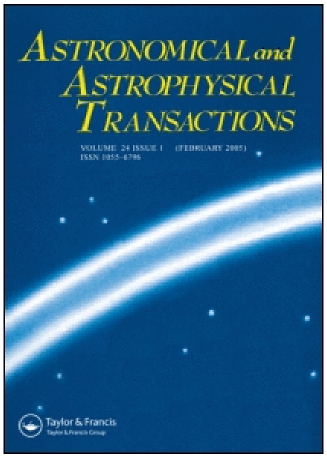

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THE LUMINESCENCE FLAW DETECTION AS A METHOD FOR REVEALING IRON METEORITES MICROSTRUCTURE FEATURES

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The method of luminescence flaw detection has been applied to the detection of microstructure features of meteorites, minerals and samples of cosmic matter. The present paper considers the basic procedures of the method. We discuss main experimental results of the application of the method to meteorites from the Sikhote Alin meteorite shower, alongside with practical recommendations for its implementation. Besides, some other related questions are discussed.

INTRODUCTION

Microstructure features of meteorites and samples of cosmic matter are studied using a great variety of conventional methods of detection differing in the degree of their complexity, resulting efficiency and other characteristics. Extensive application of the common methods, however, should not stand in the way of attempts to search for and use new ones.

The problem stated in the present work consisted in developing and further using a new method of detecting microstructure features in meteorites and cosmic matter samples, that would provide such properties as good efficiency, simplicity and cost-efficiency of the procedures.

Speaking of those properties we imply that an efficient method should ensure detection and viewing of a 1 μm microstructural element; its simplicity has to ensure as few operations or procedures as possible, and as low a demand for designing and producing special facilities and devices, including electronic optical systems; and cost-efficiency of the procedures means low expenditures and minimum consumption of materials. After a considerable search, we chose the method of luminescence flaw detection which seemed to meet all the above requirements. In some earlier papers [1, 2, 3] we gave theoretical reasoning concerning application of the luminescence flaw detection method to meteorites, cosmic matter samples (including lunar soil samples) and terrestrial and cosmic minerals.

Proceeding from theoretical reasoning to practice, we applied the method of luminescence flaw detection to two meteorites from an iron meteorite shower of the Sikhote Alin. The method was improved and refined in the course of the experiments. The luminescence flaw detection method facilitated viewing of considerably fine microstructural features ranging from 0.5 to 325 μm, both in the melting crust and cut sections. The detected features included caverns, hollows, microfissures, etc. The method proved to be quite good, simple and cost-efficient, therefore appropriate to examine the microstructure of iron meteorites.

As the experiments were conducted using conventional equipment, there was hardly any need in a specially designed laboratory. Luminescence flaw detection of meteorites can be performed in physical, geological, cosmic or geochemical laboratories of universities and research centers.

The present paper sums up the experimental results obtained for meteorites of the Sikhote Alin and is mainly concerned with the development of a method enabling specialists of different fields of science to carry out similar experiments and further improve the method itself.

ON THE BASIC PROCEDURES OF THE LUMINESCENCE FLAW DETECTION METHOD

The luminescence flaw detection is a method comprising a certain sequence of operations. It ensures viewing various microstructure features in the melting crust and cut sections of iron meteorites. Luminescence flaw detection makes visible many of the voids in their surfaces.

The experiments involving the application of luminescence flaw detection to iron meteorites from the Sikhote Alin demonstrated a certain difference between theory and practice, which call for quite significant corrections to be made in the experiments; namely, the introduction of new steps, alterations in the conditions of other procedures, etc. The final list of steps of the experiments (i.e., the structure of the method) has been consequently drawn up:

1. selection and preparation of the necessary materials and devices;
2. preparations of the meteorite surfaces for luminescence viewing;
3. checking the meteorite surfaces through a luminescence microscope;
4. treating the meteorite surfaces with a luminophor;
5. removing the luminophor off the meteorite surface;
6. drying the meteorite surface;
7. examination of the meteorite surface through a luminescence microscope; photomicrography of the surface.

