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# ВЛИЯНИЕ УСЛОВИЙ ЗАМОРАЖИВАНИЯ-РАЗМОРАЖИВАНИЯ УГЛЕЙ НА ИХ ГРАНУЛОМЕТРИЧЕСКИЙ СОСТАВ И МЕХАНИЧЕСКУЮ ПРОЧНОСТЬ

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***Аннотация:*** Регионы Крайнего Севера и Арктики являются стратегическими для экономического развития Российской Федерации. Последнее включает в том числе добычу углей (помимо прочего, ценных марок) из месторождений, территориально расположенных в указанных регионах. Считается, что экстремальные климатические условия Крайнего Севера и Арктики с сезонным и суточным переходом температуры через ноль являются причиной снижения качества угольной продукции (образование мелочи, окисление и др.) при ее доставке до конечного потребителя. Настоящая работа посвящена исследованию влияния низкотемпературных циклических воздействий на склонность углей к разрушению и изменения их прочности в зависимости от температуры замораживания. В качестве объектов исследований были выбраны угли разных видов (каменные и бурые) из месторождений, приуроченных к регионам Крайнего Севера и Арктики. Исследовали изменение гранулометрического состава на пробах крупностью 0—3 мм и механической прочности на кусках 25—50 мм после замораживания углей при температурах –20, –40, –60 °С и последующего размораживания. Установлено, что изменение гранулометрического состава углей крупностью 0—3 мм после низкотемпературных воздействий не зависит от температуры замораживания. Обнаружено, что некоторые угли устойчивы к низкотемпературным воздействиям и не разрушаются после замораживания-оттаивания. Другие угли, напротив, подвержены разрушению, так как после замораживания до —20 °С и ниже выявлено значительное (на 20 % и более) уменьшение доли крупных частиц. Механическая прочность углей (крупностью 25—50 мм) после низкотемпературных воздействий изменяется по-разному. Угли Печорского бассейна отличаются по характеру изменения прочности в зависимости от температуры замораживания. Для одного угля наблюдается постепенное снижение прочности при понижении температуры замораживания. Другой, напротив, упрочняется при понижении температуры. Это, по всей видимости, связано с известными различиями в структуре витринита этих углей. Для углей Апсатского месторождения, отличающихся высоким содержанием витринита, установлено значительное снижение прочности только после цикла замораживания-оттаивания при температуре –40 °С. Последнее предположительно связано с образованием и разложением гидратов углекислого газа. Прочность угля того же месторождения, но с большим содержанием мацералов группы инертинита, практически не изменялась после замораживания при всех температурах. Закономерности снижения прочности бурых углей Кангаласского месторождения в зависимости от температуры замораживания обусловлены различиями в содержании в них влаги, а также интенсивностью ее потери при замораживании при разных температурах.

***Ключевые слова:*** уголь, низкотемпературные воздействия, гранулометрический состав, механическая прочность.

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# Freeze-thaw conditions effects on coals grain size composition and resistance to breakage

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***Abstract:*** Far North and Arctic zones are the strategic regions for the Russian Federation economic development. This includes the current and future mining of coals, including those of the valuable brands. Extreme climate conditions of such regions, with seasonal and daily temperature transition through zero are considered to cause the coals products losses (fines formation, oxidation, etc.) when the latter is being delivered to the end consumers. The current paper is dedicated to investigation of the effects of low-temperature cyclic impacts on coals tendency to destruction and variation of their resistance to breakage depending on the freezing temperatures. Coals of various types (bituminous and lignites) from deposits confined to the regions of the Far North and the Arctic were selected as the objects of research. The alterations of the grain size composition (on samples with a size of 0—3 mm) and resistance to breakage (on pieces of 25—50 mm) were investigated after freezing at temperatures of –20, –40, –60 °C and subsequent thawing. It was found that the change in the grain size composition of coals (with particles size of 0—3 mm) after low-temperature treatments does not depend on the freezing temperature. At the same time, it was found that some coals are resistant to low-temperature impacts and do not change after freezing-thawing. Whereas, some others (from differing origins) are prone to destruction, since after freezing down to –20 °C and lower, there is a significant (by 20% or more) drop in the proportion of large particles. The resistance to breakage of the studied coals (of particles size 25—50 mm) after low-temperature treatment changes in different ways. The coals of the Pechora basin vary in the character of resistance changing depending on temperature. For one coal, there is a gradual decrease in resistance with the freezing temperature. Another one, on the contrary, hardens with a decrease in the freezing temperature. This may be connected with the known differences between the vitrinite structure of these coals. For coals of the Apsatsky deposit, whose organic matter is dominated by vitrinite, there is a significant decrease in resistance to breakage only after freeze-thaw cycle at the lowest temperature of –40 ° C. The latter is presumably associated with the formation and destruction of carbon dioxide hydrates. The resistance to breakage of coal of the same deposit, but with larger inertinite contents, there was practically no change in its resistance to breakage observed after freezing at all considered ending temperatures. The nature of the change in the resistance to breakage of lignites of the Kangalassky deposit is due to the differences in moisture content in them, as well as the intensity of its loss during freezing at different temperatures.

