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USE OF BISTABLE CENTERS IN CdF_2 FOR HOLOGRAPHIC RECORDING

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It is shown that metastable centers in various semiconductors can be used for efficient hologram recording. This type of holographic materials has a very high dynamic range and sensitivity. Their major drawback for possible applications is low metastability temperature. This problem can be overcome by using CdF_2:Ga crystals, which exhibit metastability below 240 K. This material is suitable for writing thick and multiple holograms both in static and dynamic regimes.

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1. Introduction

A search for materials for high density information storage has been continued already for decades. Recently much attention has been devoted to holographic techniques since it seems that holography may be the optimal method of information storage [1, 2]. The theoretical limit of holographic storage capacity is equal approximately to $1/\lambda^3$ (λ is the laser wavelength used for recording) that is of the order of several Tbytes/cm^3. However the main advantage of holography is probably very fast data access time. It seems to be quite feasible now to build holographic memory with storage capacity of the order of Tbytes/cm^3, with a transfer rate faster than 1 Gbytes/s and random access time of about 100 μs [1]. The first commercially available holographic devices show that possible applications of holography could be very broad [3].

The photorefractive effect based upon the local change of the refractive coefficient via the electric field patterns photogenerated in the host is thoroughly used for holographic recording. Most of holographic memories use this effect as the storage mechanism in various electrooptic materials, including many oxide crystals, various semiconductors, photorefractive polymers and semiconductor quantum structures. The $LiNbO_3$ crystals have been found to be one of the best for applications in holography [4, 1]. Although they have already been optimized for writing holographic patterns and found some practical applications, the search for alternative writing mechanisms and/or materials continues.

Recently, a novel approach to the problem was proposed. There is a class of defects in solids that exhibits photoinduced metastability at low temperatures. Illumination above a photoionization threshold for the localized state of the metastable defect, either generates carriers in the conduction band or/and populates hydrogen-like effective mass, bound states of the defect. At low temperatures, carriers cannot return to the initial deep state due to the vibronic barrier separating the ground, localized state from all effective mass excited states and the conduction band [5–7]. The defect phototransformation causes bleaching of the deep state absorption and the appearance of absorption related to the metastable occupied state (either bound state absorption or free carrier absorption). A change in the bound carrier localization causes a local change of the polarizability, and thus a change of the local refractive index. If all relevant optical transitions are allowed, the resulting change of the local refractive index can be large ($\Delta n \approx 10^{-4}$ to 10^{-3} for a density of photoinduced states in the 10^{18} cm^{-3} range and with readout wavelengths between 0.5 and 1.5 μm) [8, 9].

Various metastable defects have been identified in many semiconductors. Among them the best known are the EL2 defect in GaAs, DX or the DX-like centers in GaAlAs and other III–V and II–VI semiconductors. These metastable centers have been proposed for applications in nonlinear optics and holography for several years. Klein et al. [10] have used EL2 defect in GaAs for room temperature phase-conjugation by four-wave mixing at 1.06 μm. By applying the ac square-wave electric field they were able to enhance the beam-coupling gain and the degenerate four-wave mixing reflectivity. Semi-permanent holographic gratings were written at the same laser wavelength in semi-insulating liquid-encapsulated Czochralski GaAs at temperatures below 140 K [11]. The crystal exhibited much larger diffraction efficiency than expected from the traditional photorefractive diffraction efficiency for the same temperature, which has been attributed to the quasi-equilibrium Fermi-level modulation, during which the EL2 defects were optically quenched within the bright fringes of the hologram but were left unaffected in the dark fringes. The band bending of the formed in this way p–i–p–i superlattice generated strong electrooptic gratings, with the grating amplitudes much larger than possible by thermal diffusion alone. The 1.32 μm radiation was used for non-destructive readout of the holograms. Also Delaye et al. studied photorefractive wave mixing in undoped Czochralski GaAs at various wavelengths in the near-infrared region [12].

