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Hydrophobic Interactions in the Diamond–Organic Liquid–Inorganic Luminophore System in Modification of Spectral and Kinetic Characteristics of Diamonds

V. V. Morozov^a, V. A. Chanturia^a, G. P. Dvoichenkova^{a*}, and E. L. Chanturia^a

^aAcademician Melnikov Research Institute of Comprehensive Exploitation of Mineral Resources–
IPKON, Russian Academy of Sciences, Moscow, 111020 Russia

*e-mail: dvoigp@mail.ru

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Abstract—The authors studied theoretically and experimentally the compositions of luminophores in modification of spectral characteristics of anomalously luminescent diamonds to the effect of their recovery. Attachment of a luminophore-bearing composition at the diamond crystal surface takes place owing to the stable aggregation of the diamond, organic liquid and inorganic luminophores by the mechanism of hydrophobic interaction in polar medium. Stability of such aggregates is ensured by intense adhesion of the components having similar surface energy. For diamond and zinc sulfide, the dependence of the wetting angle, generated by the organic liquid drops in the water medium, on the surface tension of the organic phase exhibits the extreme nature. By the coordinate of the maximum of the wetting angle–organic liquid surface energy curve, the surface energy of diamond and zinc sulfide is determined. It is proved that the organic liquids and oil products, which are used as organic collectors and have surface energies similar to the luminophore surface energy, maximize the force of hydrophobic interaction and stability of the diamond–organic liquid–inorganic luminophore aggregate.

Keywords: Diamonds, X-ray luminescence separation, luminophores, composition, organic collector, hydrophobic interactions, spectral and kinetic characteristics, modification.

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INTRODUCTION

A promising way to solve the problem of reducing the loss of weakly and anomalously luminescent diamonds in the process of X-ray luminescence separation is the development and implementation of a technology for modifying their spectral characteristics using specially selected compositions of luminophores [1]. The technology for modifying spectral and kinetic characteristics of weakly and anomalously luminescent diamonds is based on the effect of concentrating specially selected luminophore mixtures in an organic liquid with its subsequent attachment at the diamond surface [2]. Increasing the stability of the heterophase diamond–organic phase–luminophore complex is the main condition for the effectiveness of the developed technology. Both the extraction of luminophore with organic liquid and subsequent attachment of the luminophore-bearing composition at the diamond surface belong to the group of hydrophobic interactions in a polar medium and obey similar patterns. [3, 4].

This paper aims at developing approaches and procedures for selecting liquid organic collectors that provide the necessary stability of luminophore-bearing compositions, their effective attachment at the diamond surface and preservation of the required spectral and kinetic characteristics.

1. GENERAL APPROACH TO SELECTING ORGANIC LIQUID COLLECTORS FOR LUMINOPHORE-BEARING COMPOSITIONS

The processes in the working environment during the preparation and application of a luminophore-bearing composition can be considered from the standpoint of the theory of hydrophobic interactions. Hydrophobic interaction is the attraction between non-polar and weakly polar particles in water or other polar solvents, which is governed by thermodynamic disadvantage of water contact with non-polar substances [5]. Hydrophobic interactions arise in water solutions due to the interaction of polar water molecules with non-polar hydrophobic particles, molecules or non-polar radicals of molecules. For condensed media, the concept of hydrophobic interaction is related to the adhesive interaction between two hydrophobic substances immersed in water [6]. The energy of hydrophobic interaction, which determines the attraction between macroscopic hydrophobic surfaces in water, is governed by the interfacial energy per unit surface, and is called surface tension [7].

The surface tension of organic liquids used as the basis of a luminophore-bearing composition depends significantly on their chemical structure. Surface tension differs for various hydrocarbon groups: it is maximum for aromatic and minimum for paraffinic. As the molecular weight of hydrocarbons increases, the surface tension rises [8]. When the temperature and pressure rise, the surface tension in the gas-liquid system decreases [9].