***Key words:*** coal, low-temperature treatment, grain size composition, resistance to breakage.

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## Introduction

The majority of coals deposits, including those containing the most sought-after and valuable coals brands, are located in Eastern and Nothern regions of the Russian Federation. There regions include the Far North and Arctic [1]. Such regions are characterized by extreme climate conditions including seasonal and daily temperature changes with transition through zero. Thus, storage under such conditions and also transportation of coals to the consumers to the other regions could lead to the treatment of coals by the cyclic freezing and thawing. Such an impacts could reason for coals matter strength degradation [2–6] and pores size increase [7–9]. Current researches on effects of coals degradation under freezing and thawing are mostly dedicated to investigation of their permeability alteration for successful methane recovery and carbon dioxide storage [10, 11]. Whereas, there are only a few of works dedicated to coals strength losses in terms of quality of the products supplied to the end consumers. Some of the latter are dedicated to interconnections of freeze-thaw cycles and propensity to oxidation [6] and internal damage growth [12].

The current work is dedicated to the extension of the aforementioned articles and considers the effects of low-temperature cyclic impacts on coals tendency to destruction and variation of their resistance to breakage depending on the freezing temperatures.

## Materials and Methods

### *Coals characteristics*

The objects of study were coals from different deposits of the Russian Federation located in the Arctic zone and the Far North. These include 3 bituminous coals of the middle stage of metamorphism of the Apsatsky deposit (Transbaikalia) — coals No. 1 — 3, 2 bituminous coals of grade Zh (fat coals) of the Pechora coal basin (No. 4, 5), 2 lignites of the Kangalassky deposit (Yakutia) (No. 6, 7). The characteristics of the coals are shown in Table.

Coals of the Apsatsky deposit (1 — 3) were sampled from adjacent seams at the same elevations along their strike. These coals have a similar stage of metamorphism, but differ in terms of the «plastic layer thickness» (y), which determines their belonging to different brands, including coking and energy. Coal No. 2 contains more inertinite compared to coals No. 1 and 3, as well as a higher ash content.

Coals 3, 4 were sampled from one seam, but from packs that differ in their potential outburst hazard. These coals have previously been studied in terms of the relationship between vitrinite structure and propensity to destruction. It was revealed that coal 4 (sampled from a potentially outburst-hazardous seam pack) is characterized by high heterogeneity and a low degree of cohesion of the vitrinite structure, and belongs to the II genetic type according to the degree of vitrinite reduction. Coal No. 3, on the contrary, is characterized by a homogeneous structure of vitrinite and belongs to the IV genetic type. A detailed analysis of the features of the structure and properties of these coals is given in the work [13].

Lignites of the Kangalassky deposit (No. 6, 7) were sampled from two seams: lower and upper. Coals differ in the content of total moisture, volatiles contents and the calorific value on dry, ash-free basis.

### *Modes of low-temperature coals treatment*

The tests consisted of a single cyclic freeze-thaw of the samples at different ending negative temperatures: −20 °C, −40 °C, −60 °C in accordance with the freeze-thaw modes shown in Fig. 1.

