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Measurement of Speed of Sound in Shungites by Laser-induced Ultrasound

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Abstract
Shungite rocks are unique composites with interpenetrating carbon and mineral matrices. Shungite rocks can be used as fillers and modifiers of various composite materials. Nanostructured shungite materials are effective fillers for composites, they can impart conductivity and be used for electromagnetic shielding, additionally shungite fillers are able to improve strength and antifriction properties. The speed of sound in small shungite samples was measured by laser optoacoustic technique. This method can be used for evaluation of carbon content in natural composites.

Keywords: shungite, materials properties, laser-induced ultrasound, optoacoustic conversion

1. Introduction

Industrial synthesis of carbon-based nanoparticles is expensive. In this regard, the interests of researchers in the field of nanotechnology are turned to natural sources of carbon nanoparticles, such as Karelian shungite [1,2]. The promising application of shungite is associated with development of batteries [3]. One of the effective methods for studying the physical parameters and internal structure of mining materials is the method of laser ultrasonic spectroscopy [4, 5], with the help of which the sound velocity was measured in this article. The estimation of sound velocity can reveal morphology of the specimens, content of carbon and their microstructure.

2. Samples and methods

The samples of Karelian shungite with different carbon contents in the form of square thin plates (Fig. 1), the area of which did not exceed 36 mm^2, were studied. The thickness of sample I and II are 1.5 mm and 1.05 mm, respectively. The sample I is shungite carbon (98% C), and sample II is massive schungite rock (32% C), that was confirmed by the results of thermal analysis and Raman spectroscopy.

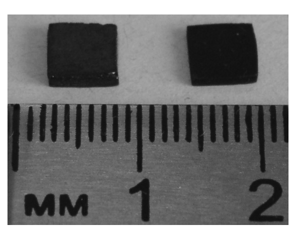

Fig.1. Samples of shungite. The thickness of the left specimen is 1.05 mm, right specimen thickness is 1.5 mm

The scheme of the experiment is described below and presented in Fig. 2. The surface of schungite samples was irradiated by Nd:YAG laser pulses at a wavelength of 532 nm with a duration of approximately 10 ns, and an energy of less 5 mJ, as shown in Fig. 2. The diameter of the laser spot on the surface of the sample is approximately 3 mm. The absorption of optical radiation is significant (around 1000 cm^{-1}) in shungite. The heating of the specimen leads to the optoacoustic conversion and as a result mainly longitudinal ultrasonic pulses with a pronounced compression phase and an upper frequency in the spectrum reaching 50 MHz are excited in the sample. In order to measure the velocity of longitudinal waves, the water layer of about 1mm between schungite samples and transducer was used to provided the acoustical contact that relates to the immersion technique. Transducer based on a polarized polyvinylidene fluoride (PVDF) film with a thickness of 25 μm and a sensor diameter of 2 mm was on the opposite side of specimen that allowed broadband registration of separate pulses.

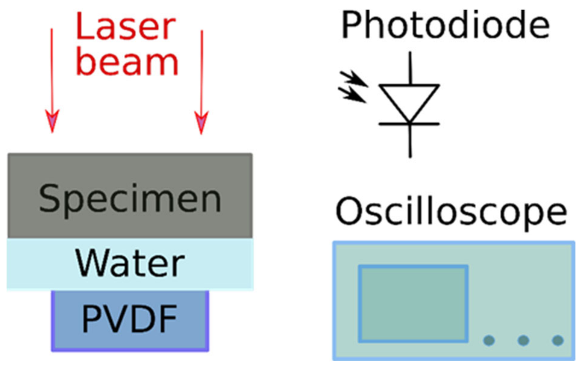

Fig.2. Sketch of experimental setup

A preamplifier made on one operational amplifier and operating in a “short-circuit” mode was placed in the aluminum case of the transducer, which allowed recording ultrasonic pulses in the frequency band from 0.1 to 90 MHz [3]. The signal from the preamplifier was applied to the input of an oscilloscope with an analog band of 200 MHz and a sampling frequency of 1 GHz. The photodiode was used to trigger the oscilloscope in the time moment of sample surface illumination that was necessary for accurate measuring of the velocity.

3. Measurement results

As can be seen from Figs. 3 and 4, a pulse excited at the upper water-sample boundary runs through the sample and the second water layer, the thickness of which from the estimates of the delay time on the measured signals is approximately 1 mm, after which it reached the transducer.

