fractures is not selective. Fractures often cross the matrix and crystals too. The origin of the fractures is due to the stress on the margins of bounded minerals with different cooling character (different rheological behaviour) creating original rock. The structure is documented by raster electron microscope (REM) in the mode of secondary reflected electrons (Fig. 2). The structure consists of brittle matrix based on Al and Si (Fig. 3) with 2 phase types.

First phase having the same colour as a matrix with size of 100 – 200 μm is mainly separated from the matrix. According to the energy dispersive X-ray analysis (EDAX), the first phase has a pure silica base (Fig. 4). It is probably non melted particle of the quartz SiO_2. Based on the number of fractures, it can be assumed that this silica phase is brittle.

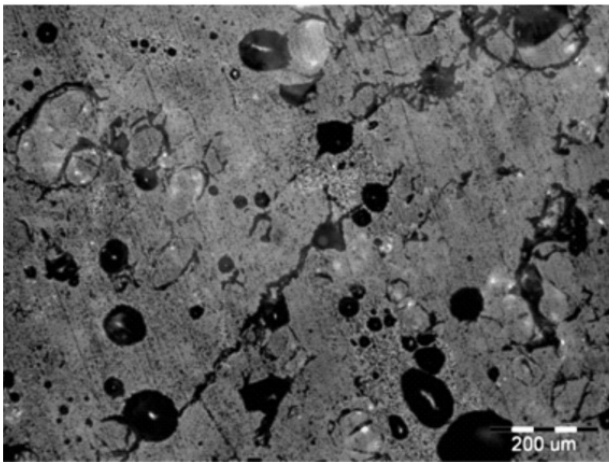

Fig. 1. The phase structure of the V2 granite sample after melting and cooling process with many fractures and bubbles – optical microscopy.

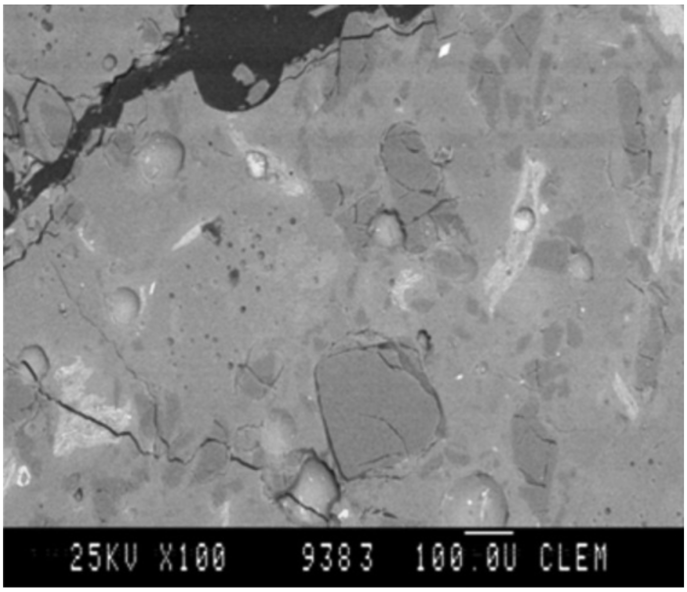

Fig. 2. The character of the phases and fractures of the V2 granite sample after melting and cooling process – REM.

Morphologically very interesting second phase consists of fine antheriferous and unidirectional ordered particles with a diameter of 1 µm and length up to 10 µm. Unidirectional orientation of particles probably resulted from no oriented stress field applied during heating and cooling process. The size of the material within this phase is various. Some extend to 300 µm. Comparison of the EDAX analysis results of the matrix and this phase (Fig. 6) shows that only iron and titanium are extra in this phase. It can be assumed that antheriferous particles are based on iron and titanium and are unidirectionally oriented in the matrix composed of Al_2O_3 + SiO_2. The result gives the impression that this is no “real” phase, but rather rectified and locally precipitated metallic particles in certain content of the matrix. The particles probably precipitated the iron as diffusion at almost solid state of the matrix material.