For experimental work, a programmable climatic chamber «KTHV-150» (NPF Tekhnologiya, St. Petersburg) was used with the following characteristics: chamber

***Characteristics of the coals samples***

***Характеристика углей, использованных в работе***

| No. | Origin | $W_t$, % | Proximate analysis, % mas | | | $Q_s^{daf}$, kcal/kg | $S^d$, % | Petrographic composition, vol. % | | | | | $R_{o,r}$, % | Y, mm |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | $W^a$ | $A^d$ | $V^{daf}$ | | | *Vt* | *Sv* | *I* | *L* | *MM* | | |
| 1 | Bituminous coal, Apsatsky deposit | 1,0 | 0,6 | 9,7 | 22,7 | 8581 | 0,29 | 69,4 | 1,0 | 29,4 | 0,1 | 0,1 | 1,32 | 10 |
| 2 | Bituminous coal, Apsatsky deposit | 0,9 | 0,5 | 12,3 | 21,9 | 8426 | 0,22 | 56,1 | 5,6 | 37,4 | 0,1 | 0,8 | 1,36 | 12 |
| 3 | Bituminous coal Apsatsky deposit | 0,8 | 0,5 | 6,9 | 23,5 | 8668 | 0,30 | 71,0 | 1,9 | 25,5 | 0,0 | 1,6 | 1,27 | 15 |
| 4 | Bituminous coal, Vorkutaugol, Pechora basin | 1,3 | 1,2 | 7,9 | 32,3 | 8447 | 0,53 | 84,6 | 2,3 | 11,4 | 1,7 | 0,0 | 0,91 | 20 |
| 5 | Bituminous coal, Vorkutaugol, Pechora basin | 1,2 | 1,1 | 6,5 | 33,4 | 8504 | 0,47 | 77,2 | 1,6 | 18,5 | 2,6 | 0,1 | 0,90 | 27 |
| 6 | Lignite, Kangalassky deposit, upper seam, Republic of Sakha (Yakutia) | 29,0 | 8,7 | 14,8 | 49,0 | 6807 | 0,24 | 82,0 | – | 4,8 | 6,8 | 6,4 | 0,50 | n/d |
| 7 | Lignite, Kangalassky deposit, lower seam, Republic of Sakha (Yakutia) | 32,7 | 10,6 | 12,8 | 46,1 | 6729 | 0,33 | 85,6 | – | 9,2 | 3,6 | 1,6 | 0,45 | n/d |

Note $W^t$,% — total moisture content; $W^a$,% — analytical moisture; $A^d$,% — ash content on dry basis; $V^{daf}$,% — volatiles content (on dry, ash-free basis); $Q_s^{daf}$, kcal / kg – calorific value (on dry, ash-free basis); $S^d$,% — sulfur content on dry basis; *Vt, Sv, I, L, MM* — content of vitrinite, semivitrinite, inertinite, liptinite, and mineral inclusions in the coal matter; $R_{o,r}$ — vitrinite reflection index. «N / d» stands for 'not determined'.

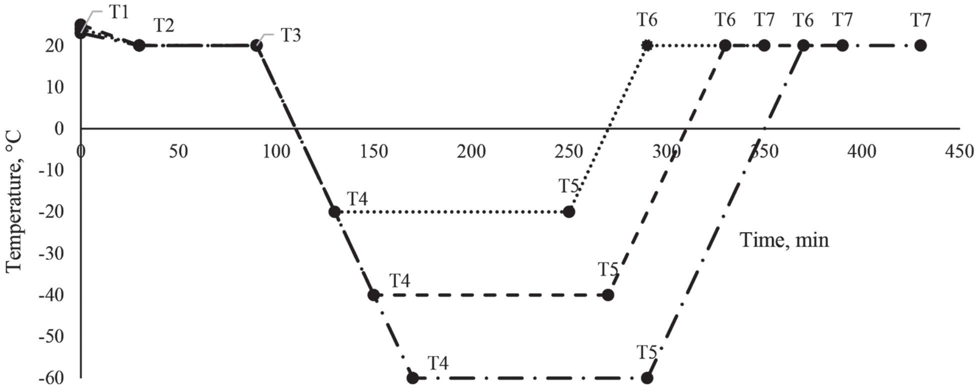

*Fig. 1. Modes of low-temperature treatment of coals*
*Рис. 1. Режимы циклических низкотемпературных воздействий на образцы углей*

volume — 150 liters, set temperature range from —70 to +150° C (temperature control accuracy ±0,5° C). The range of maintaining the relative humidity in the chamber is from 20 to 98% with a tolerance of 1 to 3%. A closed air cooling of the working area is implemented in the chamber.