The surface tension of organic compounds is significantly lower than that of pure and tap water that does not contain surfactants (Table 1), which is the reason for the reduction in the contact area of these liquids during interfacial processes. The surface tension of diesel fuel and other oil products used as an extractant for luminophores is a variable depending on the amount of surface-active components contained in them. High molecular organic liquids and refined oil products have a fairly high surface tension at the interface with water. As the concentration of surfactants increases, the surface tension of the organic liquid decreases. Natural surfactants that reduce the surface tension of oil and oil products are alcohols, phenols, resins, asphaltenes, and various organic acids [8]. The surface tension of organic substances obtained from oil products can be estimated by calculation [9].

An important factor in assessing the stability of disperse systems is the consideration of dynamic factors, e.g. strengthening of surface tension at the particle-bubble contact under variable loads in a turbulent medium, discovered for flotation processes by V. I. Melik-Gaikazyan [13]. Based on the foregoing, when selecting an organic liquid that extracts and retains a luminophore, it is expedient to be guided by the parameter of interfacial energy per unit surface, i.e., surface tension.

Table 1. Surface tension (surface energy of the substance-vapor interface) of water and organic compounds [10–12]

| Substance | Surface tension, mJ/m ² | Substance | Surface tension, mJ/m ² |
|----------------------|------------------------------------|--------------|------------------------------------|
| Water | 72.5–72.8 | Oil | 26–30 |
| Tap water | 68.0 | Gasoline | 21.0 |
| Pyridine | 36.5 | Kerosene | 24.0 |
| Aniline | 44.0 | Oil fuel | 45–48 |
| Carbon disulfide | 37.7 | Diesel fuel | 30.8 |
| Carbon tetrachloride | 28.0 | Vaseline oil | 31.8 |
| Formamide | 52.7 | Motor oil | 41–48 |
| Phenol | 36.5–42.3 | Hexane | 18.5 |
| Benzol | 29.0 | Octane | 21.3 |
| Toluene | 36.1 | Decane | 23.9 |
| Glycerine | 59.4–63.4 | Hexadecane | 27.6 |

2. PROCEDURES FOR STUDYING AND MODELING THE PROCESSES OF FORMATION AND USE OF LUMINOPHORE-BEARING COMPOSITIONS

To analyze the mechanism of interaction between diamonds, luminophores and organic liquids in the water medium, the method of luminescent and combined microscopy was used. Reflected-light luminescent microscope is the most common method for observing and recording images of macro- and micro-objects in the light of fluorescence [14]. In these studies, Micromed 3 LUM luminescent microscope was used.

The energy characteristics of hydrophobic interactions were evaluated by measuring the wetting angles [15]. The surface of minerals was treated with the water system under study, a drop of organic matter was applied, and the wetting angle was measured. The measurements were carried out on an OCA 15EC Package 1 setup containing a main instrument with a single SD-DM direct dosing module, an ESR-N electronic syringe module, and a SCA 20 software module for drop contour analysis. The wetting angle was determined from the image of an organic liquid drop on a flat area of the diamond surface immersed in the aqueous phase.

The efficiency of luminophores attachment on the surface of diamonds and kimberlite minerals was evaluated by the image of objects studied in ultraviolet radiation. To obtain images of diamonds and kimberlite minerals after treatment with luminophore-bearing media, a Luch-1F luminoscope equipped with ultraviolet radiation sources having a wavelength of 273 and 365 nm was used. The obtained images were diagnosed by a visiometric analysis, which allowed estimating the concentration and distribution of luminophore over the surface of analyzed samples [16].

Devices that record the final X-ray luminescence signal are traditionally used to determine the results of modifying the diamond surface with luminophore-bearing compositions. In these studies, a portable separator Polyus-M which reproduces the settings of industrial XRF separators was used [17]. The separator has a luminescence-absorption analysis mode, which assumes the location of the X-ray tube and the luminescence receiver on opposite sides of the material flow. The analog-to-digital recording system made it possible to work both in the amplitude-time and in a special mode of increased selectivity, using the kinetic characteristics of the X-ray luminescence signal: convolution, decay time constant, slow component amplitude, fast component amplitude, ratio of component amplitudes.

3. MACRO MODELING AND STUDY OF HYDROPHOBIC INTERACTIONS IN THE DIAMOND–ORGANIC LIQUID–INORGANIC LUMINOPHORE SYSTEM

Stable heterophase diamond-organic liquid-inorganic luminophore system is obtained by intense hydrophobic interactions between the components of the system. The desired result can be achieved by selecting or modifying their properties to ensure efficient extraction of an inorganic luminophore with an organic liquid and adhesion of luminophore-bearing composition (a mixture of luminophore and organic collector) on diamonds.

