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The internal defects detection in crystals by digital holographic methods

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Abstract. The internal defects detection method is suggested for crystals intended for the use in the IR part of spectrum. The method is tested on samples of the $ZnGeP_2$ monocrystals, the experimental results are shown.

1. Introduction

Nowadays the production of new materials is rapidly developing, and these materials are widely used in different areas of science and technology. To provide the preset materials useful properties and to ensure the reliability of their further use, the well-honed and certified procedures of their manufacturing and quality control are required. In the case of growing crystals for infrared range of wavelength (IRC) the procedure of quality control is required at different stages of their growth and processing. Worth noting that such procedure is not always implemented in practice.

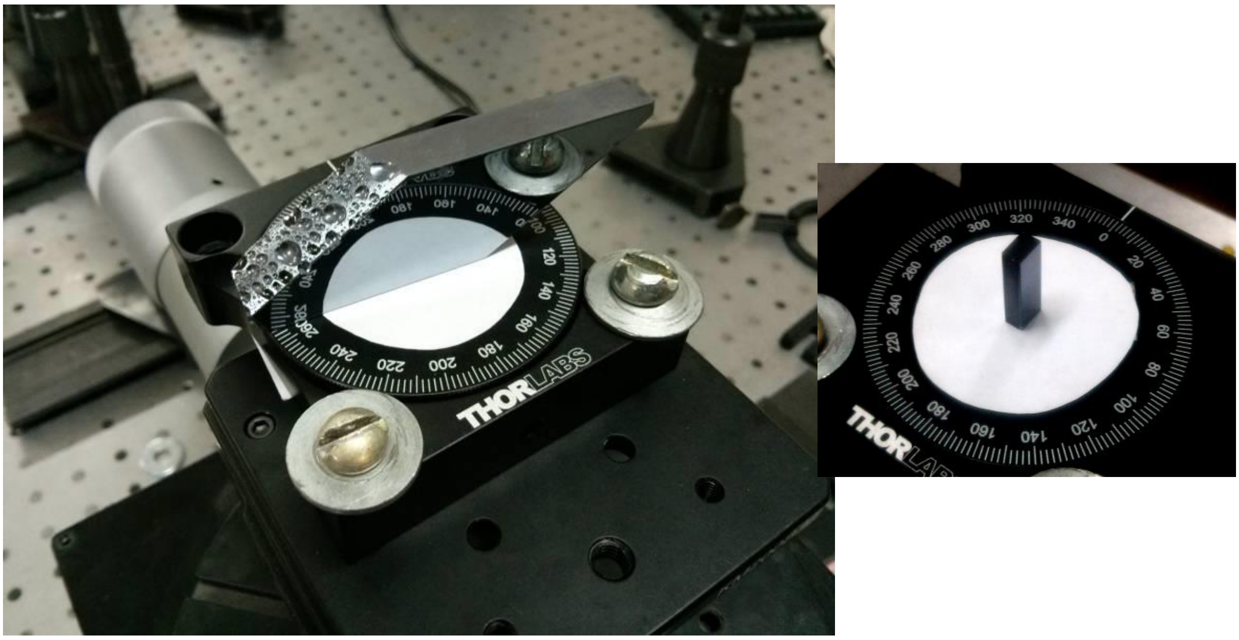
One of the most important indicators of the quality of optical materials is presence or absence the surface and internal defects. The first type of defects includes scratches, roughness, chips, surface non-flatness and tends to occur due to imperfections in the surface treatment of the sample and / or improper working. The presence of such defects can be quite easily founded using microscopic and interference methods [1,2]. They can be eliminated by additional processing of the sample surface (if they are not internal irregularities, such as cracks and striae that appeared on the surface). The second type of defects is internal defects that include internal cracks and inhomogeneity of chemical composition, and physical inhomogeneity of the crystal structure. Their appearance is primarily dealing with the material manufacturing technology and defects can not be technically removed from the final sample. The presence of internal defects in the crystal is the main factor in terms of its use availability. For example, if a crystal is used as a laser active medium the high energy pumping may lead to its degradation

Conventionally, the detection of defects in the sample is performed after full manufacturing cycle. The quality control is usually performed by its testing on destruction. For example, a powerful laser radiation is injected in optical crystals, and defective samples are degraded. Thus there is a chance when the sample with defects passed such test. In this situation the sizes of defects after tests may be increased, and they can cause unexpected degradation during the sample further operation. Therefore, a reliable quality control of manufactured samples is an actual task.