The tests were carried out for two types of samples: pieces with a particle size of 25—50 mm and coal crushed to a particle size of 0—3 mm. The samples were placed in open aluminum containers in the chamber, the temperature in the chamber was stabilized at 20° C for 30 minutes. Thereafter, the temperature in the chamber was lowered at the same rate of 1° C/min to −20° C, −40° C or −60° C. A separate sample of coals was used for each final temperature. At the same time, the temperature inside the samples was recorded. When the temperature of the coal reached the predetermined negative temperature, the chamber was programmed to hold the latter for 120 minutes. After the end of freezing, the temperature in the chamber was raised at the same rate of 1° C/min for all modes to +20° C, and then the samples were left at this temperature for 60 minutes.

After testing, coal samples were placed in a sealed plastic container to prevent their oxidation.

On samples of coals with a particle size of 0—3 mm (initial and after low-temperature treatment), sieve analysis was carried out on sieves with a mesh size of 1,0 mm, 0,2 mm.

### *Tests to determine the resistance to breakage of coal samples in a tumbler*

The tests were carried out according to a procedure similar to GOST 33620-2015 (ASTM D441-07). A cylindrical tumbler with a cover, a drive shaft, a gearbox, and an electric motor was used to determine the resistance to breakage. The tumbler has an inner diameter of 200 mm, a height of 70 mm, and is made of 2 mm thick sheet steel with a polished inner surface. Inside two symmetrically arranged strips of sheet steel are welded, 70 mm long, 30 mm wide and 2 mm thick. The tumbler is closed with a lid with a felt or rubber gasket and is screwed in with four wing nuts. The tumbler is placed in a horizontal position on a support and rotates around a cylindrical axis.

For tests, samples of coal were used (initial and after low-temperature treatments) of 25—50 mm size, with a total mass of 100 to 150 g. Pre-weighed samples were placed in a tumbler, the lid was tightly closed and the rotation was turned on for 10 minutes at 52 rpm.

After turning off the tumbler rotation, samples were removed, weighed and sieve analysis was performed. The following sieves were used with mesh sizes: 10 mm; 5,6 mm; 2,8 mm; 1 mm; 0,5 mm; 0,2 mm. The remaining material after sieving on each sieve was weighed and the yield of each size class was recorded, determining its percentage in the total sample.

The resistance to breakage was calculated in accordance with GOST 33620-2015 (ASTM D441-07) as an opposite value of friability, by determining fraction of unbroken particles after testing in a tumbler.

## Influence of low-temperature effects on the grain size composition of coals and their resistance to breakage

Fig. 2 shows the particle size distribution of coal samples before and after low-temperature treatment. For the coals of the Apsatsky deposit (No. 1—3), no significant differences in particle size distribution between the initial samples and samples after freezing-thawing were revealed (Fig. 2, *a*).

For the coals of the Pechora basin, after freezing, there was a decrease in the proportion of large particles (larger than 1 mm) (Fig. 2, *b, c*) with a simultaneous increase in the proportion of particles of small classes (1—0,2 mm and less than 0,2 mm). The indicated drop in the proportion of particles of large classes for both considered coals was more than 20%. It is interesting to note that the increase in the proportion of particles of classes less than 0,2 mm for coals 4 and 5 is different. For coal from a pack that is not hazardous in terms of potential outbursts, it amounted

