

## РАЗРАБОТКА МЕТОДИКИ ЦИКЛИЧЕСКОГО НАНОИНДЕНТИРОВАНИЯ ДЛЯ ОЦЕНКИ СКЛОННОСТИ УГЛЕЙ К ОБРАЗОВАНИЮ ПЫЛИ

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**Аннотация:** Склонность углей к образованию пыли при добыче и транспортировке важна для изучения, так как угольная пыль является одной из основных причин возникновения аварий на предприятиях, а также опасна с точки зрения воздействия на окружающую среду. Недавние исследования выявили новую информацию о связи между хрупкостью углей и их механическими свойствами на низкоразмерных масштабных уровнях. Показано, что наноиндентирование приводит к раскрашиванию угольного вещества под индентором, то есть к образованию частиц (пыли) с размерами значительно менее площади контакта между образцом и иглой индентора. Однако традиционный метод наноиндентирования неприменим как универсальный подход для характеристики склонности углей к образованию пыли при механических и других (например, окислительных) воздействиях. В статье представлен подход к оценке изменений механических свойств углей и их склонности к раскрашиванию с образованием мелкой пыли, основанный на экспериментах по циклическому наноиндентированию. Методика включает в себя способы подготовки образцов, подходы к выбору площадок для экспериментов, условий проведения экспериментов и обработки результатов, а также их интерпретацию. Эксперименты выявили различия в склонности к раскрашиванию образцов двух антрацитов из разных месторождений РФ и природного графита. Это выявлено при исследовании изменения их жесткости и показателя нарушенности с увеличением максимальной нагрузки при циклическом нагружении. Различия в склонности образцов к образованию мелкой пыли (частиц) при механических воздействиях на малых масштабах могут быть связаны с известными данными об изменении их структуры в ряду низко- и высокометаморфизованных антрацитов, а также природного графита.

**Ключевые слова:** уголь, антрацит, графит, раскрашивание, пыль, циклическое наноиндентирование, модуль упругости, показатель нарушенности.

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### On using cyclic nanoindentation technique to assess coals propensity to fine dust formation

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**Abstract:** Coals propensity to dust release during mining and transportation is an urgent topic of research due to the fact that coal dust is one of the main reasons for various accidents and environmental impacts. Recent studies revealed new information on connections between coal brittleness and their mechanical properties at low-dimensional scales. It has been shown that nanoindentation at coals leads to their crushing under the indenter tip. But the traditional nanoindentation technique cannot be considered as a universal approach for characterization of coals proneness to fine dust formation under mechanical and other (e.g. oxidation) impacts. This paper presents an approach for assessing changes in the mechanical properties of coals and their tendency to crushing with fine dust formation based on experiments on cyclic nanoindentation. The methodology includes samples preparation technique, approaches for the selection of areas for experiments, conditions for conducting the experiments and processing the results, as well as their interpretation. The experiments revealed the dissimilarities in the tendency to crushing of samples of two anthracites from different deposits of the Russian Federation and a natural graphite. This was revealed by characterization of changes in their stiffness and the fracturing ability with increasing of maximal load during cyclic loading. The differences in the samples proneness to fine dust (particles) formation under the mechanical impacts at low scales could be connected with the known data on alteration in their structure in the row: relatively low- and high-metamorphosed anthracites and natural graphite.

**Key words:** coal, anthracite, graphite, crushing, fine dust, cyclic nanoindentation, elastic modulus, fracturing ability.

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## Introduction

Coals propensity to dust formation is one of the important issues for attempts of characterization [1, 2]. This is urgent due to the fact that coal dust is one of the main reasons for various accidents in the mining area (see, e.g. [2, 3]).

Recent studies allowed for new knowledge on connections between coal brittleness and their mechanical properties at low-dimensional scales [4, 5]. It has been numerously proved that nanoindentation at coals separate macerals leads to their crushing under the indenter tip [5]. Some parameters were identified as ones that may characterize coals ability to crushing, such as simultaneous elastic moduli  $E$  (as a measure of coal stiffness) and fracturing

ability  $R_w$  distribution heterogeneity over a selected area of vitrinite macerals [6]. This was achieved during nanoindentation at coals of the same deposit but from packs of the seam differing in their potential sudden outburst hazard [6]. But, unfortunately, the traditional nanoindentation technique is not applicable as a universal approach for characterization of coals proneness to fine dust formation under mechanical and other (e.g. oxidation) impacts. On the other hand, fine dust formation is tightly connected with irreversible changes that appear after the mechanical effects at coals and other brittle materials [7–9].