At cutting blanks stage the quality visualization of existing defects (if any) is quite important. The knowing the spatial location of the defects and their sizes can allow performing the cutting procedure when the resulting samples have no defects. Such approach leads to increasing the efficiency of sample preparation and allows improving the technological process. Examples of a single crystal $ZnGeP_2$ before and a blank after the cutting stage are shown in Fig. 1

In this paper, we propose to detect contactless "defective" spaces in blank on the crystal cutting stage before testing on radiation strength. This method conducts without destruction (degradation) of blank, and will significantly improve the efficiency of crystal quality control. The use of the results of this method can lead to reduction of required resources (financial and time) due to optimization of single crystal cutting procedure.

The aim of this work is to develop methods, hardware and software for the detection, measurement and identification of internal defects in crystals intended for the use in the infrared wavelength range (IRC).



a b

Figure 1. Example of $ZnGeP_2$ single crystal before (a) and blank after (b) the blank cutting stage

2. Techniques description

To detect internal defects in crystals a method based on in-line digital holograms registration is suggested. Thus the wavelength of the coherent illumination has to be selected in the area of sample transparency.

The procedure of further hologram reconstruction and data processing allow evaluating the size, shape and 3D location of all internal defects in field of vision. The method compares favorably with analogs as it is non-destructive and allows exploring the entire volume of the sample recorded on the hologram with high resolution.

Technique of detection of internal defects in crystal is worked on $ZnGeP_2$ single crystals. These single crystals have research and applied interest [3,4], because they are most effective nonlinear optical materials for the near and mid-infrared region (wavelengths of 0.7-12.0 μm). They are used for CO_2 and CO lasers harmonics generation, optical parametric oscillator (covering the wavelength range of 3-10 microns) and for coherent THz radiation sources creation. The main advantages of the single crystal are high threshold of optical breakdown, good thermal conductivity, mechanical strength, and resistance to high humidity and aggressive media.

To register digital hologram of internal volume of the sample it is necessary to choose the light source and receiver correctly. On the one hand, the sample has to be transparent to the laser source radiation; on the other hand, the receiver must be able to register this radiation.

Fig. 2 shows the spectral transmission of single crystals ZnGeP2 [5] and the typical spectral sensitivity of the CCD camera (for example, Videoscan 4021) [6]. The diagrams show that the use of the YAG: Nd laser, generating 1053nm wavelength may provide the registration of digital holograms of the sample. At the given wavelength single crystal transmits radiation, although it has a significant absorption. The curve in Figure 2b does not allow saying definitely that the CCD receiver is able to register this radiation. So this fact further will be confirmed experimentally.

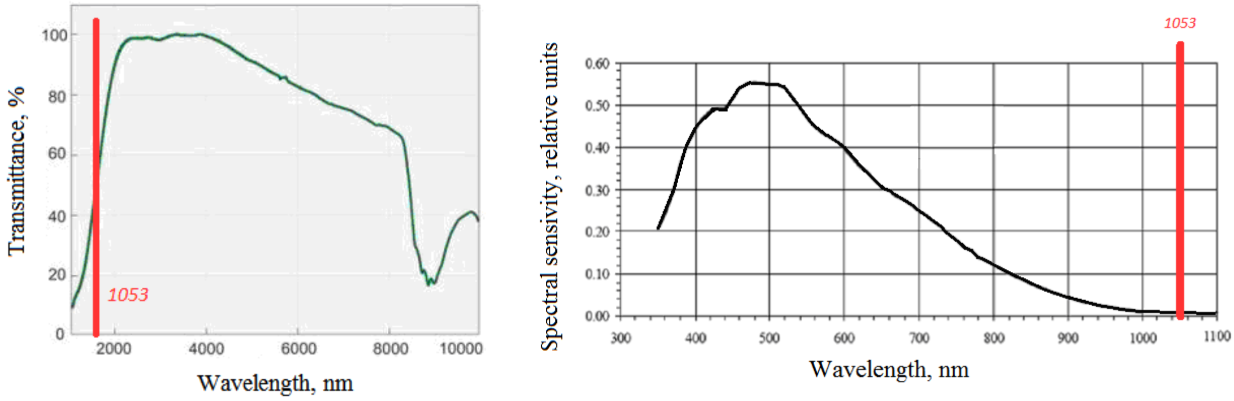


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Figure 2. The spectral transmittance of the single crystal sample $ZnGeP_2$ (a) [5] and the curve of Videoscan 4021 CCD-camera spectral sensitivity [6]

The information can be extracted from digital hologram about size, shape 3D position of all defects located in volume of the sample and registered on hologram The procedure of holographic data processing and information extracting is described in detail in [7,8], and is not the subject of investigation in this paper.