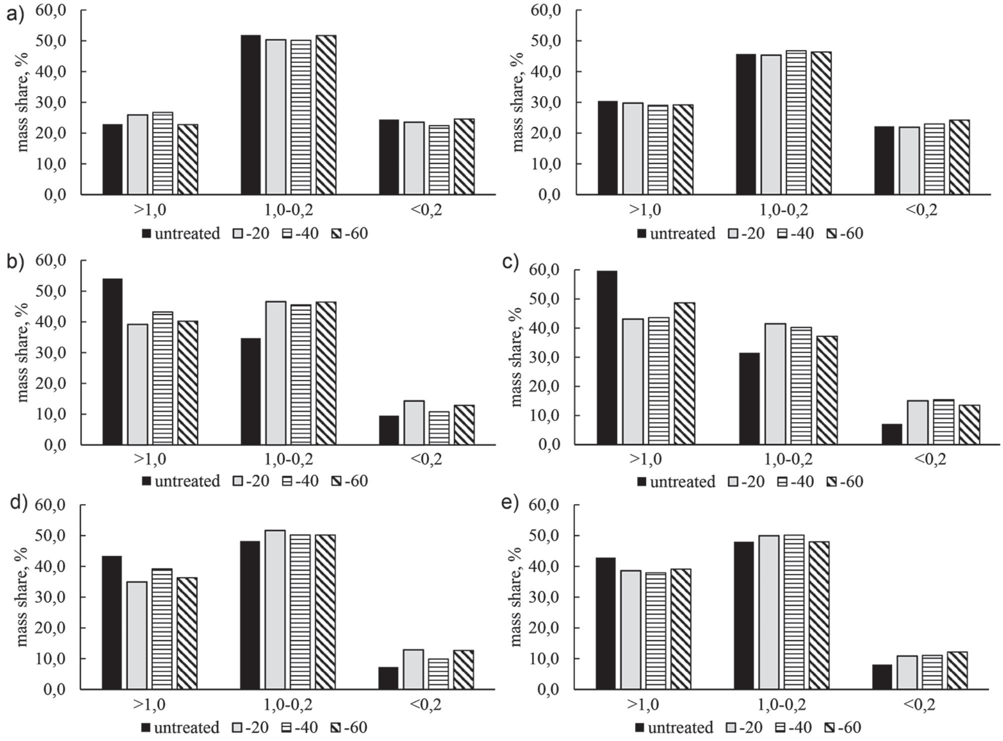

*Fig. 2. Changes in the particle size distribution of coal samples less than 3 mm depending on the processing temperature for coals: 1, 2 (a); 4 (b); 5 (c); 6 (d); 7 (e)*

*Рис. 2. Изменение гранулометрического состава проб углей менее 3 мм в зависимости от температуры обработки для углей: 1, 2 (а); 4 (б); 5 (с); 6 (д); 7 (е)*

