UDC 622.02
DOI: 10.15587/1729-4061.2019.157307

# CHANGE IN THE PHYSICAL-MECHANICAL AND DECORATIVE PROPERTIES OF LABRADORITE UNDER THERMAL EXPOSURE

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***Експериментально досліджувалися зразки з чотирьох родовищ крупнозернистого лабрадориту, який видобувається в Україні. Випробування зразків лабрадориту проводилося високими температурами 200, 300, 400, 500, 600, 700, 800, 900 °C.***

***Руді плями на поверхні зразків є результатом окислення металу $Fe^{2+}$, в різних родовищах лабрадориту вони покривають різну площу поверхні зразка природного каменю, коливається в межах 39–60 %. Аналіз полірованої поверхні лабрадориту після нагрівання показав, що руді включення рівномірно розподілені на поверхні зразків лабрадориту. Окислення мінералів, яке візуально спостерігається на всіх зразках лабрадориту, починається при температурі 300 °C. Однією з особливостей проведення досліджень, які описані в статті, є застосування обробки цифрових зображень з метою кількісної оцінки площі окислення Fe (рудих плям) на полірованій поверхні зразків лабрадориту. До температури 500–600 °C відбувається поступове збільшення окисленої площі поверхні зразків. При температурі вищій за 700 °C відбувається різке збільшення окисленої площі поверхні зразків. В цілому окислені плями металів займають від 40 до 60 % поверхні зразків лабрадориту.***

***При нагріванні зразки лабрадориту світлішають до 50 % від першопочаткового значення показника L кольорової системи Lab.***

***Зниження швидкості поширення ультразвукової хвилі в зразках лабрадориту відбувається рівномірно без стрибків. Причиною зниження швидкості ультразвукової хвилі є утворення дефектів і тріщин в зразках лабрадориту через нерівномірність теплового розширення мінералів. При температурі 700 °C і вище відбується уповільнення швидкості зниження поширення ультразвукової хвилі в зразках природного каменю.***

***При нагріванні відбувається зниження показників блиску у всіх зразках лабрадориту. В цілому при нагріванні лабрадориту до 900 °C зразки Очеретянського лабрадориту втратили 11,21 % блиску, Невирівського – 4,03 %, Осниківського – 33,57 %, Катеринівського – 15,3 %***

***Ключові слова: лабрадорит, високі температури, показники блиску лабрадориту, декоративність природного каменю, поширення ультразвукової хвилі***

## 1. Introduction

In many situations the rocks are affected by high temperatures that lead to drastic changes in their physical-mechanical properties [1]. For example, this phenomenon is observed during geothermal energy generation, in underground radioactive waste repositories, tunnels or buildings affected by fires. Research into heated rocks has become an important area in geomechanical engineering [2]. Industrial generation of geothermal energy is widely applied, which in turn puts forward new challenges to engineers and geologists. One of them is to explore the physical-mechanical properties of natural stone under the action of high temperatures [3]. Laboratory testing is an important aspect in the mechanics of rocks, which provides source data for designing engineering structures in the Earth's crust and mantle.

Wide spread are the ventilated facades that are decorated by natural stone. Although the natural stones are non-combustible materials, the effect of fire and heat can cause irreversible changes to their structure and physical-mechanical properties that influence the durability and static behavior of stone-made structures [4]. Following a fire in buildings, there is an issue related to the renovation of these facilities. In this case, it is necessary to take into consideration the change in the physical-mechanical and decorative properties after exposure to high temperatures.

Therefore, it is an important scientific and applied task to study changes in the physical-mechanical properties of labradorite under the action of high temperatures.

## 2. Literature review and problem statement

Paper [5] investigated the influence of high temperature on the physical and mechanical properties of Turkish limestone and marbles, but the authors did not study the effect of temperature on the velocity of ultrasonic wave propagation in natural stones. That would improve a possibility to diagnose the strength of natural stone by applying a non-destructive technique. Micro-defects in natural stones, which emerged as a result of heating, reduce the velocity of ultrasonic wave propagation. Thus, in [6], it was found that for limestone the dependence of ultrasonic wave propagation on temperature of heating is described by the third-order polynomials. In this case, the velocity of ultrasonic wave propagation was tested using cubic samples with the size of edges of 3 and 5 cm. This suggest that measuring the velocity of ultrasonic wave propagation contained an error because for the converter (transmitter and receiver) with a frequency of 54 kHz the base of 5 cm is not sufficient.