3. Results and discussion

To detect internal defects in IRC the in-line scheme of digital holograms registration is implemented. YAG: Nd DTL-399QT diode-pumped laser that generates at three wavelengths (1053, 527 and 351 nm) simultaneously is used. To receive the laser radiation CCD camera Videoscan 4021 is used. The optical system adjustment is carried out on 527 nm wavelength, and after adjustment this radiation is eliminated by a filter. As objects of holographing in this experiment a single crystal $ZnGeP_2$ and opaque bolt behind it are used (Fig. 3a). An example of a registered digital hologram is shown in Fig. 3b It is seen that the radiation passed through the sample is recorded CCD receiver. This fact confirms that the used laser source and CCD camera provide recording digital holograms of the internal volume of single crystal.

A number of digital holograms of optical single crystals $ZnGeP_2$ are recorded in a laboratory experiment. To increase the image contrast the background was subtracted, that was previously recorded in the absence of testing crystal (this procedure is illustrated in Fig.4 a-c). Focused reconstructed images of crystals scratches located on its different surfaces (marked by white rings) are presented on Fig. 4d,e. Defects which exceed the size of 20 microns (that corresponds to the scheme spatial resolution) are not found in the volume of a single crystal between these scratches. The detection of smaller defects requires the use of additional microscopic system.

Digital holograms of various samples are given as examples in Fig. 5. Dark areas near the boundaries of the single crystals are located in the pictures. One of the reasons of their appearance is related to nonflatness and nonparallelism of sample surfaces, and lead to vignetting (refractive index of $ZnGeP_2$ is more than 3.1).

Examples of internal defects detected for another single crystal are shown in Fig. 6a According to the number of detected defects, it is possible to reject it as defective and prevent further test.