It should be noted that it is not only an accurate performance at each step what is important, but also the sequence of the procedures in the above list. Neither the sequence, nor the number of the operations can be changed, as this will deteriorate the efficiency of the method. However, although the sequence of the operations has to be strictly followed, the conditions of their performance can be varied (i.e., the luminophor temperature, grinding time, duration of exposition, etc.). If any other experiments were supposed to be similar to ours, then the conditions have to be similar to those in our experiments. However, if the method has to be updated and improved, the conditions can be varied within present ranges. Let us consider each procedure.

Selection and Preparation of the Necessary Materials and Devices

As the title shows, the procedures to be carried out at the first step are basically of preparatory and organizational nature.

It is important to choose the most suitable meteorites; namely, they have to be only iron meteorites, small enough to fit into special bowls, microscopes, etc.

Another important point is to choose the most suitable luminophor. The luminophor used in our experiments was:

a) liquid,
b) effective, i.e., penetrating into the voids,
c) of bright and steady luminescence, and
d) neutral.

After some search, we choose an organic luminophor "Noriol-A" synthesized from petroleum at the Research Institute of Chemistry, Academy of Sciences of the Republic of Georgia. "Noriol-A" met all the above requirements. It is a slightly oily dark-brown liquid capable of penetrating to a 15 μm depth and thus revealing a microstructural feature of up to 0.22 μm size. The luminescence of the luminophor was excited by UV radiation in the wavelength range 340–380 μm. "Noriol-A" provided bright and steady luminescence in the yellow-green visible range, with the best performance achieved at 550 nm wavelength. The luminophor afterglow did not exceed 0.01 s at +10 to +20°. Therefore, throughout the experiments the luminophor was constantly irradiated by UV emission, which was ensured by a luminescence microscope. The afterglow of "Noriol-A" grows longer with the temperature decreasing below 0°C, reaching some seconds at −40°C. At this point the luminophor changes from liquid into solid. As temperature goes up to +45°C and higher, the luminophor gradually loses its luminescent properties. The luminescent properties also appear to decline after "Norial-A" has been exposed to fresh air or daylight for over 30 hours running. Therefore, the luminophor treatment and further examination of the treated surface through a luminescence microscopy ought to be performed on the same day. Both the luminophor and the treated samples ought to be stored in air- and light-proof containers. As our experience has shown, properly stored treated samples can be used more than once to examine their microstructure through a luminescence microscope. Being a neutral substance, "Noriol-A" does not affect the physical and chemical properties of a treated surface. It is not harmful, either. The luminophor can be transported by any means of transportation, with special care to ensure tight packing required only for air transportation.

The trade name "Noriol" is derived from a place-name "Norio", an oil-field not far from the city of Tbilici (Republic of Georgia), since it was the oil of the field that luminophor "Noriol" was synthesized from. "Noriol-A" is one of the latest modifications of the luminophor.

Still another important point in the work is to keep up proper working state of all the laboratory instruments, tools and supplementary materials, particularly, the luminescence microscope. Make sure that all the working surfaces of the tools and instruments are clean since any foreign matter occurring on them may turn out to be luminescent.

It has been confirmed in our experiments that the surfaces of the iron meteorites to be examined should be specially prepared for viewing. This special pre-treatment must involve both the melting crust and the cut sections.

The reliability of the experimental results depends on the quality of that pretreatment.

The melting crust is to be carefully cleaned in order to remove all the foreign matter, including soil particles. Special care should be taken while removing the identification numbers or indices usually stuck on the surfaces of the specimens. As a rule, such numbers or indices are written on pieces of adhesive tape which should be cautiously peeled off the melting crust and the surfaces exposed underneath should be thoroughly cleaned using soft and blunt tools to remove glue particles avoiding any scratching or other mechanical damage of the surface. It is actually impossible to clean completely such spots on the melting crust bearing remains of the glue; besides quite a few sticking or gluing substances manifest luminescence properties under UV radiation, thus making it difficult to examine the microstructure of such surfaces using the luminescence effect. Such a spot should be clearly marked and outlined, and the luminescence detection should be performed over a larger and clean melting crust surface of a meteorite, free of any foreign matter.