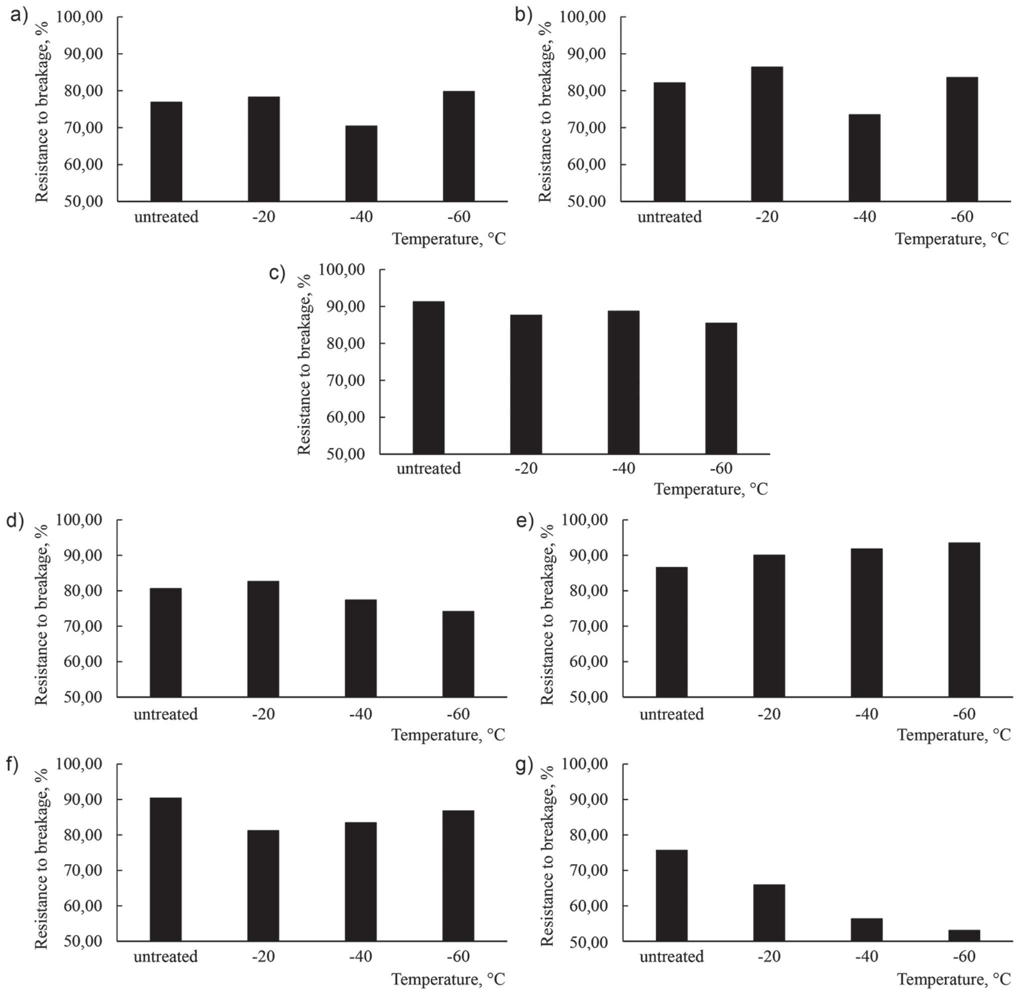

*Fig. 3. Relative resistance to breakage of coals depending on the temperature of low-temperature treatment for coals: 1(a); 3 (b); 2 (c); 4 (d); 5 (e); 6 (f); 7 (g)*

*Рис. 3. Относительная механическая прочность углей в зависимости от температуры низкотемпературной обработки для углей: 1 (а); 3 (б); 2 (с); 4 (д); 5 (е); 6 (ф), 7 (г)*

to a maximum of 50%, while for coal from a pack potentially prone to outbursts, it was over 117%. The dependence of the change in the grain size distribution on the freezing temperature for the coals of the Pechora basin was not revealed.

For lignites of the Kangalassky deposit, the change in particle size distribution is generally similar to that for the fat coals of the Pechora basin. The decrease in the proportion of the large particles for coal 6 was about 19%, for coal 7 — about 11%. The change in the yield of particles of a class less than 0,2 mm for lignites is comparable (no more than 70%, on average 50%). The dependence of the particle size distribution on the freezing temperature was not revealed also.

Fig. 3 shows graphs of the change in relative resistance to breakage for coals before and after the low-temperature treatment.

The resistance to breakage of the Pechora basin untreated coals (No. 4, 5) is relatively similar. But the change in resistance after low-temperature treatment occurs for these coals in different ways. Coal 4 is characterized by a gradual decrease in resistance with a change in the freezing temperature from −20 to −60° C, which is consistent with the concept of a change in the strength of natural materials and composites during cyclic freezing-thawing [14, 15].

For coal 5 (from a potentially hazardous pack), on the contrary, the effect of resistance gradual growth with a decrease in the lowest temperature was revealed. It is similar to the phenomenon of soil compaction under the influence of cyclic freezing-thawing. [16]. Apparently, the differences in the change in the resistance to breakage of coals 4, 5 under low-temperature treatment are due to the peculiarities of their structure. It was previously established that the vitrinite of coal 4 is homogeneous, while the vitrinite of coal 5 is heterogeneous and is represented by clastic organic matter with weak bonds between individual elements [13].

Untreated coals of the Apsatsky deposit differ in resistance to breakage. Coals 1, 3 have a similar value of relative resistance. For coal No. 2, the resistance is higher than for coal 1, 2. This is probably due to differences in the petrographic composition of these coals.

After freezing-thawing of coals 1, 3, their resistance to breakage practically does not change for temperatures of −20° C and −60° C. The significant decrease (by 20 percent or more) was observed at a freezing temperature of −40° C. Similar abrupt changes in the rigidity and brittleness of natural materials and tissues were shown in the works [17, 18]. Authors associate these observation, first of all, with porosity and other structural features of objects [17]. Presumably, these abrupt alterations of resistance could be connected with the conditions for the formation of carbon dioxide hydrates. So, under certain conditions of low temperature and pressure inside the pores, it is possible to reach the boundary point corresponding to the simultaneous occurrence of the processes of formation and decomposition of $CO_2$ hydrates [19]. Holding the sample at a given