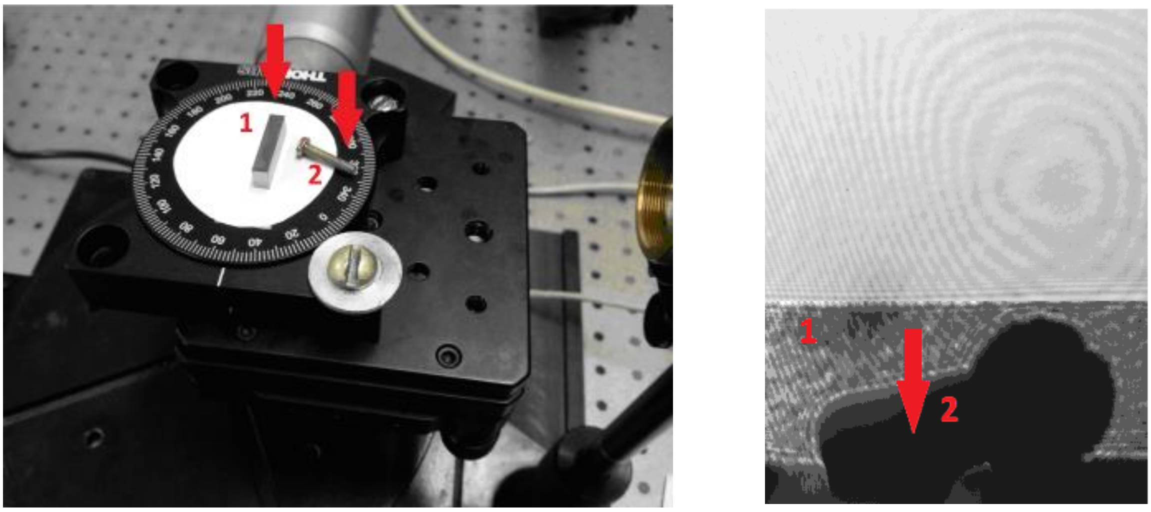

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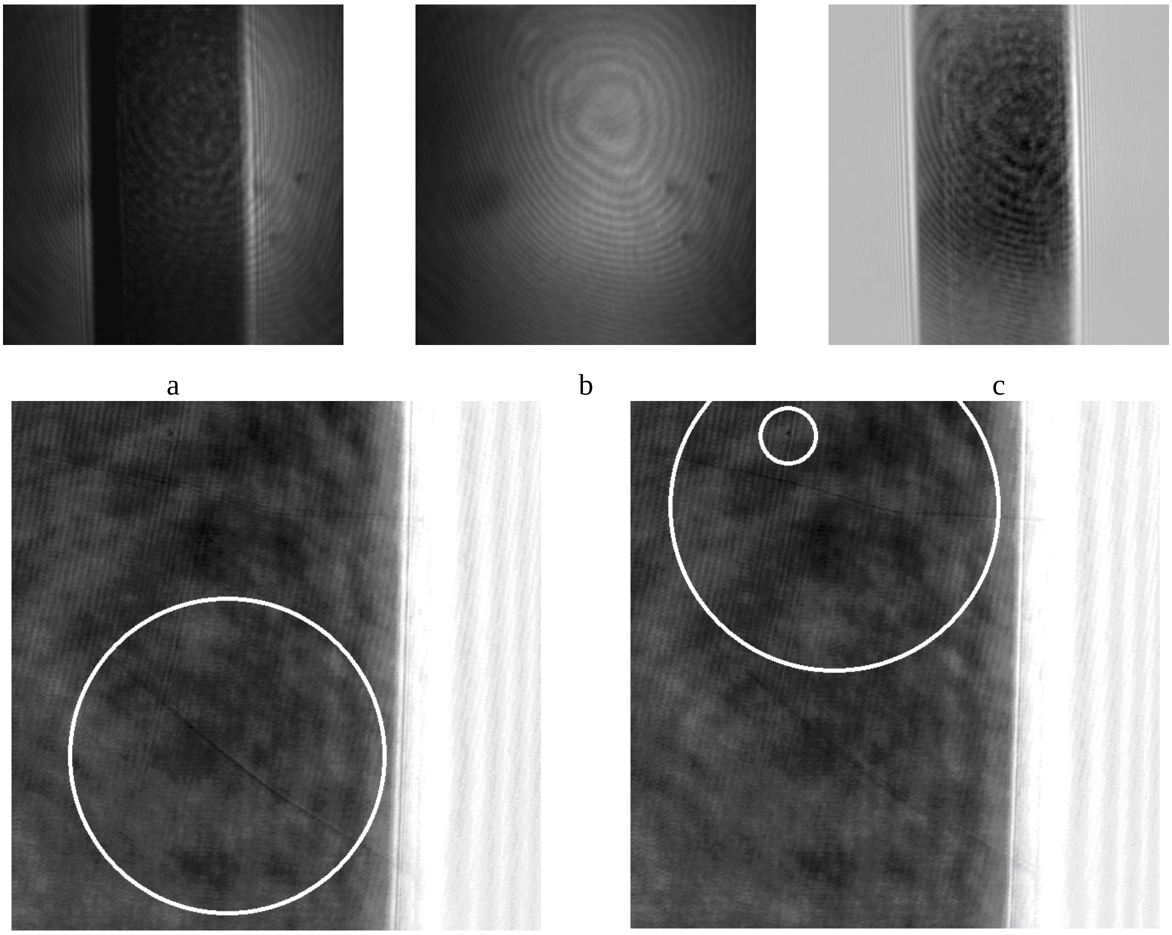
Figure 3. Subject scene (a) and its digital hologram (b).



d e

Figure 4. Single crystal $ZnGeP_2$ defects investigation by digital holography
Note: a digital hologram of $ZnGeP_2$ sample, b – background, c - result of background subtraction, d, e - reconstructed images of single crystal scratches on different surfaces of the sample.