Let us now consider pre-treatment of cut sections of iron meteorites. It is a common knowledge that cutting tools frequently damage the cut surface leaving scratches, hollows, etc. on it. A cut surface in such a condition is certainly unfit for luminescence microscopy, which can be carried out only after the cut has been roughly ground and polished. Grinding and polishing are supposed to remove all the major mechanically caused damages from the cut surface. A mirror-like smooth finish obtained after grinding and polishing must only preserve the natural microstructural features of the meteorite substance.

In our experiments, grinding was performed by different methods, one of which consisted of the following stages: first, the cut surface was ground with a coarse abrasive grit, then a less coarser one; finally it was finished with finest sand. The abrasives were also of different kinds, namely, corundum and siliconcarbide (Carborundum). Among other abrasives, sands of 80 μm mesh were used. The total grinding time was up to 1.5 h.

Some problems arose in the process of polishing. It turned out that some of the applied polishing stuffs (including pastes) produced bright luminescence when exposed to UV radiation. Therefore, those substances were rejected as unfit for polishing. We used a similar common method of polishing out sections of iron meteorites: a cut surface was polished by means of a disk covered with soft cloth and rotating it at a high speed. The total polishing time was up to 20 min. We also tried hand polishing with small pieces of soft cloth. The manual polishing took about 1.5 h. The cloth used for polishing can be of any kind of soft fabric, like soft flannel, provided it does not produce luminescence.

The expedience of such a simple procedure might be guestionable, but our experiments have confirmed the validity of such a treatment since it ensures a mirror-like finish on the cut section.

Checking the Meteorite Surfaces Through a Luminescence Microscope

Following the above pretreatment of the meteorites, their surfaces are to be checked by luminescence microscopy.

As mentioned above, the method of luminescence flaw detection can be applicable only to clean surfaces on the melting crust, and any site that may contain foreign matter should be accurately measured and marked. If the foreign matter in those sites happens to be luminescent, they can be measured and marked during luminescence microscopy checking.

Foreign particles can be left on the cut surfaces after grinding and polishing. If those microparticles turn out to be luminescence, both their exact location and luminescent colour must be estimated, and again, it can be done in the course of luminescence microscopy checking.

Cut surfaces may contain patches of various minerals which, if luminescent, can be accurately located and outlined through a luminescence microscope.

Foreign matter, microparticles and minerals which are not luminescent can be ignored in the experiment since we are concerned only with all kinds of sources of luminescence on meteorite surfaces. A meteorite to be checked is placed under a luminescence microscope and viewed at different powers of magnification: low, medium and high. Should any luminescent foreign matter, particle or mineral occur on the examined surface, it must be accurately located, measured, marked and its luminescence colour must be identified. These data will help to distinguish luminescence of any foreign matter or body from that of the luminophor filling the microstructural voids.

Treating the Meteorite Surface with a Luminophor

After all the above procedures have been over, the meteorite surface is to be treated with luminophor. The luminophor is to be poured out of its container into a vessel of a definite capacity. With all the laboratory tools (glass spoons, bowls, etc.) being ready at hand, the meteorite is placed on a laboratory table with the surface to be treated facing upwards. The said surface is carefully covered with an even layer of the luminophor. As soon as this operation is completed, start up a clock and let the specimen stand for 10 minutes. Otherwise, the meteorite can be dipped into a bowl filled with luminophor and left there for a 10-minutes period.

The luminophor takes 10 minutes to penetrate deep into the microstructure filling the major voids, i.e. caverns, hollows,microfissures, etc. During this step of the experiment, care must be taken to avoid direct sunrays or electric light upon the meteorite.