An experimental study into the physical-mechanical properties of sandstone and granite exposed to high temperatures [7] shows that the compressive strength, the speed of longitudinal waves and the modulus of elasticity are different macro- indicators for the physical-mechanical properties of rocks, which are not fully consistent with a change in temperature. However, this publication did not study the impact of petrographic characteristics on a change in the physical-mechanical properties of rocks exposed to high temperatures.

The difference in dependences of ultrasonic wave propagation in the samples of natural stone on heating can be explained by the difference in mineral composition, the structure and texture of stone. Up to now, a dependence of ultrasonic wave propagation in labradorite samples on heating has remained insufficiently studied.

The main factor that affects construction materials at fire is the warmth that the flame emits. This, in turn, changes the decorative properties of natural stone. Most granites [8] demonstrate an increase in the value for component L (image of a stone sample becomes brighter) in the color system CIELab. However, some samples of granite show a decrease in the value for component L. Color was measured by the spectrophotometer MINOLTA CR-200 with the lightbox D65, a diffuse beam of xenon light with a diameter of 8 mm. This method of measurement may produce errors when defining color because of the difficulty associated with determining the representative value for color in a heterogeneous material, in which the grain size may exceed the diameter of the device.

Study [9] confirms an increase in the value for component L (image of a stone sample becomes brighter) in the color system CIELab system in marbles under the influence of high temperature. Color was estimated using the colorimeter Hunter CIELAB, which has a measuring aperture of 8 mm, which reduces the accuracy of measurement. These aspects remain unexplored for labradorites.

In addition, when stone is heated, a change in color is observed, and some rocks exhibit red spots. Spots arise due to the oxidation of iron, which is contained in the minerals of rock. Large areas of oxidized iron appear at the surface of samples of gabbro [7] and limestone [6]. To a lesser extent, oxidized iron appears at the surface of granite samples [8]. However, there is no information about the quantified area of spots related to oxidized iron at the surface of stone samples. Another unresolved issue refers to a change in the color of labradorite at heating.

The above allows us to argue that it is expedient to undertake a research aimed at studying the behavior of labradorite under the action of high temperatures. That would help solve the task on predicting changes in the decorative properties and physical-mechanical characteristics of labradorite at heating.

## 3. The aim and objectives of the study

The aim of this study is to establish dependences of changes in gloss, brightness, inclusions of metal oxides at the surface of samples, as well as ultrasonic wave propagation, on a high temperature. That would make it possible to select a deposit of labradorite with the decorative and physical-mechanical properties required for tasks set by engineers.

To accomplish the aim, the following tasks have been set:

– to determine the surface area of the sample, which is taken by the oxidized metals at heating of labradorite;

– to determine the quantitative values (brightness) for component L in the color system Lab for the polished surface of samples when heated;

– to establish the velocity of ultrasonic wave propagation in labradorite samples at heating;

– to quantify a value for gloss of the polished surface of samples when heated.

## 4. Materials and methods to study the influence of high temperatures on the physical-mechanical and decorative properties of labradorite

We studied the influence of heat treatment on the physical-mechanical properties of large-grain labradorite from Ukraine, which has a violet irisation of labrador grains. We examined experimentally samples from the four fields of labradorite (Fig. 1), every deposit of labradorite was represented by 4 samples.

Samples of labradorite were heated in a furnace at a rate of 1 °C/min to the nominal temperature. A low rate of rise in temperature is used to maximize the temperature effect. The samples were heated in the electrical furnace (Fig. 2) to 200, 300, 400, 500, 600, 700, 800, 900 °C, followed by cooling to a temperature of 20 °C.

After each heating, we measured gloss of the polished front surface of samples using the gloss-meter PCE-100 GM with a measurement geometry of 20°/60°/85°. The front surface of labradorite samples was digitized using the Canon scanner CanoScan LiDE 700F. To determine the strength of natural stone and patterns in the development of cracks, we measured in a stone sample the velocity of surface ultrasonic wave propagation using the ultrasonic device UK-14MP, which is equipped with a surface sound sensor with a fixed base of 120 mm (Fig. 3). This surface sound sensor with a fixed base demonstrated its effectiveness in papers [10, 11]. We measured the propagation of ultrasonic waves along the diagonals of samples; the data acquired were averaged.

