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# Laser study of phase changes in the surface layer of porous materials

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*Abstract.* The paper presents some aspects of the use of interference patterns observed upon reflection of laser radiation from the surface of a porous solid (laser speckles) for the study of moisture condensation in the near-surface layer.

Keywords: laser speckles, porous solids, moisture.

# 1. Introduction

The nondestructive studies of various properties of materials are very important application of lasers. The laser radiation in these studies fulfils the role of the precise sensor, which does not affect the state of a tested material. At present, the interaction of laser radiation with porous solids, in particular, dielectrics is poorly investigated. The nondestructive laser methods can be applied, for instance, in the material engineering and in the protection of monuments. This paper is devoted to this fields.

The surface of a porous body is very rough with respect to the laser light wavelength. When nearly monochromatic light is reflected from such a surface, the resulting optical wave at any sufficiently distant point consists of many coherent wavelets, each of them reflected from different regions of the surface. The optical paths between these wavelets may differ by several wavelengths. The waves scattered from a such rough surface have not only random phases but also random real amplitudes. The interference of the dephased but coherent, secondary spherical wavelets results in the granular intensity pattern, which is called the speckle pattern (speckles). This pattern is not directly related to the macroscopic structure of the illuminated object, but rather have disordered, chaotic character described by the probability theory and statistical methods [1]. When there are microscopic water droplets near the surface or on it, some waves reflected from these droplets acquire the additional difference of optical paths, making an additional contribution to the interference pattern.

It was shown earlier [2-4], that the parameters of the laser light reflected from the surface of a porous body

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Received 16 August 2000 *Kvantovaya Elektronika* **31** (4) 318–320 (2001) Contributed in English; edited by M N Sapozhnikov depend on the moisture content in the body. However, because of the complex microstructure of the porous body surface (which can be also investigated using lasers [5]) and indicate physical processes proceeding on it, only some effects can be studied. At present there is no a theory that would describe the relation between the parameters of speckles produced by the laser light reflected from the surface of the porous body and the surface structure and humidity.

The aim of this work is to study the influence of the moisture content in the near-surface layer of a porous body on the parameters of the laser light reflected from it, and the choice of the optimal measurement geometry and analysed parameters. Porous samples of gypsum suspension were studied, which were measured at different geometries of the experimental setup.

#### 2. Experimental

In the first studies of radiation reflected from moist porous bodies [2], which revealed the dependence of the reflected light parameters on the moisture content, the intensity of the reflected beam was measured with a photodetector within a narrow solid angle in the direction of specular reflection. In this paper, a more complicated setup was used. Laser speckles were detected using two CCD cameras (the first camera was placed in the mirror reflection direction, and the second one, perpendicular to the sample surface). Fig. 1 shows the schematic of the experimental setup. This



Figure 1. Schematic diagram of the experimental setup.

setup consist of the 17-mW cw helium-neon laser (Melles-Griot). The laser beam was incident on the sample surface at an angle of  $30-45^{\circ}$ . The sample was placed on the Peltier table which allowed to heat the sample up to  $35^{\circ}$ C and cool it down to  $-5^{\circ}$ C. The temperature was measured at four points (1-4) with thermocouples and the humidity was measured at two points (3 and 4) with polymer humidity sensors. A microcomputer was equipped with the frame grabber card and analog-to-digital converter. Between the laser and a sample as well as between the surface and cameras, lenses and apertures were placed.

Figure 2 shows the specklograms obtained for a dry gypsum sample. One can see, that the speckle size in the mirror reflection direction are smaller than in the perpendicular direction, but in the former case the image is sharper (has a better contrast).



**Figure 2.** Specklograms for the laser beam reflected from the surface of a dry gypsum sample in the mirror reflection direction (a) and perpendicular to the sample surface (b).

Fig. 3 presents the dependence of the reflected light intensity on the position (given by the point number) along arbitrarily selected line of the specklogram. This dependence gives some of the speckle parameters such as the average line length, the area under the curve, the average intensity (dashed line), the number and the average diameter of speckles or the image contrast. By using the histogram shown in Fig. 4, we can calculate the average intensity of the reflected light and statistical parameters such as the speclogram skewness and its intensity kurtosis.

## **3.** Experimental results

The preliminary experiment [6] was conducted using the setup described above. The specklograms of dummy samples saturate with water were detected at a constant temperature



Figure 3. Dependence of the reflected light intensity on the position on an aribtrarily chosen horizontal line.



Figure 4. Histogram of the reflected light intensity digitised over gray levels.

22°C. It was found that: the area under the curve for the mirror reflection direction (corresponding to the average light intensity) decreased quasi-linearly with increasing water content in the sample. Thus, the specklogram contrast and kurtosis increase with increasing water content. On the contrary, for the perpendicular direction, the area under the curve, the curve length and average light intensity decrease with the water content, specklogram contrast and kurtosis increase. The other speckle parameters vary negligibly.

Further studies were conducted under conditions close to the natural weather, by slowly cooling samples  $(0.5 - 2.0^{\circ}C/h)$  from room temperature to about  $-1^{\circ}C$  on the Peltier table. Figs 5,6 show the results.

Of interest is the temperature dependence of the skewness of specklograms detected perpendicular to the sample surface (Fig. 5a), since it has the minimum at the temperature  $T_0$  at which the condensation of water vapour probably starts on the sample surface (the dew point). This temperature  $T_0$  corresponds to the maximum on the area curves detected both perpendicular to the sample surface (Fig. 5b) and in the mirror reflection direction (Fig. 6a). These curves can be approximated by the third order polynomial. Fig. 6b shows the temperature dependence of the kurtosis obtained for the mirror reflection direction with a maximum at  $T_0$  (see Figs 5, 6). The temperature  $T_0 \approx 11^{\circ}$ C theoretically calculated for the smooth surface.



**Figure 5.** Dependences of the specklogram skewness (a) and the area under the curve in Fig. 3 (b) on the temperature of the gypsum sample surface with porosity of about 60 % for the specklograms detected in the direction perpendicular to the sample surface (R is the correlation coefficient).



**Figure 6.** Dependences of the area under the curve in Fig. 3 (a) and the speclogram kurtosis (b) on the temperature of the gypsum sample surface with porosity of about 60% for specklograms detected in the mirror reflection direction.

## 4. Conclusions

The dependence of the laser speckle parameters on the water content in the surface of porous bodies was found. This dependence is important for the understanding of the interaction of laser radiation with porous bodies and for future applications (for example, the remote control of the humidity of walls of ancient buildings, museums, etc.). The other applications of the method, such as the study of the effect of the air motion over the sample surface and in a laser beam as well as the sensitivity of the method, are the subject of further studies.

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