The calculation of the velocity of longitudinal ultrasonic pulses is performed by the formula:

$$c_l = 2\,{}^{h}/_{dt} \qquad (1)$$

where h is the thickness of the sample; dt is the travel time between the primary ultrasonic pulse and the first ultrasonic echo which passed through the sample (Figs. 3 and 4).

Fig. 3 and fig. 4 show the optical-acoustic signals obtained in the samples of schungite I and II. A section of track located between the straight a probe bipolar pulse and a second bipolar pulse (after double reflection from the rear and front surfaces of the sample), carries information about the propagation velocity of ultrasonic pulses and the internal structure of the sample. According to the measurement results, the longitudinal pulse velocity was: 3.83 ± 0.03 km/s in sample I and 4.66 ± 0.04 km/s in sample II.

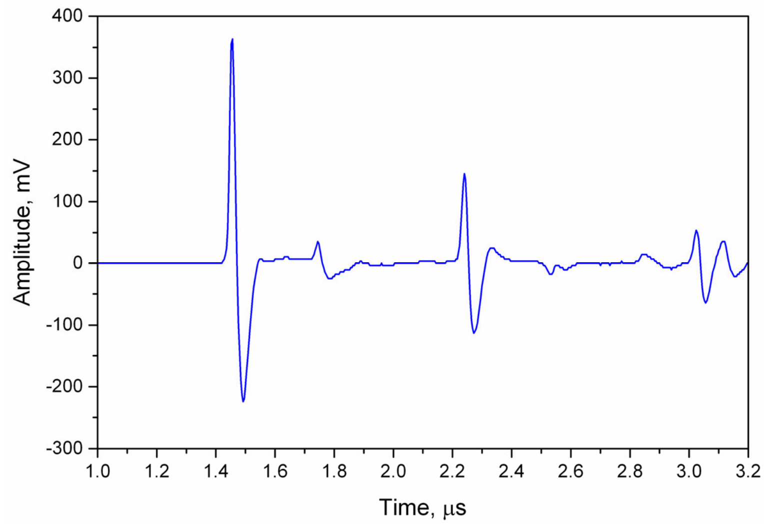

Fig. 3. Signal measured in 1.5 mm thick shungite I

The estimations made by the direct Fourier transform demonstrated in the spectrum the band of the direct probe pulse from 5 to 20 MHz at the 1/2 level. The measured velocity in the vein quartz present in both samples was ≈ 6.3 km/s. The difference between the longitudinal wave velocities in the samples is determined by the internal structure and composition of the samples, since an increase in the quartz content increases the longitudinal wave velocity.

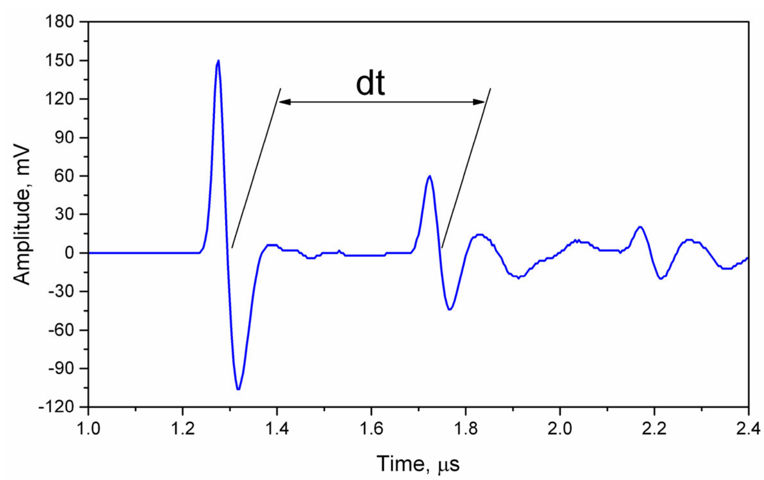

Fig. 4. Signal measured in 1.05 mm thick shungite II

4. Conclusions

The method of laser ultrasonic spectroscopy has proven itself in measuring the velocity of longitudinal waves on small shungite samples. Further development of the method will make it possible to measure the velocity of shear pulses and evaluate the mechanical properties of statistically isotropic materials.

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