One of the most efficient mean of determination of materials ability to irreversibly deform or crush is depth-sensing

cyclic indentation [10–13]. According to this approach, the sample's surface is being loaded (with help of the indenter tip) until the specified penetration depth or load is reached, then the unloading is performed. After this, the next cycle of loading-unloading is being done at the same area of the sample's surface. The number of cycles is defined by the experimental conditions and the apparatus specification. In the view of characterization of materials ability to be destroyed, the most informative experiment is based on the cyclic indentation where at the consequent cycles the maximal loading is increased [11, 13]. According to the works of B.A. Galanov and co-authors [7–9], when indentation of brittle material is being done, a core of finely crushed material is formed under the contact area of indenter and the sample. In case of cyclic repeatable indentation when the maximal load is being increased from cycle to cycle, the area of experiment effect is widening with each cycle and, therefore, the core of deformed and/or destroyed material is being enlarged in-depth of the sample [8, 9]. Therefore, the possibility appears to calculate the degree of the crushing. The conclusion on the characteristics of the samples properties (i.e. irreversible structural changes or destruction) is made based on the diagrams 'load-penetration depth' (or P-h curve), e.g. their sharp bends, changes in the shape from cycle to cycle, also different indices that were measured based on these curves and characterizing energy losses during indentation [14, 15]. It is also important to observe the changes of the elastic modulus from cycle to cycle as the previously crushed matter (i.e. the fine particles that were already formed) may serve as a dumping agent and alter this parameter, e.g. allow it to grow gradually [16].

In the view of above, the aim of the current work is to develop a new approach to evaluation of coals ability to crushing

under the mechanical impacts on the basis of the modern technique of cyclic nanoindentation with further analysis of alteration of their mechanical properties (such as elastic moduli and fracturing ability) during increase of the loading from cycle to cycle.

### Materials and Methods

Two anthracites from different deposits of the Russian Federation were selected as samples for the study. Sample 1 — anthracite of the Omsukchan coal basin with vitrinite reflectance  $RO_r = 2.57\%$  vol., Sample 2 — anthracite of the Donetsk coal basin with vitrinite reflectance  $3.58\%$  vol. Also, a third sample was chosen representing the natural graphite originating from the same deposit as anthracite 1 and formed under similar conditions with addition of the contact and thermal metamorphism as mentioned in [17]. Its reflectance was found to be  $5.55\%$  vol. Therefore, the samples vary in their structure as indicated by the growth in the reflectance values.

The preparation of samples for the experiments was carried out in accordance with the technique described earlier in [18]. The polished sections were prepared with a nanoindentation surface oriented perpendicular to the bedding direction. This supposed not to use any binding additives and mechanical packing.

The experiments were carried out using a TI750 UBI precision triboindenter (Hysitron Inc, USA) with a built-in surface topology analyzer and TriboScan software for automated experiment and data processing.

The experiments were carried out on microcomponents of the vitrinite group (for anthracites samples). At least two different (distant) regions were selected on each sample's surface, the distance between which was at least 50 mm. In the indicated areas, zones of  $70 \times 70 \mu\text{m}$  were selected so that the surface roughness did

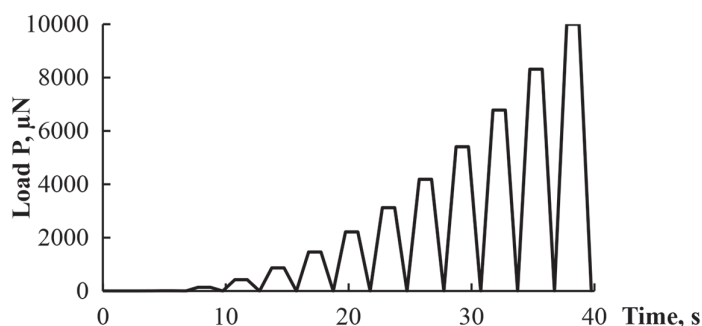


Fig. 1. Selected cyclic nanoindentation loading and unloading mode

Рис. 1. Режим циклического нагружения-разгрузки для исследований

not exceed  $0.5\text{ }\mu\text{m}$ . Further cyclic nanoindentation experiments were carried out at selected areas, at four different points.