Removing the Luminophor off the Meteorite Surface

After the above 10 minutes have elapsed, the meteorite is rinsed in cold running water for 6 minutes, turning it smoothly and carefully in the current. Be careful to hold it so as not to touch the areas to be examined. The luminophor will thus be washed off the whole surface. After the 6-minutes rinsing, shake the biggest water drops off the meteorite. In this way the meteorite surface will be clear of the luminophor, however, part of it remains in the voids. Take special care not to touch the treated surface of the meteorite with your fingers or any objects or tools during the rinsing operation and later on till the end of the experiment.

Drying the Meteorite Surface

The water-rinsed meteorite is mounted on a laboratory table with the treated surface turned upwards and left there for 15–20 minutes during which that surface gets dry. The drying process should not to be enhanced or induced in any way, such as exposure to the sunlight or bright electric light, or drying with a piece of cloth or hot air jet. Such induced drying can only adversely affect the results.

Luminescence Microscopy. Photography

This step comprises the last and principal operations. The meteorite is mounted in a liminesence microscope which ensures different magnification powers during viewing. Each microstructural feature is examined at low, medium and high magnification, with all the necessary measurements, record, drawings, etc. being done during the examination.

A camera is mounted behind the microscope eye-piece. Make sure to get good focusing before taking a photograph. A high-contrast film of the "Micrat-200" type should be used for the photography. In our experiments the exposition lasted from 1 to 1.5 minutes. The film was developed in a high-contrast developer for 7 minutes, at a temperature +22°C. The photographs were made using high-contrast chemicals. Series of 20–25 frames of each viewed surface were made with the subsequent development of the film and printing of the photographs.

A luminescence microscope of the "Luman R5" model was quite successfully used in our experiments.

Another instrument used as a supplementary tool in our experiments was a so-called luminoscope (presumably, known under some other name elsewhere). This device is of a rather simple design, comprising a small box with a standard source of UV emission inside it. One of the sides of the box is provided with a sliding glass window. The meteorite is placed inside the box, the window is shut, and with the UV emitter on, the surface can be quickly examined, revealing major microstructural features.

This actually completes the whole sequence of the procedures commonly used in luminescence flaw detection applied to iron meteorites.

On the Ways of Removing Luminophor from the Microstructure

After the experiments are over, the luminophor can be removed from the voids in the microstructure, if required, or its luminescence can be quenched. It is, however, rather difficult to deal with either of those procedures. There are, certainly, chemicals capable of decomposing, disintegrating a luminophor. For instance, "Noriol-A" is readily disintegrated with pure petrol or any other related substance. Still, some of the voids may remain filled with luminophor after petrol treatment.

Luminescence of a luminophor can be quenched by its prolonged exposure to fresh air and bright light or heating up to +50°C.

Some Investigation Results for the Sikhote Alin Meteorites

The method of luminescence flaw detection was used to study two meteorites from an iron meteorite shower of the Sikhote Alin. The melting crust was studied in one of them, and four cut sections in the other. The latter specimen designed for the cut section examination, was given the shape of a rectangular parallelepiped. Varied microstructures were observed both in the melting crust and the cut sections. The microstructure in the melting crust was quite intricate. The caverns, hollows, channels discovered and their systems were frequently connected with one another. The microstructure observed in the melting crust appeared to be mainly a result of processes occurring in the meteorite surface during its fall through the Earth's atmosphere (melting, evaporation, etc.). The microstructure of the cut section was dense and discrete. Its elements stood apart from each other. Besides caverns, there was small, irregular fissures running in different directions. The microstructure of the cut sections indicated to the origin and evolution of the meteorite matter. The melting crust appeared to contain 0.5 to 325 μm microstructural features, those under 0.5 μm occurring but rarely. In the course of the above studies, quite a big series of photographs was made, some of which are shown in Appendices A and B.

Some Hints on the Application of the Method

The experimental results related to the examination of the microstructure of the Sikhote Alin iron meteorites using the luminescence flaw detection method confirm that the above method can be successfully used in solving various problems.

The implementation of the method in various laboratories can hardly call for any significant financial expenditures since the method is a simple one.