Other observed feature of cooled granite powder melt is the presence of “bubbles”. The surface of the bubble hollows is smooth or uneven according to the place of their origin. Smooth surface resulted from bubbles which originated in the single phase environment. If bubbles originated in the places with precipitated iron, the iron is present on the surface of such bubbles. It can be clearly visible on the Fig. 5. This fact suggests the period of bubble origins. Bubbles originated in the last stage of melting when the silica phase structure was finished.

Andesite (V3)

Amphibole-pyroxene andesite was taken from a locality in the eastern part of Slovakia, from the Slanske Vrchy Mts. Based on geological division of Slovakia (Kaličiak et al., 1991), formation Lysá Stráž – Oblik crops out in this area. The formation consists of shallow intrusive bodies of diorite porphyres and extrusive andesite bodies. In the area between Hubošovce and Kapušany, extrusive dome-shaped and lobe-shaped andesite bodies reached the surface.

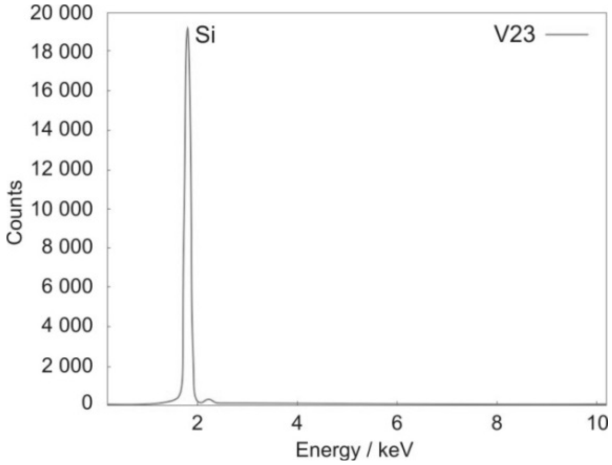

Fig. 3. EDAX matrix spectrum of the granite sample V2 - location V21 in Fig. 5.

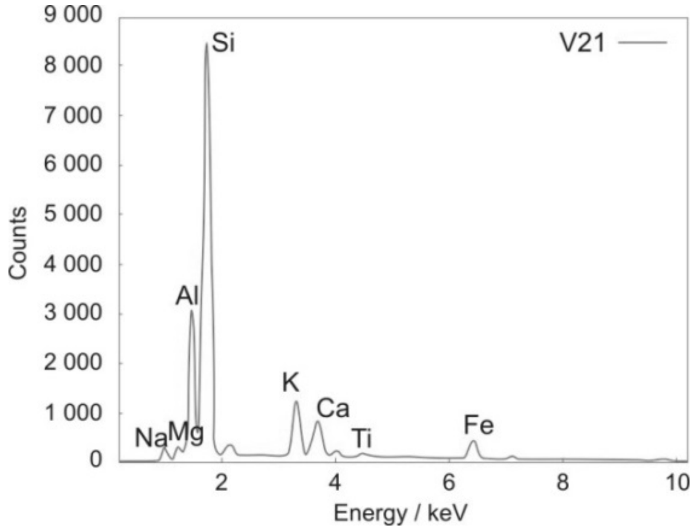

Fig. 4. EDAX phase spectrum of the granite sample V2 – location V23 in Fig. 5.