In the loading mode, 12 steps were implemented with an exponential load increasing from  $0.01$  to  $10\text{ mN}$  (Fig. 1).

An example of the location of selected points for cyclic nanoindentation is shown in Fig. 2.

As it was mentioned above, to assess the mechanical properties of the samples and their alteration during cyclic indentation, the following indices were selected: the elastic modulus  $E$  (GPa) (characterizing the material stiffness, that is, its ability to resist the loading) and the fracturing ability  $R_w$  (%).

The elastic modulus was determined automatically for each cycle (using the built-in software of the device) from the known relations derived by Bulychev, Alekhin, Shorshorov [19] and the formula for determining the contact area between the indenter and the sample presented by Oliver and Farr [20, 21].

The fracturing ability index  $R_w$  was evaluated for each loading-unloading cycle according to the previously introduced ratios between the area of the hysteresis loop on a separate  $P$ - $h$  diagram and the calculated work of forces on loading of the sample [6]. The fracturing ability then was calculated manually using MS Excel software.

Thus, for each of the samples, a dataset of eight cyclic indentation experiments was obtained, each consisting of twelve values. The array was averaged separately for each of the twelve cycles, obtaining the mean values of the elastic moduli and the fracturing ability for each of the cycles. The values of the standard deviations determined in this case did not exceed 5% of the calculated mean values. The indices then were characterized by their alteration starting at the fourth cycle as the initial cycles were performed at the reached maximal depths values that were of the apparatus measurement error.

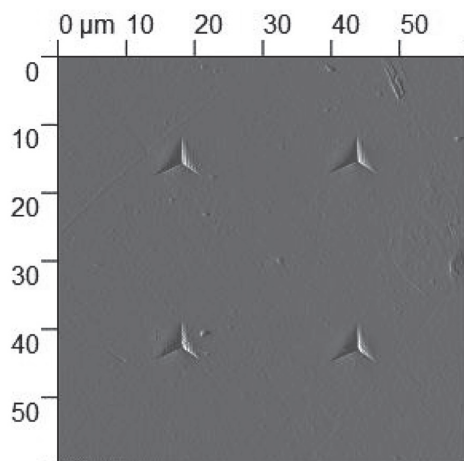


Fig. 2. Location of selected points for cyclic nanoindentation at the chosen area

Рис. 2. Позиционирование выбранных точек для циклического наноиндентирования на выбранной площадке

## Results

As the results of the experiments, we considered the change in the average values of the elastic moduli and the fracturing ability with an increase in the number of cycles during nanoindentation.

Fig. 3 shows a comparison of the change in the elastic modulus for anthracites 1 and 2 with an increase in the number of cycles.

Anthracites 1 and 2 differ, first of all, in the values of the elastic moduli. For anthracite 2 (with a higher vitrinite reflectance) they are 1.7 times larger than for anthracite 1. The character of  $E$  alteration with an increase in the number of cycles also differs. Thus, anthracite 1 ( $RO, r = 2.57\%$ ) is characterized by an increase in the elastic modulus (by 1 GPa), which presumably indicates on the successive compaction of the destroyed coal matter (i.e. fine particles) formed in the previous cycles [16].

For anthracite 2 ( $RO, r = 3.58\%$ ), the values of the elastic moduli practically do not change with an increase in the number of cycles. This presumably indicates that anthracite 1 is more prone to destruction than anthracite 2. Graphite is characterized by the largest values of elastic moduli in the considered set of the samples. But also it is characterized by increase of its measured stiffness by 3 GPa during the cycles. Such a drastic growth of elastic modulus clearly represents the fact of formed fine powder compaction during nanoindentation cycles.

Anthracite 1 is characterized by higher values of the fracturing ability as compared to sample 2 (see Fig. 4) (almost twice). Graphite has the largest values of fracturing ability index among all the samples set.