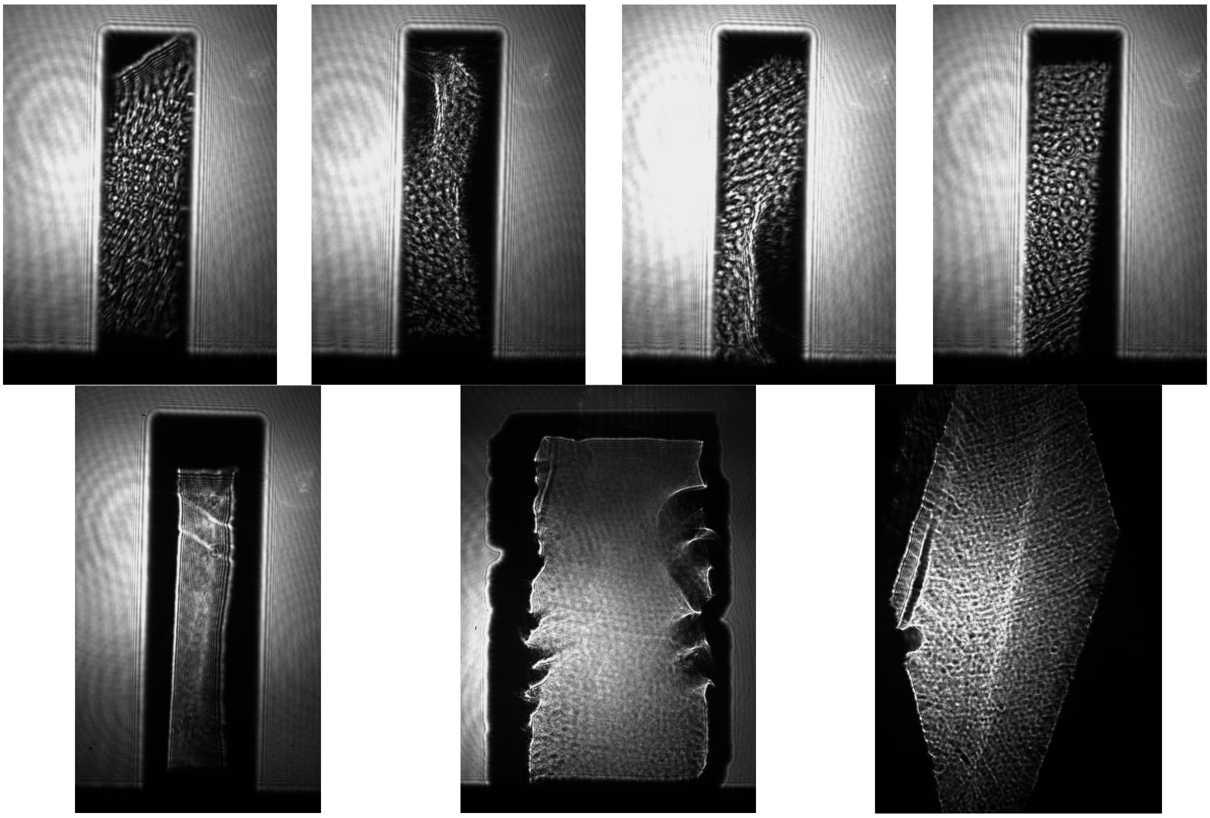
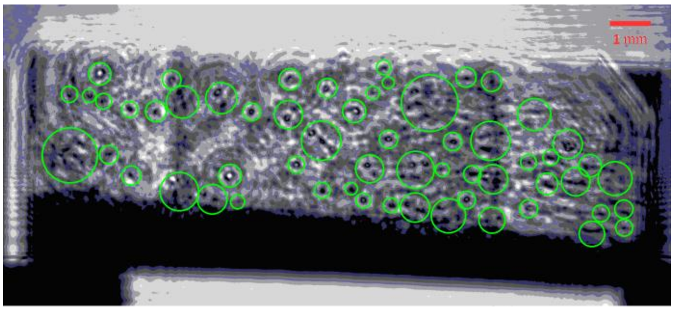


Figure 5. Digital holograms of several single crystals $ZnGeP_2$



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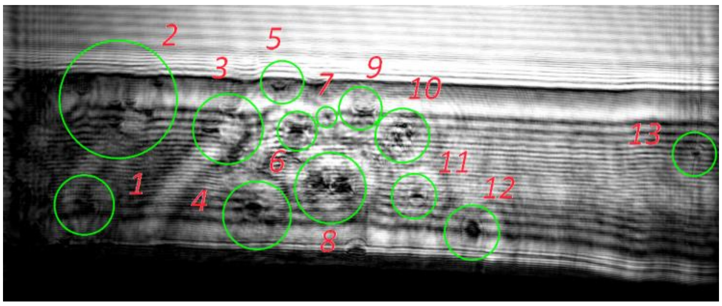


b

Figure 6. Defects in $ZnGeP_2$ single crystal visualized by holographic visor at 1.06 nm