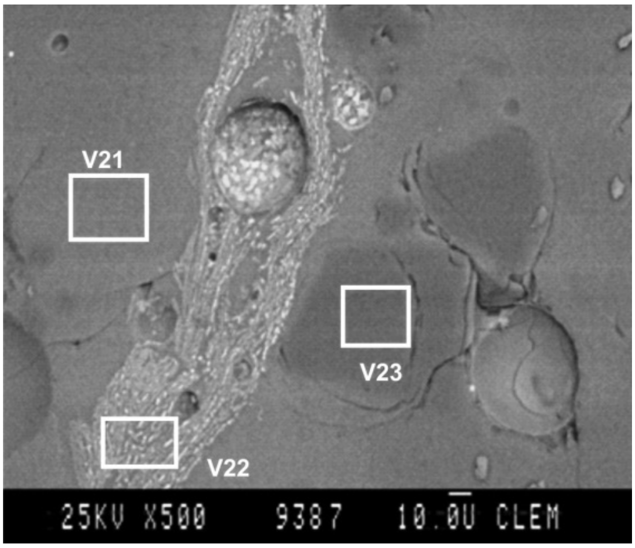

Fig. 5. Character of iron particle precipitations (part of it is marked by square V22) - REM. Squares mark the locations of the EDAX analyses.

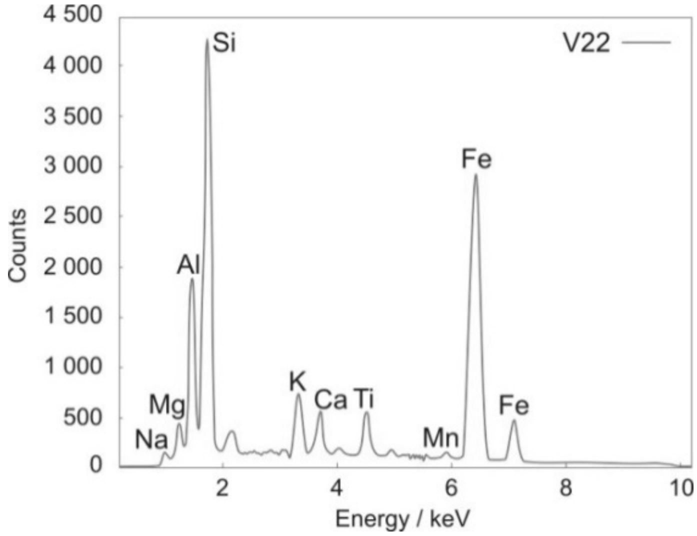

Fig. 6. EDAX spectrum of the iron phase of the granite sample V2 after melting and cooling process - location V22 in Fig. 5.

Macroscopic description: The rock is light grey with macroscopically visible phenocrysts of dark and light minerals. Average size of phenocrysts visible to the naked eye is 1-3 mm, occasionally more than 4 mm. It is not possible to identify minerals by naked eye due to the mineral sizes in the rock. Structure is massive, without pores and preferred orientation of mineral components. The rock sample has no evidence of weathering or alteration. It is no disrupted by macroscopically visible dislocations.

Microscopic description: The rock has aphanitic porphyric structure with felsitic matrix. Using optical methods, only larger mineral grains can be identified in the matrix. The rock is not disrupted by microscopical dislocations.

Mineral composition: The matrix is present in 30 – 40 % of the volcanic rock where the phenocrysts of opacitized amphibole, orthopyroxene and mainly plagioclase are present. The phenocrysts evolved from long crytalization in the magma chamber before the volcanic effusion. The matrix, which stiffened after the magmatic mash effusion, contains quartz, plagioclase, orthopyroxene and Ti-magnetite. Hipidiomrphic lamella to twinning grey-white plagioclase creating grain assemblies on several places prevails in the phenocrysts. The composition is similar to anorthite - labradorite (see chemical analyses). Matrix plagioclase has andesine composition. K-feldspar is hypersolve alkali feldspar with maximum 30 mole. % of albite component. Feldspar alteration – sericitization along fractures is partly present. Pyroxene (3-5 vol. %) phenocrysts are light and elongated with hipidiomorphic habitus. Pyroxene is often altered. Light amphibole is replaced by disintegration opacite hems, and has allotiomorphic habitus. It is Mg-hastingsite with secondary hems of actinolite. Relatively big opaque minerals are often noticed: Ti-magnetite and hematitized ilmenite.