The nature of the  $R_w$  alteration for anthracites also differs with an increase in the number of cycles. For a lower metamor-

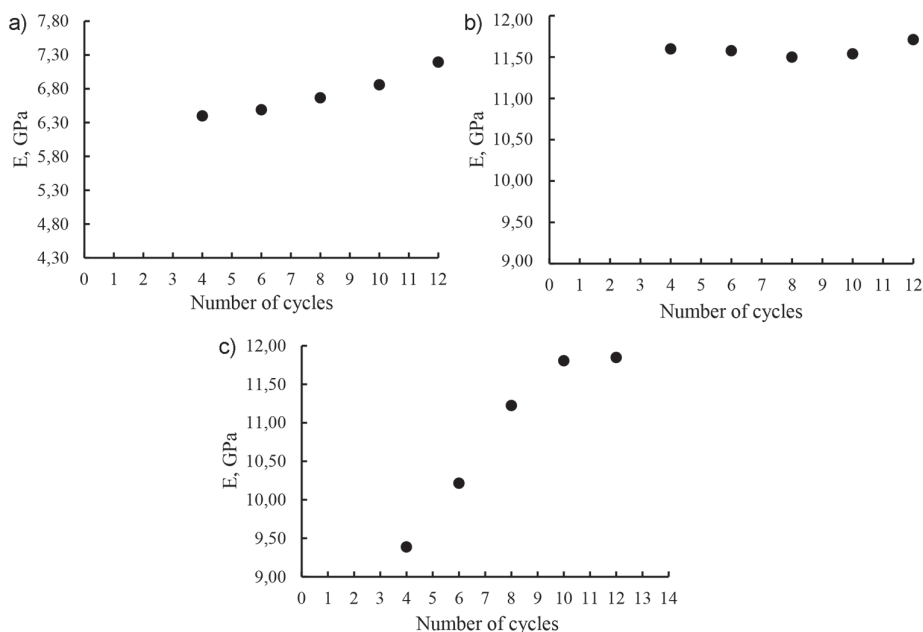


Fig. 3. Dependence of the elastic modulus  $E$  on the number of loading cycles for anthracite samples 1 (vitrinite reflectance 2.57%) (a) and 2 (vitrinite reflectance 3.58%) (b) and graphite (reflectance 5.55%) (c)

Рис. 3. Зависимость модуля упругости  $E$  от числа циклов нагружения для образцов антрацита 1 (показатель отражения витринита 2,57%) (a) и 2 (показатель отражения витринита 3,58%) (b) и графита (показатель отражения 5,55%) (c)

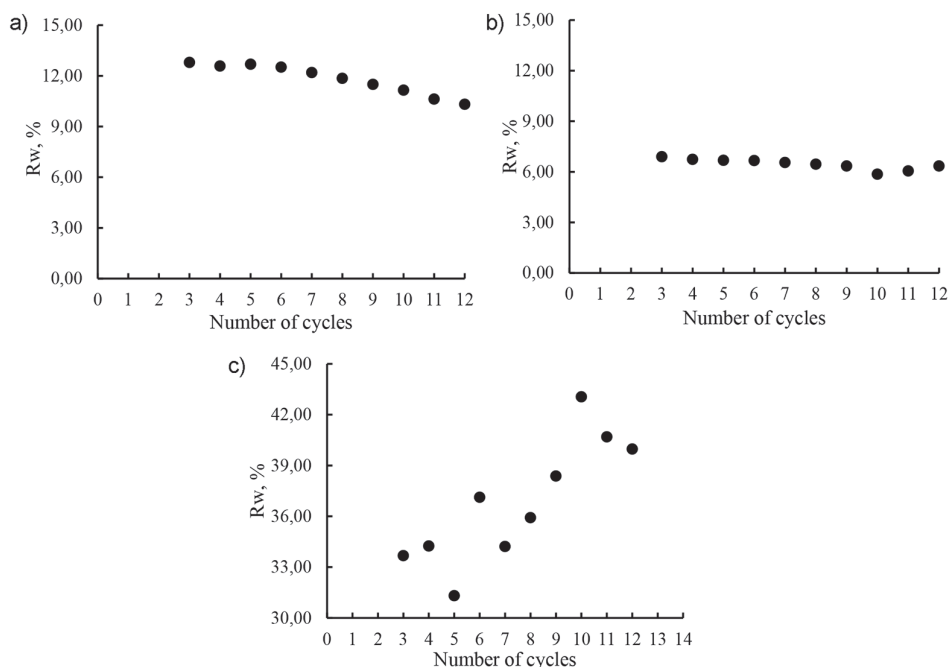


Fig. 4. Dependence of the fracturing ability  $R_w$  on the number of loading cycles for anthracite samples 1 (vitrinite reflectance 2.57%) (a) and 2 (vitrinite reflectance 3.58%) (b), graphite (reflectance 5.55%) (c)