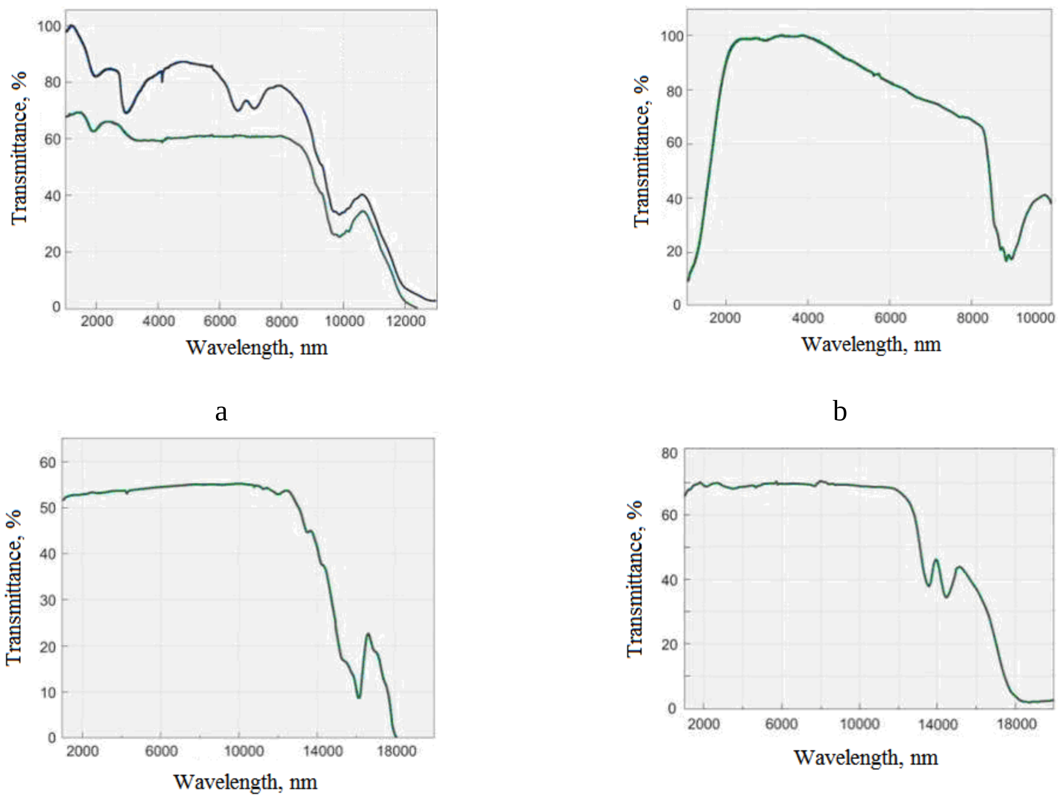
The images reconstructed from a digital hologram, allow us to determine the type of defect, to measure its dimensions and position (Fig. 6a). Such data can not be received by shadow projection image only. The results of digital hologram processing of another single crystal sample are presented below. For advanced visual representation of received data, 2D display of reconstructed volume holographic image of the sample according to the method described in [9] is used. Based on received data the size of defects are determined and presented in Table 1.

Table 1. Sizes of defects in $ZnGeP_2$ sample defined by digital holography

Defect №	1	2	3	4	5	6	7	8	9	10	11	12	13
Size, μm	150	100	100	150	120	300	50	400	150	100	100	350	80

4. Conclusion

Results of this paper show that in order to get successful implementation of the quality control method it is very important to match the crystal transmittance, the wavelength of the laser radiation and the spectral sensitivity of the CCD. Since the crystals may have very different spectral transmission (Fig. 7 shows the diagrams for some IRC), it is necessary to choose properly the receiver and the laser source in each case



a b c d

Figure 7. The spectral transmittance of single crystals: a – $AgGaS_2$, b – $AgGaSe_2$, c – GaSe, d – ZnGeP [5]

The method proposed in this paper is tested on 20 samples, defective blanks are identified. It was established experimentally that the method allows detecting and evaluating the location of internal $ZnGeP_2$ single crystals defects, the presence of chips, cracks, and the irregularities of refractive index.

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