Features after the melting and cooling process: Andesite sample melted in standard furnace differs from the sample V2 in the occurrence of fractures. No fractures were observed in the studied andesite sample (Fig. 7). Besides two phases (silica phase and phase with iron anthers) described in the sample V2, pure metallic phase occurs (Fig. 9). Its chemical composition was determined using EDAX analysis (Fig. 10), indicating occurrence of titanium and iron. Iron particles have different morphology. They precipitate as oval- or circle-shaped particles (Fig. 8).

Interstices within the sample V3 are very similar in number and size to those observed in sample V2. The character of the internal bubble surface is also similar. According to the bubble position within the sample, surfaces are clear or covered by the particles (Fig. 7).

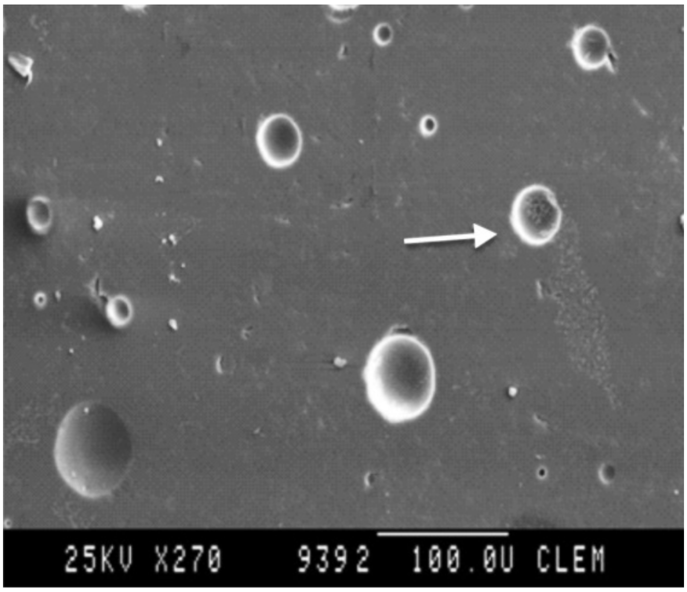

Fig. 7. Bubbles (marked by white arrow) within the sample andesite V3 – REM.

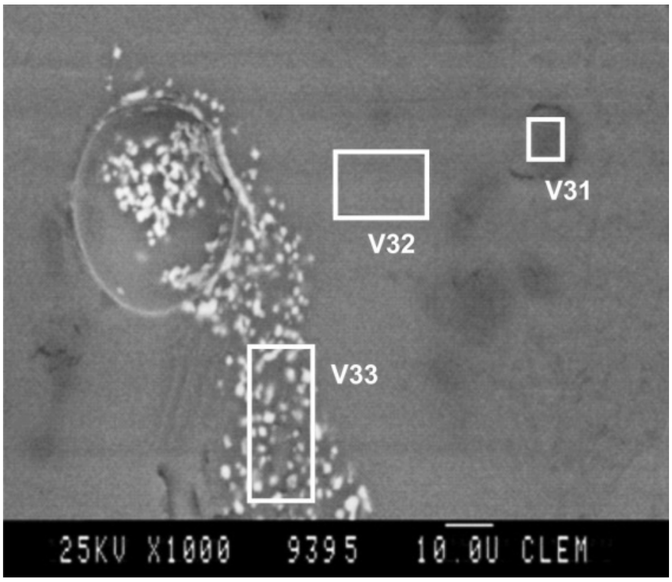

Fig. 8. Analysed locations of the andesite V3 sample – REM.