Attachment of a luminophore-bearing composition at the diamond crystal surface includes the stable aggregation of the diamond, organic liquid and inorganic luminophores (Fig. 1). Drops of an organic liquid with volumetrically or surfacely extracted luminophore that attached at the diamond crystal surface are the source of the X-ray luminescence signal, which modifies the diamond's own signal.

The group aggregation of diamonds, in which individual crystals are interconnected by a luminophore-bearing liquid, was confirmed by the results of luminescence microscopy (Fig. 2a). After drying, the luminophores contained in the organic liquid are stably attached at the diamond crystal surface (Fig. 2b).

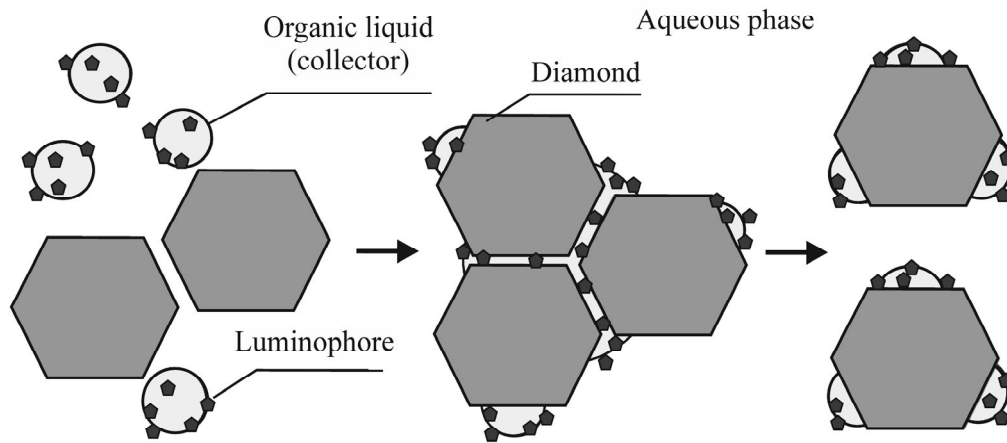


Fig. 1. Attachment of luminophore-bearing composition at diamond crystal surface.

In case of using compositions with inorganic luminophores that are insoluble in organic liquid and water, group aggregates of diamonds and luminophore grains are formed, interconnected by drops of organic liquid (Fig. 3a). Such an aggregation in water medium is the result of interactions between hydrophobic components of the system. Its stability is ensured by the effective adhesive interaction of the organic liquid with diamonds and luminophores. After removal of excessive organic liquid and drying, stable attachment of inorganic luminophore grains at the diamond surface is achieved through a film of unevaporated high molecular fraction (Fig. 3b).

4. STUDY OF ENERGY PARAMETERS OF DIAMOND AND LUMINOPHORE SURFACE

Interfacial interaction energy is determined by the surface tension forces of water and organic liquids and the wetting of the surface of minerals or inorganic luminophore by liquids. The equilibrium state of a heterophase system is described by the Gibbs equations, which assume that the total surface energy tends to a minimum [18]. The generally accepted criterion for the equilibrium energy state of a heterophase system, including the solid-liquid-liquid system, is the three-phase wetting angle defined as the angle between the surface of the solid phase and the linear part of the liquid-gas interface adjacent to the three-phase contact line [15, 18] or for the system under consideration—the linear part of the aqueous phase-organic liquid interface.