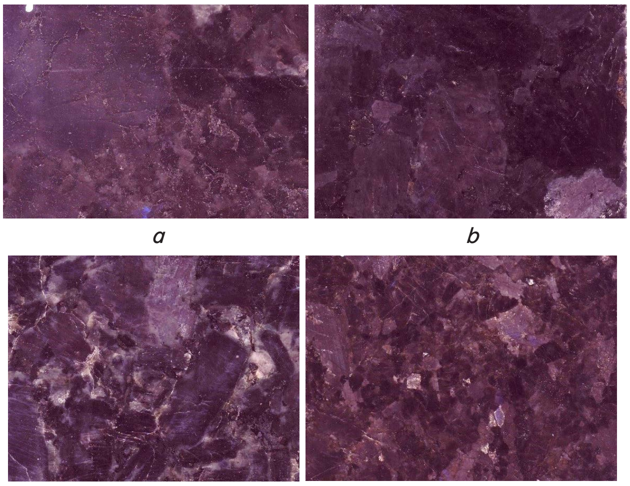


Fig. 1. Physical appearance of labradorite samples: *a* – Ocheretyansky deposit; *b* – Neviryvsky deposit; *c* – Osnikivske deposit; *d* – Katerinovsky

Fig. 2. Physical appearance of labradorite sample when heated to 800 °C


Fig. 3. Ultrasonic device UK-14MP

We counted the red stains at the surface of natural stone in the digital images of the polished surface of labradorite using the software MdiStones, whose operation algorithm is given in publication [12]. Evaluation of brightness of the images of samples of natural stone was performed using the CIELab system L component, responsible for the quantification of image luminosity. This parameter is a universal in contrast to other parameters *a* and *b* in the system CIELab, which are responsible for image coloration. Since coloration is unique for each deposit of decorative stone, it is almost impossible to compare samples of stone from different deposits based on parameters *a* and *b* in the system CIELab.

## 5. Results of estimating the stability of labradorite to high temperatures

When heated, labradorites changed their physical-mechanical and decorative properties. To a temperature of 300 °C, we observed minor changes in the color and texture of natural stone (Fig. 4, *a*). At a temperature of 400 °C, minor red spots appeared at the polished surface. At 500–600 °C, there appeared a significant number of red spots (Fig. 4, *b*). At a temperature of 900 °C, all the examined samples were covered with red spots (Fig. 4, *c*). Red spots can be explained by the oxidation of metal $Fe^{2+}$, contained in the minerals of natural stone.

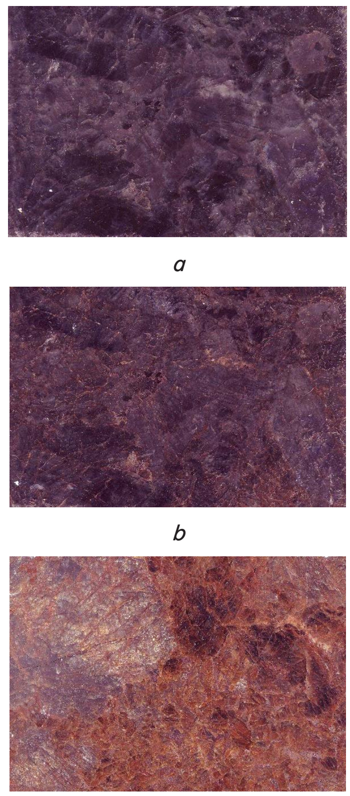


Fig. 4. Physical appearance of labradorite sample from the Ocheretyansky deposit, after heating to: *a* – 300 °C; *b* – 600 °C, *c* – 900 °C

At a temperature of 500–600 °C, one can visually observe the cracks that formed between the grains of minerals in labradorite samples.

### 5. 1. Determining the surface area of the sample occupied by the oxidized metal at labradorite heating

The heating of labradorites led to the emergence of red spots at the polished surface of samples. This is explained by a phase transformation of crystals and the oxidation of $Fe^{2+}$-elements in such minerals as pyroxene and magnetite to $Fe^{3+}$.