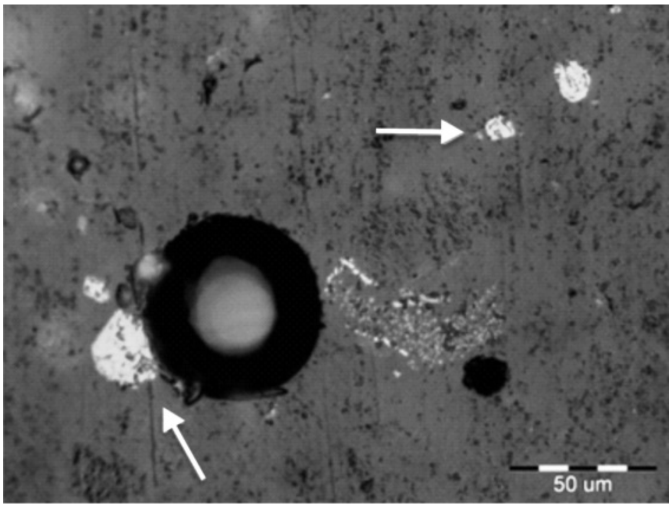

Fig. 9. Metallic phases of the andesite V3 sample after melting and cooling process (marked by white arrows) based on Ti and Fe, phase sizes: 20 ~ 30 µm.

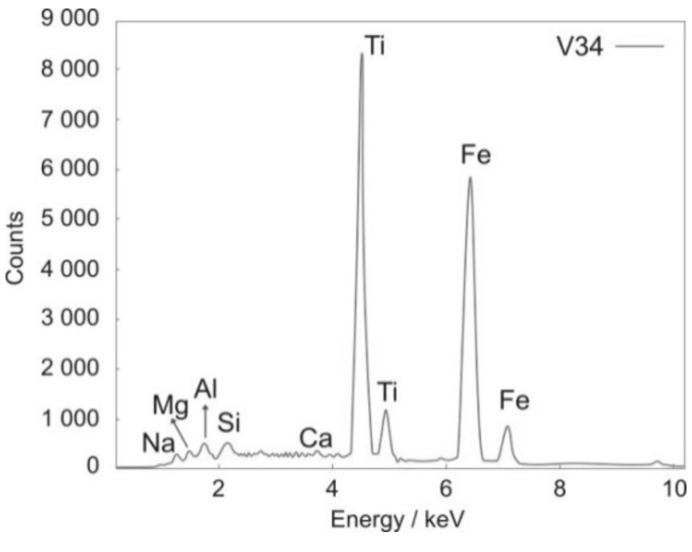

Fig. 10. EDAX spectrum of metallic phases of the andesite sample V3 from Fig. 9.

Sandstone (V5)

Geologically, the area of the Mulik quarry, from which the arkose sandstone sample used in this study was taken, belongs to the Zuberec Formation of Central Carpathians Paleogene. Sediments of this formation deposited in deep-marine environment and the flysch sequences within the formation can be characterized as typical turbidite sediments. The area of the Mulik quarry is built by the Kežmarok Member of Zuberec Formation – flysch with the occurrence of sandstone beds which consist in the total superiority of graywackes, less often subgraywackes, arkose sandstones, arkoses, sporadically limestone sandstones forming 0,5 – 2 to 3 m thick layers (Gross et al., 1999).

Macroscopic description: The rock sample of the arkose sandstone has grey-brown colour. It is a clastic sedimentary rock from the group of psammites – sandstones. It is possible to see numerous flakes of light micas which rarely reach the size of 0,35 mm. Dark clasts, up to 1,5 mm big, are also rarely present. They are irregularly placed without preferred arrange. Remaining clasts forming major part of the rock are macroscopically unidentifiable. Conglomerate accumulation, 11 cm long and 3 cm thick, was observed in the sandstone sample. Psefitic material is suboval to oval shaped. Occurrence of other conglomerate within the rock sample was not observed. Rock texture is omnidirectional without preferred clast orientations, porous. The structure is psamitic (fine grained). The rock sample has no evidence of weathering and alteration. It is not disrupted by macroscopic dislocations.

Microscopic description: the rock is silica-clastic, fine grained. It contains 10 % of the matrix formed by an alert fraction of small quartz grains, altered feldspars, micas and other unidentified components.