Holography has been also used for modulation of the two-dimensional carrier density in GaAs–$Al_xGa_{1-x}As$ heterojunction by Weiss et al. [13] in order to obtain direct information about the density of states in such a structure. Diffraction from optically written persistent plasma gratings in doped compound semiconductors have been reported in several papers by Linke et al. [18]. They used $Al_xGa_{1-x}As$ layers with the DX centers for recording phase holograms. Experimentally observed induced changes of the refractive index of the layer were very large and equal to about 5×10^{-3}. They also reported successful optical writing of the metallic patterns in insulating $Al_xGa_{1-x}As$:DX epilayers, resulting in modulation of the free-carrier density, which was evidenced by an anisotropy of the sample conductance parallel and perpendicular to the photoinduced fringes [14].

The anisotropy was persistent at low temperatures. It has been established that the features can be written with a better than 1000 Å resolution.

The thick plasma gratings were recorded in CdZnTe:In crystals [15], which is an example of the II–VI compound semiconductors with the DX-like centers. By using near-band edge excitation (λ = 823 nm) the persistent photoconductivity has been induced resulting from ionization of the DX centers. Two gratings were recorded in the same volume of the crystals; subsequently they were decoded by angular multiplexing at 1530 nm wavelength. A very high sensitivity of the photorefractive recording process, defined as the refractive index change per incident fluence, was noticed. It was about two orders of magnitude greater than in $BaTiO_3$ crystal, very often studied for holographic recording.

Persistent volume absorption holograms were also recorded in the other III–V semiconductor crystals with DX centers, namely heavily doped AlSb:Se [16] and recently in CdMnTeSe:In [17].

All above-mentioned materials suffer an important drawback: the metastability temperatures of all the systems studied are below 140 K. The metastability temperature is related to the value of the energy barrier separating the metastable and the ground state of the system. The eventual application of this class of materials to holographic optical memories would require larger regeneration energies.

One of the material that may offer desired properties is CdF_2. It is already well established that some of the group III dopants introduce bistable centers in this crystal [18, 19]. In fact, the CdF_2:In crystals were one of the first, where this type of defect bistability has been identified [18, 19]. Later on Ga was shown to exhibit the same bistable properties, but with a notably higher temperature [19]. According to the original model of bistability [18], the ground state of In and Ga impurities is localized. Ionization of this state reduces local screening of the Coulomb attraction between neighboring F^- ions and defect core, thus causing a local collapse of the lattice. The electron returns to the ground state freely only at high temperatures. At lower temperatures it is being captured at the hydrogenic excited state of the impurity. This state, as well as the ionized state of the defect, is separated by vibronic barrier from the ground state, which is a consequence of the local lattice relaxation.

Very recently the general model of the bistabilities (the existence of two states strongly differing in localization, separated by a vibronic barrier resulting from a large lattice relaxation) has received a strong support from ultra-precise measurement of the photoinduced lattice dilatation [20]. Indeed, the crystal contracts metastably at low temperature upon illumination that causes photoionization of the deep state. The transfer of the electrons from the localized to delocalized state must cause a local change of the polarizability, and thus should affect the local refractive index. Guided by these considerations we showed that indeed such a process occurs and the metastable change of the refractive index is in the 10^{-4} range [21]. Crystals are very sensitive and thus we could successfully perform writing holographic patterns first in indium doped crystals [9], and subsequently in CdF_2:Ga [21]. The latter are especially interesting, as the holographic storage is efficient up to room temperature.

In the second part of this paper we shall reanalyze the holographic measurements within the framework of a newer model of bistable defects in CdF_2. In contrast to the original one-electron model, the new one requires two electrons to form the deep, localized state, and thus, the Hubbard correlation energy U must be negative. It must be pointed out that holographic data alone are not sufficient to decide the sign of U. In fact, the shape of the temperature dependence of the diffraction efficiency is more consistent with older ($U > 0$) model [18]. The kinetics of the infrared ionization absorption of metastable impurity, the measurements of the quantum efficiency of the phototransformation between the deep and the shallow state [22] and recently the magnetic susceptibility measurements of CdF_2:In [23, 24] strongly indicate the "negative-U" character of bistable donors in CdF_2, similar to the properties of DX centers in III–V compounds.