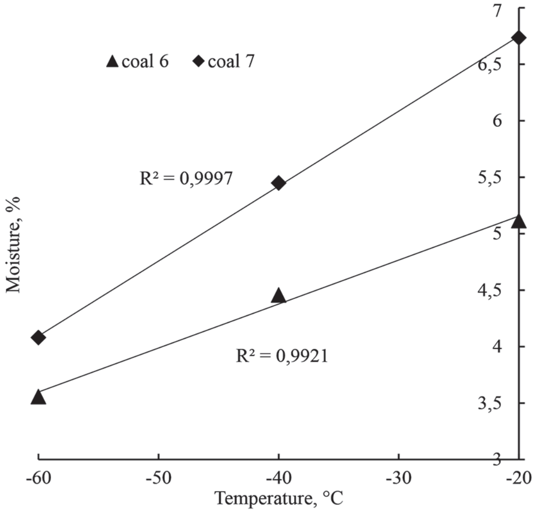

*Fig. 4. Dependence of moisture loss of lignites 6, 7 on temperature during cyclic freezing-thawing*

*Рис. 4. Зависимость потери влаги бурых углей 6, 7 от температуры при циклическом замораживании-размораживании* temperature for some time can lead to constant pressure jumps inside the pores [20]. This, in turn, can lead to the formation of a large number of disturbances within the coal matter (for example, the formation of cracks). At the same time, upon reaching lower temperatures (e.g. –60 °C), the freeze-thaw cycle does not lead to the formation of these disturbances due to a single passage of the specified temperature point.

As for coal No. 2 of the same deposit, there was no significant alteration of its resistance to breakage observed after low-temperature treatment. As it was mentioned above, this coal differs from those of No. 1, 3 in its petrographic composition. The latter may be reasoning its different behavior.

It is noted that the untreated lignites of the Kangalassky deposit are characterized by different resistance to breakage. The specified indicator for coal 6 is significantly higher than for coal 7. After low-temperature processing of coal 6 at –20° C, a drop in resistance occurs, then it practically does not change with a decrease in the processing temperature. For coal 7, the drop in the resistance values occurs linearly depending on the value of the freezing temperature. Apparently, the differences in the change in the resistance to breakage of lignites are associated with different rates of moisture loss during freezing-thawing (see Fig. 4). For coal 7, moisture loss occurs more intensively than for coal 6, which may cause the formation of disturbances (pores, cracks), leading to destruction.

## Conclusions

In this work, the analysis is given on the effect of low-temperature cyclic treatment on the particle size distribution and resistance to breakage of different types of coals.

It was found that the change in the grain size composition of coals (samples with particles size of 0—3 mm) after low-temperature treatment does not depend on the freezing temperature. At the same time, from the point of view of the redistribution of particles by size classes, the coals of the Apsatsky deposit are resistant to low-temperature impacts and do not change after freezing-thawing. Coals of the Pechora basin and the lignites of the Kangalassky deposit are prone to destruction, since after freezing down to –20 °C and lower, there is a significant (by 20% or more) drop in the proportion of large particles.

The resistance to breakage of the studied coals (with particles size of 25—50 mm) after low-temperature treatment changes in different ways.

The coals of the Pechora basin differ in the character of resistance changing depending on temperature. For coal taken from a pack that is not hazardous in terms of sudden outbursts, there is a gradual decrease in resistance with the freezing temperature. This is consistent with the known data on the change in the mechanical properties of natural and composite materials under cyclic low-temperature effects. Coal taken from a potentially outburst-hazardous pack, on the contrary, hardens with a decrease in the freezing temperature. This may be connected with the known heterogeneity degree of the vitrinite structure of this coal. This hardening effect is typical for soils located in zones subject to cyclical low-temperature impacts.

For coals of the Apsatsky deposit, whose organic matter is dominated by vitrinite, there is a significant decrease in resistance to breakage only after freeze-thaw cycle at the lowest temperature of –40° C. The latter is presumably associated with the formation and destruction of carbon dioxide hydrates. The resistance to breakage of coal 2 of the same deposit practically does not change after freezing at all considered minimum temperatures. The difference in the nature of changes in the resistance of coals 1, 3 and 2, most likely, is due to differences in their petrographic composition.

The nature of the change in the resistance to breakage of lignites of the Kangalassky deposit is due to the differences in moisture content in them, as well as the intensity of its loss during freezing at different temperatures.

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