When computing the area of red spots relative to the total area of the sample, it was found that the area of red spots in the labradorite samples (Fig. 5) increased when heated to 300 °C. For example, for the Ocheretyansky labradorite, from 0.91 % to 4 %, for the Osnikivske labradorite, from 1 % to 3 %, and for other samples from deposits the growth amounted to 1 %. At a temperature of 400 °C, one observes an increase in red spots in labradorite samples from the Osnikivske deposit by 2.7 times, from 3 to 8 %. A sharp increase in red spots was observed in labradorite samples from the Ocheretyansky deposit at a temperature of 600 °C. At 900 °C, red spots cover the surface of the samples by 39 to 60 % of the area. The spots manifested themselves the least on labradorite samples from the Neviryvsky and Katerinovsky deposits, by 41 and 39 %, respectively. Red spots were most visible on labradorite samples from the Ocheretyansky and Osnikivske deposits, by, respectively, 60 and 46 %, due to a higher content of such minerals as ilmenite ($FeTiO_3$) and dusty magnetite ($FeO{\cdot}Fe_2O_3$).

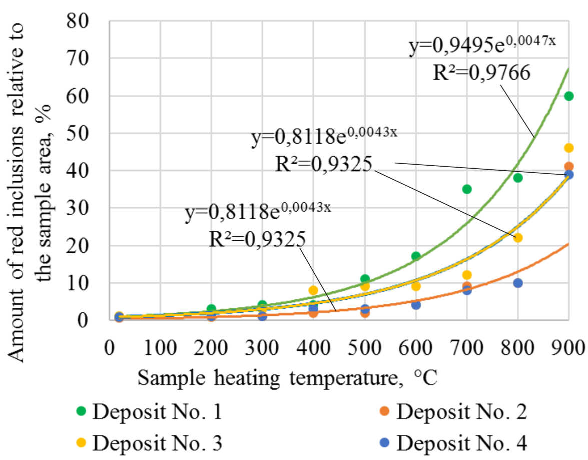


Fig. 5. Dependence of the area of red inclusions at the surface of labradorite samples on the temperature of heating: deposit No. 1 – Ocheretyansky; deposit No. 2 – Neviryvsky; deposit No. 3 – Osnikivske; deposit No. 4 – Katerinovsky

The results of this study provides an opportunity to compare labradorite deposits and evaluate the content of $Fe^{2+}$-elements in minerals. That would help select construction materials from labradorite for exterior cladding of buildings. Since at natural temperatures the oxidation of $Fe^{2+}$-elements occurs in labradorite over time under the influence of aggressive environments, red stains emerge at the surface.

**5. 2. Assessment of brightness of samples' surface at labradorite heating**

In order to establish an indicator for brightness of the samples' surface, the digital images of the surface of labradorite samples were processed by the software package MdiStones. We determined the average parameters for component L in the color system Lab, in which L is responsible for image brightness.

Fig. 6 shows that the samples become lighter with an increased temperature of heating, a sharp increase in the brightness of samples takes place at a temperature from 800 to 900 °C. The highest value for component L in the color system CIELab (the brightest image) was demonstrated by the Ocheretyansky deposit of labradorite. The darkest samples, based on the value for component L in the color system CIELab, were demonstrated by the Osnikivske and Katerinovsky deposits of labradorite.

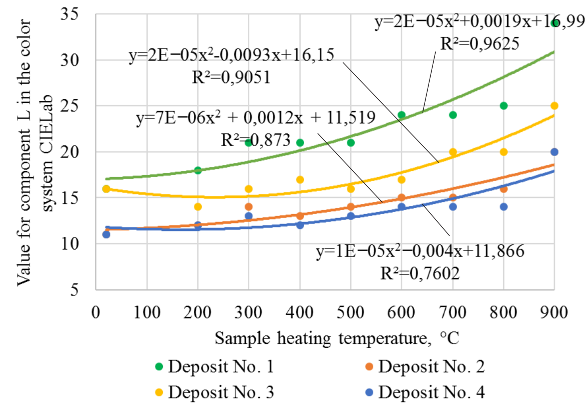


Fig. 6. Dependence of the polished brightness of labradorite samples surface on a temperature of heating: deposit No. 1 – Ocheretyansky; deposit 2 – Neviryvsky; deposit No. 3 – Osnikivske; deposit No. 4 – Katerinovsky

When heated, labradorite samples become lighter by up to 50 % of the initial values for indicator L in the color system of CIELab.