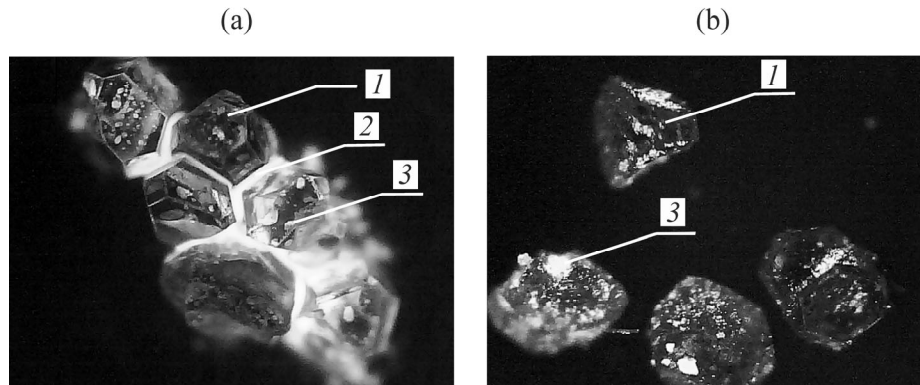


Fig. 2. (a) Group aggregate of diamonds in an organic liquid containing soluble luminophores and (b) individual diamond crystals after removal of excessive luminophore-bearing liquid and drying: 1—diamond; 2—luminophore-bearing organic liquid; 3—organic luminophore coatings on the diamond surface.

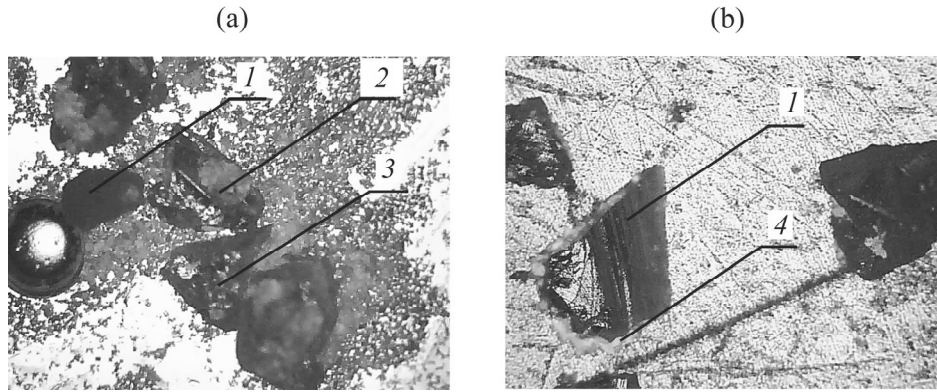


Fig. 3. (a) Group aggregate of diamonds and inorganic luminophores in an organic liquid and (b) individual diamond crystals after removal of excessive organic liquid: 1—diamond; 2—grains of inorganic luminophore; 3—organic liquid (collector); 4—particles of inorganic luminophore on the diamond surface.

The surface energy and, accordingly, hydrophobic behavior and oil receptivity of diamonds is a variable dependent on the surface condition. Based on [19–21], it is possible to estimate the heat of diamonds wetting with water and organic liquids at 30–60 mJ/m². Diamond surface energy increases with oxidation and decreases with hydrogenation [22]. A decrease in the surface tension force at the diamond-water interface is also observed when surfactants are added. According to the reference data, the heat of wetting of transition metal sulfides and aluminosilicates of insular and layered structure is estimated at 35–50 mJ/m² [23, 24]. The action of surfactants can increase and decrease the hydrophobicity of minerals [25]. Hydrophobization of zinc minerals with xanthates, aeroflots and fatty acids leads to an increase in the wetting angle and lyophilicity of the mineral surface [26]. Along with ionic collectors, various types of apolar and weakly polar reagents are used as oil wetting agents on the surface of minerals during flotation [27].

In complex systems with a variable composition of the aqueous phase, the calculation of the surface energy of solids is complicated, and the required value can be determined experimentally, based on the results of measurements of surface angle wetting with various liquids. The wetting angle is found by the ratio of surface tension forces at the interfaces of three main phases, in our case, diamond or luminophore (solid phase), aqueous phase and organic liquid (Fig. 4a).