Mineral composition: 40 – 60% of the rock is quartz. It is extensively mechanically weathered, so it is oval shaped mainly. Lesser reworked fragments refer to close source materials which are mixed. Besides quartz, angular grains of altered feldspars (mainly plagioclase), silky leaves of muscovite and chlorite are present. Rock fragments were not detected in the sample. Chlorite is regarded as a secondary product of biotite alteration. In some places, it is possible to see carbonate aggregates - max. 5%.

Features after the melting and cooling process: The outstanding feature of the sandstone powder sample melted in standard furnace is the greatest number of interstices. From the macroscopic point of view, the sample has frothy form. Type of the structure, chemical composition of the phases and interstices character is similar to the sample V3. The matrix based on Al2O$_3$ + SiO$_2$ contains areas of clear silica phase (Fig. 11 – V52). These areas are differentiable based on colour or the occurrence of fractures which separate the phase from the matrix. Metallic particles with average size of 50 μm (Fig. 11 – V 51) are well-distributed in non-metallic matrix. The results of the EDAX analysis refer to the occurrence of titanium with an infinitesimal amount of iron. Figures 11 and 12 depict the character of the microstructure with the EDAX analysis of metallic particles. Fine metallic particles grouped into individual phase, observed in the sample V3, absent in the sandstone sample.

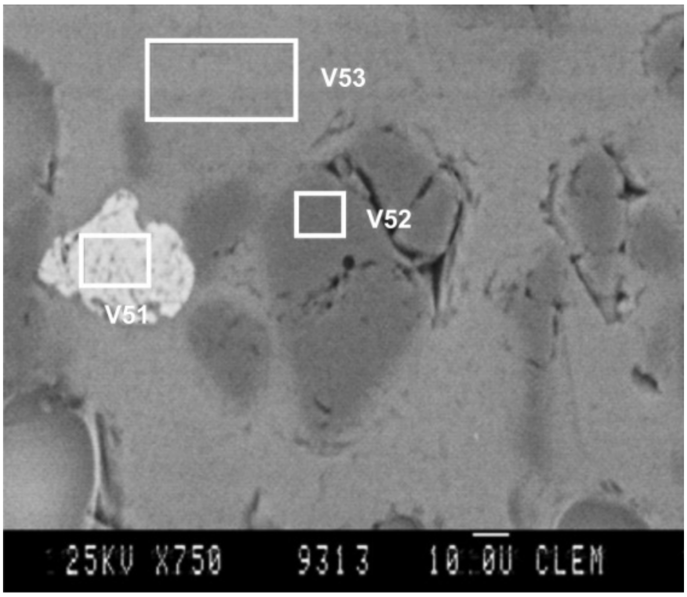

Fig. 11. REM of the sandstone sample V5: V51 – metallic particle, V53 – matrix, V52 – silica phase.

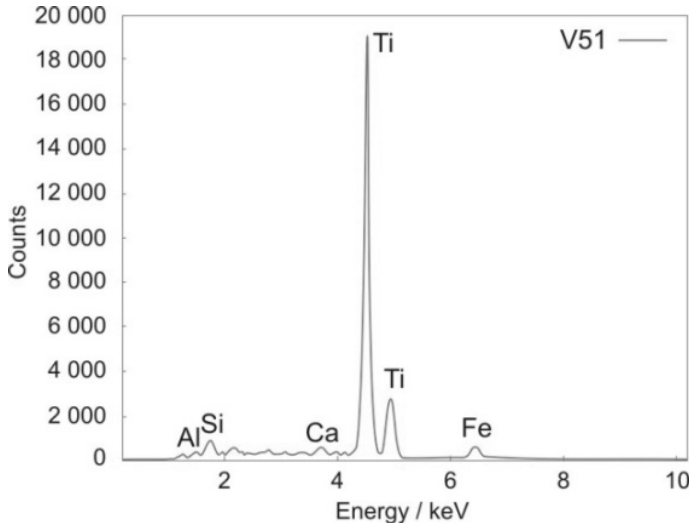

Fig. 12. EDAX spectrum of metallic phase (V51) of the sandstone sample V5 from Fig. 11.