**5. 3. Estimation of velocity of ultrasonic wave propagation in samples at labradorite heating**

In the temperature range of 200–400 °C, labradorite demonstrates the disclosure of previously existing micro-cracks. The most significant changes occur in a range from 500 to 600 °C, where there is an increase in pore openness. This is due to the formation of gaps between minerals, and merging the breaks with open pores. These phenomena are caused by the anisotropy of natural stone. The dependence of propagation velocity of ultrasonic wave on temperature is almost the same in labradorite samples. A decrease in the ultrasonic wave velocity by 80 % of the initial values occurs when heating the samples to a temperature of 900 °C. At a temperature of 700–900 °C, most labradorite samples demonstrate a decrease in the velocity of ultrasonic wave. This is explained by that the number of cracks in the studied samples reaches a threshold value.

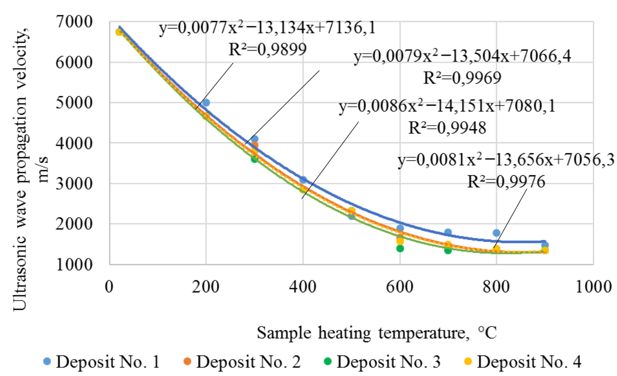


Fig. 7. Dependence of ultrasonic wave propagation velocity in labradorite samples on temperature, sample from: deposit No. 1 – Ocheretyansky; deposit No. 2 – Neviryvsky; deposit No. 3 – Osnikivske; deposit No. 4 – Katerinovsky

A change in the velocity of ultrasonic wave propagation in labradorite samples under the influence of temperature is nearly the same for all represented deposits.

**5. 4. Estimation of gloss of the polished surface at labradorite heating**

Prior to gloss measurement, the device was adjusted using a sample of black color, which is bundled with the device and has a gloss value of 97 units. We measured gloss from each labradorite sample at five points along the reference sample. The results obtained were averaged.

When investigating gloss at the polished surface of labradorite samples, it was found that at a temperature of 200 °C there is an increase in the indicators of gloss for samples from the Ocheretyansky and Neviryvsky deposits of labradorites. At a temperature of 300 °C, there is a decrease in gloss indicators for the Ocheretyansky and Neviryvsky deposits of labradorites with the further rise at a temperature of 400 °C. For samples from the Osnikivske deposit of labradorite, a decrease in gloss indicators occurs at a temperature of 400 °C and an increase at a temperature of 500 °C. This is associated with the structural changes in minerals that make up the labradorite. In general, when heating labradorite to 900 °C, samples of the Ocheretyansky labradorite lost 11.21 % of gloss, Neviryvsky – 4.03 %, Osnikivske – 33.57 %, Katerinivsky – 15.3 %.

At 400 °C, samples from the Ocheretyansky deposit became opaque, samples from the Osnikivske deposit lost their irisation, and samples from the Katerinivsky deposit demonstrated a change in ilmenite. At a temperature of 500 °C, one visually observes a better expressed gloss and cracks between minerals, samples from the Ocheretyansky deposit almost did not change, all other labradorite samples exhibited the gloss of ilmenite.