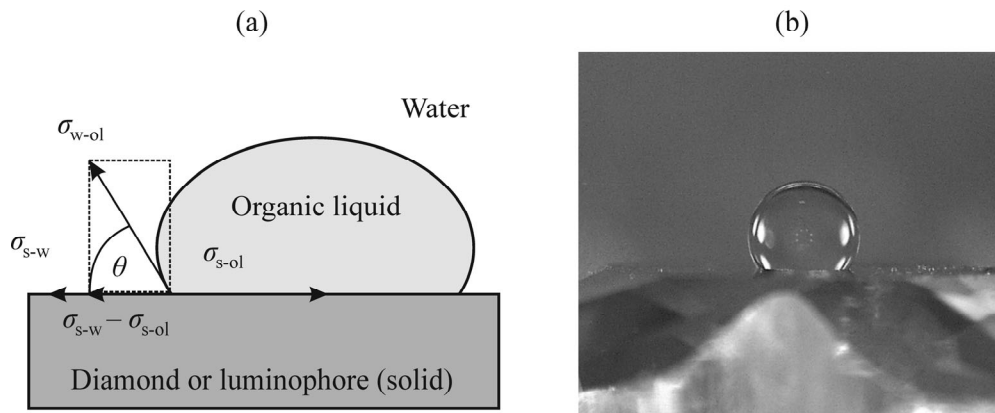


Fig. 4. (a) Schematic diagram of wetting angle θ formation as the resultant of surface tension forces and (b) image of a drop of organic matter on the diamond surface in water medium: σ_{s-w} —surface tension at the solid-water interface; σ_{s-ol} —surface tension at the solid-organic liquid interface; σ_{w-ol} —surface tension at the water–organic liquid interface.

The procedure used for measuring the wetting angles differed somewhat from the classical one [15] and included successive operations of wetting the mineral section in water medium with the formation of a thin boundary layer, applying a drop of organic liquid to the wetted section, raising the liquid level in the cuvette, photographing the sample. The adopted technique uses the dynamic mode of wetting angle formation, which is provided by changing the drop shape in water medium and approaching it to a spherical shape when the cuvette is filled with water (Fig. 4b).

The wetting angle is related to the energy parameters of surfaces according to the equation of Young's law [18]: $\theta = \arccos[(\sigma_{s-w} - \sigma_{s-ol}) / \sigma_{w-ol}]$. The values of surface tension for the liquid interfaces were calculated as the difference between surface tensions of the substance–vapor interfaces (Antonov's rule) [7]: $\sigma_{w-ol} = \sigma_w - \sigma_{ol}$. This rule is inapplicable for solid-liquid interfaces, since it is impossible to measure the surface tension values for solids at ordinary temperatures (0–25°C) [18]. Based on the analysis of known procedures, it is proposed to estimate the surface wetting energy by the Zisman method, according to which the maximum value of the wetting angle is achieved when surface energies of the solid phase and organic liquid are formally equal [28].

The formal equality of the surface tension of organic liquid and the surface energy of a solid does not mean that the surface tension is equal to zero. During adsorption or adhesion, only a part of the surface energy of both phases is compensated and interfacial surface tension reaches not a zero value, but a minimum. The dependence of surface tension at the solid-liquid interface will be minimal in the region where surface energies of the phases are formally equal, and three-phase wetting angle in the mineral-organic liquid-aqueous phase system will reach a maximum value, which is called critical surface tension [28].

The relationship between the wetting intensity and surface energy of heterogeneous components has been verified by experimental studies—measurements of wetting angles of diamond and zinc sulfide, which is the basis of luminophore E-515-115, generated by the organic liquid drops in the water medium. The extreme nature of the dependence of wetting angle on surface tension of an organic liquid was confirmed experimentally. For diamond, the wetting angle reaches its maximum value in the range of surface energies of organic liquid of 28–47 mJ/m². The largest wetting angle of zinc sulfide is observed when using organic liquids with a surface energy of 31–52 mJ/m² (Fig. 5). Approximation equations for the coordinate of maximum dependence of the wetting angle allow estimating the diamond's surface energy as 34 mJ/m², and surface energy of zinc sulfide—as 43 mJ/m².