Luminescence flaw detection can be employed to observe caverns, channels, microfissures and other voids of various forms and shapes. It can ensure identification of different microstructural elements, and the problems to be solved can be of most different kinds. For instance, some investigation procedures called for detection of multidirectional microfissures in a certain area of the meteorite surface – an issue that can be dealt with only by means of luminescent flaw detection. Both visual examination and photographs readily reveal the fact of the presence or absence of such microfissures, their prevalent direction, etc. The luminescent flaw detection method can be used for the measurements of such dimensions as diameters, length, etc. In this case quite a number of various problems can be solved, e.g., if it is necessary to find the number of caverns ranging from 1 to 10 μm in a particular area of the meteorite surface.

The total number of voids of the given size can be easily estimated during viewing or in the photographs. The method may be helpful in tracing the regularities of the distribution of various microstructural elements over the meteorite surface under examination, i.e., in revealing areas where certain elements – caverns, microfissures and others – are prevalent. It is a common knowledge that caverns are often left by minerals embedded in the meteorites, the forms and shapes of the caverns depending on the kinds of the minerals. Some of the minerals leave regular spherical caverns, others leave irregular, aspheric ones. Detection of the caverns using the luminescence effect provides a sufficiently good sharpness and contrast of their viewing. Consequently, the method in question ensures a simple analysis, counting of the total number of differently shaped caverns in the meteorite surface under study. Having counted up the caverns over all the surface, one can make conclusions concerning the prevalence or absence of certain kinds of minerals in the given meteorite.

The luminescence flaw detection method can be used to solve other problems as well. The range of its applicability can be widened after testing the method on other types of meteorites, including comet meteorites.

CONCLUSION

We have discussed the basic steps and procedures of the method in question and investigation results for the Sikhote Alin meteorites. It would be now worth dwelling on the questions of possible further development and improvement of the method. Quite a few possibilities can be envisaged concerning the study of iron meteorites. More efficient luminophors can be used, as well as other methods of grinding and polishing. It would be desirable to employ newer models of luminescence microscopes and, finally, to use higher-contrast materials for photography. Any of these improvements and changes would considerably increase the efficiency of the method, and, naturally, if all of these improvements could be made, the resulting efficiency of the method would grow many times. In the course of the development of the method it has to be tested on stone meteorites and various minerals. We have recently started a series of experiments on the application of the method to stone meteorites, and their results will be published in due course.

Finally, let us consider the problems of terminology. There is an opinion that the term "luminescence flaw detection" is not quite suitable for meteorite studies. In dealing with this problem two points should be kept in mind: one is that the term "luminescence flaw detection" is quite a common one used in articles, reference books, etc.; however, the other point is that the words "flaw detection" (or "defectoscopy" as it appears in Russian) hardly reflects the essence of the method. It is not really "flaws" or "defects" that we are trying to detect in the meteorite surfaces, but natural microstructural features occurring there. It seems reasonable to change the name of the method, and one of the tentative versions might be the "luminescence structure microscopy of meteorites" when it involves visual observation, and the "luminescence structure micrography of meteorites" when photography is performed. But we think it better to suggest these terms for further consideration rather than use them as such.

The author expresses his gratitude to Dr. L. Melikadze and Dr. E. Pirrus for their valuable remarks made in the course of the experiments.

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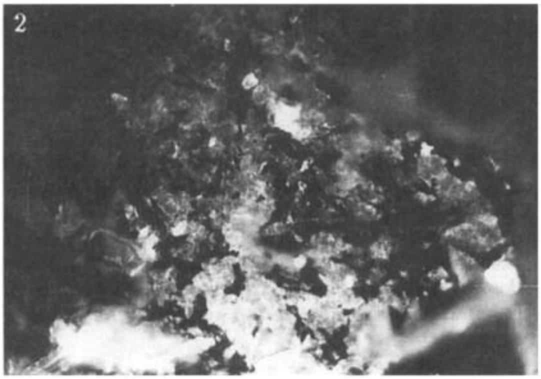
Appendix A Photographs 1, 2, 3. The microstructure of the melting crust. Overall magnification 400 X.



Appendix B Photographs 4, 5, 6. The microstructures of the cut sections. Overall magnification 400 X.