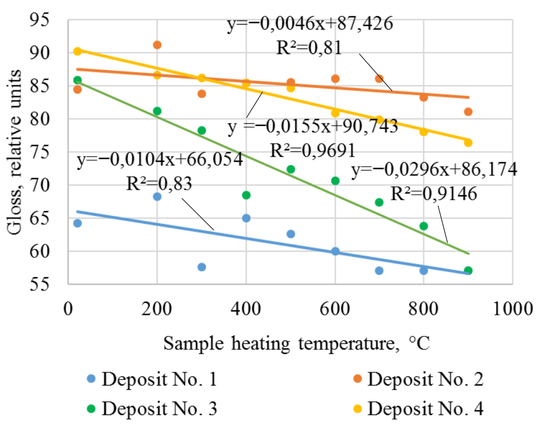


Fig. 8. Dependence of gloss on the heating temperature: deposit No. 1 – Ocheretyansky; deposit No. 2 – Neviryvsky; deposit No. 3 – Osnikivske; deposit No. 4 – Katerinovsky

Upon heating the labradorite samples to a temperature of 600 °C, the irisation was observed on samples from the Osnikivske and Katerinivsky deposits of labradorite. Red dark minerals were least observable on samples from the Ocheretyansky deposit.

## 6. Discussion of results from estimating a change in the physical-mechanical and decorative properties of labradorite under thermal exposure

When heating labradorite at atmospheric pressure, there occur the chemical reactions and transformations in the internal structure of minerals, the main of which are the phase transition and the oxidation of minerals that are rich in iron. For example, paper [13] examined changes in the physical characteristics of granite at heating due to the phase transition of quartz that occurs at 573 °C under atmospheric pressure. In contrast to granite, labradorite lacks quartz and, therefore, the oxidation of Fe contained in pyroxenes is the main source for changing the physical-mechanical properties of labradorites above 600 °C. Thus, based on a microscopic study [14], one observes that the samples of gabbro oxidize at a high temperature. It turns out that the temperature of 600 °C is the threshold to start the oxidation of minerals under atmospheric pressure. Oxidation begins as a thin coating around the pyroxenes. With an increase in temperature, oxidation develops on other crystals. At a temperature of about 800 °C, minerals that contain the inclusions of $Fe^{2+}$ are oxidized. The oxidized plagioclase looks like a red stain at the surface. Gabbro and labradorites belong to basic rocks and contain 55–98 % of plagioclase. That is why similar processes are observed at heating on labradorite samples. However, studies indicate that the oxidation of minerals, which is visually observed on all labradorite samples, starts at a temperature of 300 °C.

Fig. 5 shows that the largest number of red spots appeared at the surface of labradorite from the Ocheretyansky deposit. This is due to the presence in the mineral composition of labradorite of FeO in the amount of 4.68 %, which is the highest indicator among the represented deposits of labradorites.

The dependence of red inclusions at the surface of labradorite samples on the temperature of heating is the same for the Osnikivske and Katerinivsky deposits and is described by a power function. This is due to the presence in the mineral composition of labradorite from the Osnikivske and Katerinivsky deposits of FeO in almost the same amount, 3.91 and 3.81 %, respectively. The smallest value for red inclusions relative to the area of samples from the Neviryvsky deposit of labradorites at heating among the represented deposits of labradorites can be explained by the smallest content of FeO in the mineral composition – 1.58 %.

One of the signatures of the research described in this paper is the application of digital image processing in order to quantify the area of Fe oxidation (red spots) at the surface of the polished labradorite samples. To a temperature of 500–600 °C, there is a gradual increase in the oxidized area of samples' surface. At a temperature above 700 °C, there is a drastic increase in the oxidized area of samples' surface. In general, the oxidized metal spots take up from 40 to 60 % of the surface of labradorite samples.

Most of the previous studies were conducted when heating samples to a preset temperature level followed by testing them at room temperature. Research into the test of granite [5], limestone [6], marbles [9], and gabbro [14], shows that at a temperature to 300 °C the samples of stone are almost not damaged. However, in a temperature range from 300 to 700 °C there occur the micro-structural changes and irreversible thermally-induced cracks due to the different thermal expansion of minerals. Labradorites at a temperature of 500 °C demonstrate microcracks between the minerals.