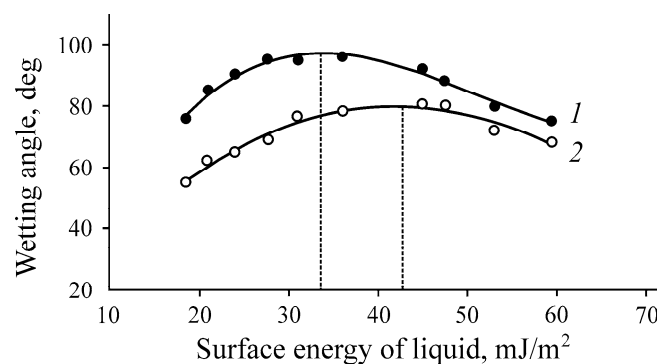


Fig. 5. Dependence of the wetting angle of diamond (1) and zinc sulfide—the basis of luminophore E-515-115 (2) on the surface energy of an organic liquid (hexane, gasoline, kerosene, hexadecane, diesel fuel, pyridine, motor oil, fuel oil, formamide, glycerine)

5. SELECTION AND STUDY OF ORGANIC LIQUIDS AS THE BASIS OF LUMINOPHORE-BEARING COMPOSITION

When implementing the technology of modifying diamond spectral characteristics with luminophore-bearing compositions, an important condition for the efficiency is correct selection of the organic liquid used as luminophores collector. The selection of an organic liquid as the basis of luminophore-bearing composition suggests a significant difference between its surface energy and water energy. At the same time, it is necessary to observe the principle of polarity equalization at the interface (the Reh binder principle), when a substance with an intermediate polarity relative to the adsorbent and solution is characterized by the highest adsorbability or adhesability. If hydrophobicity of diamond, luminophores and collector is high, the diamond-luminophore-organic collector system will be very stable. The required result can be obtained by physical and chemical modification of mineral properties and the use of additives that activate the adhesion of the organic collector on luminophores and diamonds. For the best adhesive interaction of an organic liquid with diamonds and luminophores, it is advisable to use a substance with a surface energy that is closest to the surface energy of minerals. This ensures the minimum surface tension of the interface and the maximum value of three-phase wetting angle, which is identified with the maximum lyophilicity of diamonds and inorganic luminophores.

Analytical results of the dependence of wetting angle of diamond and zinc sulfide on surface energy of the organic liquid allowed recommending organic substances and oil products with surface energies of 25–52 mJ/m² (hexadecane, diesel fuel, motor oil, fuel oil) as a collector of luminophore-bearing composition. When these substances interact with the surface of diamond and luminophore, the maximum increase in the strength of hydrophobic interactions identified with maximum values of the wetting angle is possible. The validity of conclusions is confirmed by the results of experiments on studying the interaction of drops of luminophore-bearing composition, consisting of organic liquid and luminophore extracted in it, with diamond crystals and kimberlite grains. If hexadecane is used as a collector, then luminophore is retained and diamond crystals are extracted inside the composition drops (Fig. 6a). At the same time, the required selectivity of the process is preserved, which consists in the absence of hydrophobic interaction, adhesion, and volumetric extraction of kimberlite grains onto the surface or inside the drops of luminophore-bearing composition (Fig. 6b). Aggregates of diamonds and luminophore-bearing composition remain stable after drying and are separated into individual crystals only under mechanical action (Fig. 6c).

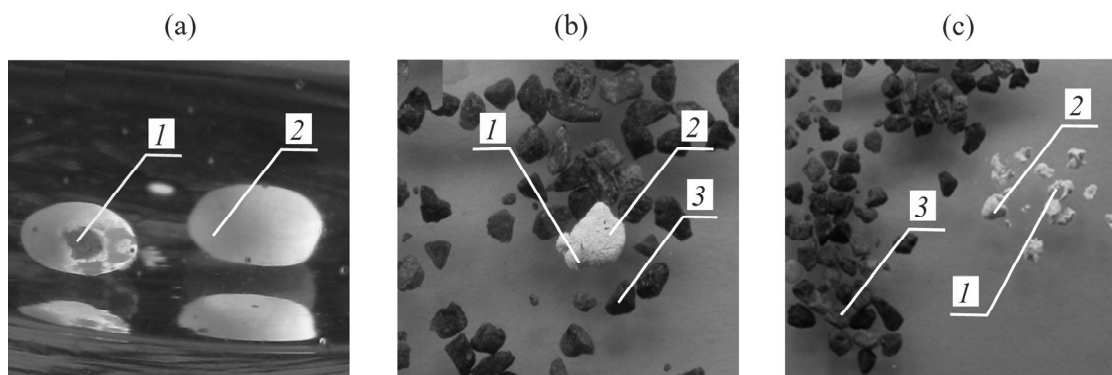


Fig. 6. Diamond aggregate with drops of luminophore-bearing composition (a) in the water phase, (b) dried and (c) after mechanical failure: 1—diamond crystals; 2—luminophore-bearing composition; 3—kimberlite grains.