Рис. 4. Зависимость показателя нарушенности  $R_w$  от числа циклов нагружения для образцов антрацита 1 (показатель отражения витринита 2,57%) (a) и 2 (показатель отражения витринита 3,58%) (b) и графита (показатель отражения 5,55%) (c)

phosed anthracite 1, the fracturing ability gradually decreases from 13% to 10%. For anthracite 2, on the contrary, it practically does not change, with fluctuations in the range of 6–7%. As of graphite, its fracturing ability tends to an increase from 30 to 45% which clearly indicates on its further crushing from cycle to cycle.

From the presented data, a preliminary conclusion can be made that the cyclic loading of anthracite 1 leads to the destruction of its structure at the initial cycles, when the release of fine particles could be found. Further, they are compacted with an increase in the applied load, preventing its further destruction. This is shown by both an increase in the elastic moduli (that is, the stiffness of the material) and a gradual slow decrease in the fracturing ability values. For the highly metamorphosed anthracite 2, cyclic loading, presumably,

does not lead to significant destruction, as indicated by the practically invariability of its mechanical characteristics under further loads. Therefore, this sample is less prone to destruction with formation of fine particles. Graphite, on the contrary, has shown its high proneness to fine particles formation under repeated loading.

The differences in the samples proneness to fine dust (particles) formation under the mechanical impacts at low scales could be connected with the well-known differences in their structure described by their reflectance values and optical anisotropy growth [22, 23].

## Conclusions

As it was previously observed, the traditional nanoindentation technique is not applicable as a universal approach for characterization of coals proneness to fine



dust formation under mechanical and other (e.g. oxidation) impacts.

This paper presents an approach for assessing changes in the mechanical properties of coals and their tendency to crushing based on experiments on cyclic nanoindentation. The methodology includes samples preparation technique, approaches for the selection of areas for experiments, conditions for conducting the experiments and processing the results, as well as their interpretation.

On the basis of the developed methodology, the dissimilarities in the tendency

to crushing of samples of two anthracites from different deposits of the Russian Federation and a natural graphite were found. This was revealed by characterization of changes in their stiffness and the fracturing ability with increasing of maximal load during cyclic loading. The differences in the samples proneness to fine dust (particles) formation under the mechanical impacts at low scales could be connected with the well-known differences in their structure described by their reflectance values and optical anisotropy growth.

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## РУКОПИСИ, ДЕПОНИРОВАННЫЕ В ИЗДАТЕЛЬСТВЕ «ГОРНАЯ КНИГА»

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### ОБЕСПЕЧЕНИЕ БЕЗОПАСНОСТИ ЭЛЕКТРООБОРУДОВАНИЯ ОЧИСТНЫХ СООРУЖЕНИЙ СТОЧНЫХ ВОД

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На основе нормативных документов приводятся рекомендации обеспечения электробезопасности электрооборудования систем очистных сооружений. Даны описание очистных сооружений и характеристика условий работы электрооборудования. Приведена структура системы обеспечения электробезопасности с описанием способов и средств защиты от поражения электрическим током.

Ключевые слова: очистные сооружения, электрические сети, напряжение прикосновения, электробезопасность, электрооборудование, способы и средства защиты.

## ENSURING SAFETY OF ELECTRICAL EQUIPMENT OF WASTEWATER TREATMENT PLANTS

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On the basis of regulatory documents, recommendations are given to ensure the electrical safety of electrical equipment in treatment facilities. The description of treatment facilities and characteristics of the operating conditions of electrical equipment are given. The structure of the electrical safety system with a description of the methods and means of protection against electric shock is given.

Key words: treatment facilities, electricity of the net, touch tension, electrical safety, electrical equipment, methods and means of protection.