Microcracks reduce the velocity of ultrasonic wave propagation in rock samples. For orthopyroxenes rich in Mg, D. Hugh-Jones [15] provided a wide range of thermal expansion values from $21 \cdot 10^{-6}$ to $36 \cdot 10^{-6}$ $K^{-1}$. Values of $32 \cdot 10^{-6}$ and $28 \cdot 10^{-6}$ $K^{-1}$ were also defined for orthopyroxenes and clinopyroxenes, respectively. Plagioclases are the matrix minerals for the investigated labradorite; value of $\alpha_v$ for plagioclases lies between $9 \cdot 10^{-6}$ and $15 \cdot 10^{-6}$ $K^{-1}$. It is almost two times less than the value for pyroxene. For ilmenite ($FeTiO_3$) that is contained in labradorite in the small amount from 0–5 %, $\alpha_v$ is about $10 \cdot 10^{-6}$ $K^{-1}$. Thus, the minerals of labradorite have a significant difference in the coefficients of thermal expansion. Study [14] shows that the ultrasonic wave velocity decreases with increasing temperature. The decline is moderate up to 600 °C. Between the temperature of 600 °C and 700 °C, one observes a drastic reduction in the ultrasonic wave velocity in gabbro (by 33 %), which occurs due to the rapid propagation of micro-cracks through the intercrystallite and intracrystalline composition. Labradorite does not undergo such drastic changes; some samples demonstrate minor fluctuations. The research shows that a decrease in the ultrasonic wave velocity in labradorite samples is described by second-order polynomials and all experimental samples almost do not have significant differences.

A decrease in the gloss indicators for the polished surface of marbles [9] occurs in a range from 4.22 to 24.91 %. This correlates with the results obtained in studying labradorite. A decrease in the gloss indicators for labradorite sample ranges within 4–34 %. We can distinguish two causes that affect deterioration in gloss indicators for the surface of samples of natural stone:

– the appearance of micro-cracks due to the anisotropic thermal expansion and compression of minerals, leading to chipping at the surface of the stone, which increases the surface roughness [8];

– the phase transition of minerals.

High temperatures caused changes in the appearance of labradorites. These changes are associated with mineralogy and texture. Relation between the color development and high temperatures can give information about the temperature of fire [16, 17].

In the course of the study, one observes a change in color and brightness (increasing the value for component L in the color system CIELab) in marbles [18] and granites [8]; the samples of grey granite demonstrated reduction in the value for component L.

All labradorite samples demonstrate an increase in the value for component L. Samples from the Osnikivske deposit have the largest change in the value for component L – 112 %, which can be associates with the largest value for the area of oxidized iron at the surface of the sample – 60 %. However, these samples lost 11.2 % of gloss. It might indicate that the surface of the stone was the least affected by microcracks compared with samples from other deposits. In samples from the Neviryvsky deposit the value for component L increases the least among all labradorite samples, by 56.2 %. The area of oxidized iron at the surface of the sample is 46 %, which is the average value among other examined labradorite samples. At the same time, samples from the Neviryvsky deposit of labradorite lost the largest number of gloss units – 33.57 %. We can assume that the surface roughness in samples from this deposit of labradorite increased most in comparison with samples from other deposits at the expense of microcracks. Thus, the indicators for gloss and color in component L are interrelated. The disadvantage of our research is the lack of data on the surface roughness of natural stone that could confirm the relationship between the obtained data. In addition, the further advancement of our research could be the acquisition of data on the loss of strength by labradorite under the influence of high temperatures.

## 7. Conclusions

1. Digital processing of images of labradorite samples has shown that the oxidation of minerals containing $Fe^{2+}$ occurs permanently. At temperatures up to 600 °C, this process proceeds slowly in most labradorite samples. At a temperature above 600 °C, the oxidation of metals occurs more intensively. Red spots at the surface of samples is the result of oxidation of metal compounds $Fe^{2+}$, at various deposits of labradorite they cover a different area of the sample's surface of natural stone, which varies within 39–60 %.

2. When heated, all labradorite samples become lighter to 50 % of the L indicator in the color system Lab; it is associated with a phase transition of minerals.

3. The dependence of ultrasonic wave propagation velocity in labradorite samples on temperature is almost the same. On average, there is a decrease in the ultrasonic wave velocity by 80 % of the initial values when samples are heated to a temperature of 900 °C. At a temperature of 700–900 °C, most labradorite samples demonstrate a decrease in the ultrasonic wave velocity. This is explained by that the number of cracks in the examined samples reaches a threshold value.

4. When heated, the gloss indicators for the polished surface of labradorite samples changed differently. When heated to 900 °C, samples from the Ocheretyansky deposit lost 11.21 % of gloss, from the Neviryvsky deposit – 4.03 %, from the Osnikivske deposit – 33.57 %, from the Katerinivsky deposit – 15.3 %.

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