Discussion and conclusion

The research presented in this paper brings following results. The (micro)fractures originated in tested samples based on melting and subsequent cooling in ceramic pot. Granite sample (V2) and andesite sample (V3), after melting and cooling process, contained metal particles within the matrix. Fractures in the rock samples evolved after their melting and cooling. Study of melted and cooled powder rock samples pointed out the mechanism of (micro)fracture origin. The fractures originated as a consequence of imbalanced tensile stress that resulted from uneven cooling of the melt in places where bounded mineral grains (mainly quartz grains – granite sample V2) are present in the matrix during the sample cooling. The lower number of fractures, which do not cross the whole sample, was observed in the studied andesite sample (V3). The reason is probably (1) low quartz content and (2) the fact that there was non-rectified stress applied. Melted and the cooled sandstone sample contains lot of gas resulting in appearing of the micropores. Fractures through the mineral did not originate. Transfer of the tensile stress into surroundings does not occur in such a porous environment. Only individual components can be disrupted within. According to the study results presented in this paper, it can be assumed that forming and range of fractures within melted rock samples evolved during the cooling process and is related to the amount of quartz within the sample.

The results of the study correspond to laboratory and in situ experiments of the previous research (Rybár et al., 2001), where rock disintegration at the high temperatures generated radial fractures around drilled vertical hole. Number of evolved fractures and their length within the rock sample is related to the quartz in the rock. Heating and cooling the rock, as described in this paper, may be sufficient to initiate the evolution of fractures around vertical hole thermic sinking

Acknowledgement: *This publication is the result of implementation of the project New detection methods and technologies for the exploitation of unconventional energetic Earth resources, ITMS 26220220031 supported by the Research and Development Operational Programme funded by the ERDF.*

The authors would like to thank to Igor Broska for help with the rock sample characteristics.

References

Bortóns, V., Tomás, R., Ivorra, S., Alarcón, J.C.: Temperature influence on the physical and mechanical properties of a porous rock: San Julian's calcarenite. *Engineering Geology, vol. 167, p. 117-127, 2013*.

Büttner, S. H.: Late Variscan stress-field rotation initiating escape tectonics in the south-western Bohemian Massif: a far field response to late-orogenic extension. *Journal of Geosciences, Czech geological society, Vol. 52, Issue 1-2, p. 29-43, 2007.*

Farkašovský, R., Zacharov, M.: The study of selected magmatic rocks thermic reworking for the purpose of thermic sinking. *Acta Geodyn. Geomater., Vol. 10, No. 1, 77-84*, *2013.*

Gahan, B.C., Parker, R.A., Batarseh, S., Figueroa, H., Reed, C.B., Xu, Z.: Laser Drilling: Determination of Energy Required to Remove Rock. In: *SPE Annual Technical Conference and Exhibition, New Orleans, Louisiana, 2001.*

Gross P., Buček S., Ďurkovič, T., Filo, I., Maglay, R., Halouzka, R., Karoli, S., Nagy, A., Spišák, Z., Žec, B., Vozár, J., Borza, V., Lukáčik, E., Janočko, J., Jetel, J., Kubeš, P., Kováčik, M., Žáková, E., Mello, J., Polák, M., Siráňová, Z., Samuel, O., Snopková, P., Raková, J., Zlinská, A., Vozárová, A. a Žecová., K.: *Vysvetlivky ku geologickej mape Popradskej kotliny, Hornádskej kotliny, Levočských vrchov, Spišsko-šarišského medzihoria, Bachurne a Šarišskej vrchoviny 1:50000. Geologická služba Slovenskej republiky, Bratislava, 1999. (in Slovak)*

Guo, Q., Qu, J., Qi, T., Wei, G., Han, B.: 2012, Activation pretreatment of limonitic laterite ores by alkali-roasting using NaOH. *International Journal of Minerals, Metallurgy, and Materials, Vol. 19, No. 2, 100-105*, *2012.*