Table 2. Spectral and kinetic characteristics of luminophore-bearing compositions from FL-530 and anthracene in diesel fuel and fuel oil F-5 and diagnostics results of processed diamonds on Polyus-M separator

| Composition of organic collector | Convolut ion | Ratio of component amplitudes | Decay time constant, ms | Component amplitude, mV | | Result of identification | |
|----------------------------------|--------------|-------------------------------|-------------------------|-------------------------|------|--------------------------|----------------|
| | | | | slow | fast | Collective mode | Selective mode |
| Luminophore-bearing composition | | | | | | | |
| DF | 0.64 | 1.9 | 3.6 | 7333 | 6937 | — | — |
| Fuel oil F-5 | 0.44 | 2.0 | 3.0 | 1230 | 2251 | — | — |
| Mixture of F-5 and DF (1:4) | 0.60 | 1.0 | 3.8 | 6360 | 5022 | — | — |
| Processed diamonds | | | | | | | |
| DF | 0.58 | 1.2 | 2.8 | 233 | 331 | Found | Found |
| Fuel oil F-5 | 0.56 | 1.1 | 3.2 | 43 | 125 | Not found | Not found |
| Mixture of F-5 and DF (1:4) | 0.57 | 1.0 | 3.8 | 183 | 330 | Found | Found |

A similar result was obtained by processing a mixture of diamonds and kimberlite minerals with a composition of inorganic luminophores FL-530 and E-515-115 with anthracene in diesel fuel (DF). During the experiment, a high stability of the luminophore-bearing composition and good adhesivity of the organic liquid toward diamond were also achieved. As in the case of using hexadecane, the volumetric extraction of diamonds with drops of an organic collector was not accompanied by the capture of kimberlite minerals. When using motor oil and fuel oil as a collector, both extraction into the volume of organic collector drop and retention of luminophore in it, as well as subsequent attachment of the composition at diamonds were observed.

The selected composition of the organic liquid should not have pronounced effects of X-ray and light radiation absorption, as well as the effect of X-ray luminescence quenching. Such requirements complicate the use of dark oil products (for example, fuel oils). Compositions of luminophores FL-530 and anthracene with fuel oil are characterized by a lower luminescence intensity (lower amplitudes of the fast and slow components) relative to the compositions based on diesel fuel with the same amount of luminophores in the composition (Table 2). However, high-molecular fractions of oil have high adhesive properties with respect to diamonds, which makes it advisable to add them to organic collectors. A mixture of diesel fuel and fuel oil in a ratio of 4:1 provides the desired efficiency for modifying the spectral and kinetic characteristics of nonluminescent diamonds (Table 2). The prospects of using compound collectors consisting of a mixture of organic liquids that meet the requirements for extraction-adhesion properties and do not degrade the spectral and kinetic characteristics of luminophores are confirmed.

The obtained results determine the possibility and expediency of using optically transparent low-polar organic liquids with a surface energy of 28–45 mJ/m², such as diesel fuel, mineral hydraulic oils, hexadecane, as well as their mixtures, as the basis for a luminophore-bearing composition.

CONCLUSIONS

A mechanism for attaching a luminophore-bearing composition (a mixture of luminophores and organic liquid) at the diamond crystal surface is determined, which consists in the formation of stable diamond aggregates, organic liquid, and inorganic luminophore during hydrophobic interaction in a polar medium. The stability of such aggregates is ensured by the intense adhesive interaction of diamonds, organic liquid, and luminophores and is achieved at maximum values of three-phase wetting angle.

It is experimentally determined that high efficiency of luminophore extraction with organic liquid and adhesion of the luminophore-bearing composition on diamonds is achieved at a significant difference in the energies of diamond and luminophore wetting with water and organic phases at similar surface energy values of luminophores and organic liquid.

It is theoretically and experimentally confirmed that the most effective basis for a luminophore-bearing composition is optically transparent low-polarity organic liquids with a surface energy of 28–45 mJ/m².

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