Kaličiak, M., Baňacký, V., Jacko, S., Janočko, J., Karoli, S., Molnár, J., Petro, Ľ., Priechodská, Z., Syčev, V., Škvarka, L., Vozár, J., Zlinská, A., Žec, B.: *Vysvetlivky ku geologickej mape severnej časti Slanských vrchov a Košickej kotliny 1:50000. Geologický ústav Dionýza Štúra, Bratislava, 1991. (In Slovak)*

Kennedy, L.A., Spray, J.G.: Frictional melting of sedimentary rock during high-speed diamond drilling: an analytical SEM and TEM investigation. *Tectonophysics, Vol. 204, No. 3-4, 323-337*, *1992.*

Kudelas, D., Štrba, Ľ., Domaracký, D., Rybárová, M.: Physical modelling of penetration of melted rock into radial cracks in the model environment. *Mineralia Slovaca, Vol. 43, No. 3, 338-340*, *2011. (in Slovak)*

Lazar, T., Sekula, F., Rybár, P.: A new technology of sinking vertical openings into rock masses. *Metalurgija, Vol. 37, No. 4, 199-202, 1998.*

Molaro, J.L. & Mckay, C.P.: Processes controlling rapid temperature variations on rock surfaces. *Earth Surface Processes and Landforms, vol. 35, nr. 5, p. 501-507, 2010.*

Nara, Y., Hiroyoshi, N., Yoneda, T., Kaneko, K.: Effects of relative humidity and temperature on subcritical crack growth in igneous rock. *International Journal of Rock Mechanics and Mining Sciences, vol. 47, nr. 4, pp. 640-646, 2010.*

Peren, F.W.: The Litho-Jet method - the economical realisation of the geothermal power systems. *Acta Montanistica Slovaca, Vol. 13, No. 1, 1-7, 2008.*

Rowley, J.C., Neudecker, J.W.: In Situ Rock Melting Applied to Lunar Base Construction and for Exploration Drilling and Coring on the Moon. In: *Lunar Bases and Space Activities of the 21st Century. Houston, TX, Lunar and Planetary Institute, 465-477, 1985.*

Rybár, P., Kyseľová, K., Cehlár, M., Zacharov, M., Sekula, F.: Changes in the properties of remolten biotitic-quartzitic gneisses from Branisko, Slovak Republic. *Metalurgija, Vol. 40, 2001, No. 1, p. 51-55, 2001.*

Rybár, P., Kyseľová, K., Cehlár, M., Zacharov, M.: Study of the hyperstene-augitic andesite from the Ruskov Area natural form and remelting in atmospheric conditions. *Metalurgija, Vol. 41, No. 3, 99-103, 2002.*

Rybár, P., Molokáč, Š., Rybárová, M., Kopčanský, P.: Viscosity measurement of melted rocks in pressure chamber during their pressing into evolved fractures. In: *New detection methods and technologies for exploitation of unconventional energetic Earth resources, Seminar, Košice, CD-ROM, 2011.*

Savoye, S., Goutelard, F., Beauccaire, C., Charles, Y. Fayette, A., Herbette, M., Larabi, Y, Coelho D.: Effect of temperature on the containment properties of argillaceous rocks: The case study of Callovo-Oxfordian claystones. *Journal of Contaminant Hydrology, vol. 125, nr. 1-4, p. 102-112, 2011.*

Singh, U.Ch., Prasad, A., Kumar, A.: A lump-integral model for freezing and melting of a bath material onto a plate shaped solid additive in an agitated bath. *Acta Metallurgica Slovaca, Vol. 19, No. 1, 60-72, 2012.*

Valix, M., Cheung, W.H.: Study of phase transformation of laterite ores at high temperature. *Minerals Engineering, Vol. 15, No. 8, 607-612, 2002.*

Vishal, V., Pradhan, S.P., Singh, T.N.: Tensile strength of rock under elevated temperatures. *Geotechnical and Geological Engineering, vol. 29, nr. 6, p. 1127-1133, 2011.*