2. Metastability of CdF_2:In and Ga crystals

The bulk CdF_2 crystals are grown by the modified Bridgman method at temperature equal to about 1050°C. Crystals can be easily doped with many 2+ and 3+ dopants. The absorption spectra of CdF_2:In and CdF_2:Ga crystals are shown in Fig. 1 [18, 19]. The visible-ultraviolet (VIS-UV) absorption is dominated by a photoionization process of the ground localized In and Ga states. Illumination with light, coinciding with these absorption bands, causes their bleaching and the appearance of intense infrared (IR) absorption peaking at about 8 μm. The IR absorption originates from photoionization of the hydrogen-like, photo-populated, bound state of the In or Ga donor and is quite similar to IR absorption in semiconducting CdF_2 crystals doped with other shallow donors. The absorption spectra of CdF_2:In and CdF_2:Ga crystals are quite similar. The VIS-UV band of the gallium doped crystals is shifted more towards the UV than the spectrum of CdF_2:In.

At temperatures close to the onset of metastability, the free carrier concentration is negligible. Thus, free carrier absorption, that has been used in forming

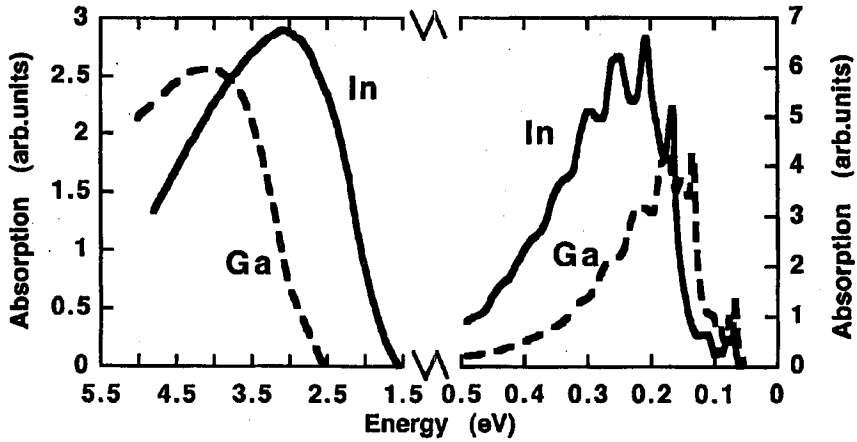

Fig. 1. The absorption spectra of CdF_2 crystals doped with In and Ga at $T = 4$ K.

holographic patterns in III–V compounds with DX centers, cannot be used here. Therefore, the writing process in CdF_2:Ga or In should be more local than in the III–V semiconductors with DX centers.

The "negative-U" character of the metastable centers in CdF_2 implies a bimolecular character of the rate equations describing the deep-shallow center transformation upon illumination and temperature [22]. In the simplified case the concentration of the shallow centers N_s ($Me^{3+} + e^-$) is described by the following equation:

$$\frac{dN_s}{dt} = -cN_s^2 + I\sigma(N_0 - N_s), \tag{1}$$

where c is the kinetics parameter, I is the light intensity, σ is the photoionization cross-section of the deep state and N_0 is the total concentration of the "active" ions.

Then the concentration of the shallow donors after the switching of the light at the time $t = 0$ is described by the formula

$$N_s(t) = \frac{N_s^0}{N_s^0 ct + 1}, \tag{2}$$

where N_s^0 is the initial concentration of shallow centers at $t = 0$.

The kinetics parameter c is exponentially dependent on temperature

$$c(T) = c(0)\exp\left(-\frac{\Delta E}{kT}\right) \tag{3}$$

with ΔE being the sum of the barrier separating shallow and deep state and the shallow donor binding energy. The very strong change in the electron localization at the In or Ga impurity during phototransformation must lead to a change in the local polarizability, and thus a change in the refractive index of the host. We estimated the magnitude of this effect employing a two-oscillator model, in which the broad, impurity photoionization bands were replaced by two effective oscillators, characterized by the oscillator strengths f_i and the center frequencies ω_i. For the IR band, this frequency is much smaller than the laser probe frequency, and thus may be omitted. Therefore [9]

$$\Delta n = \frac{2\pi e^2}{mn}\left(\frac{\Delta N_{VIS} f_{VIS}}{\omega_{VIS}^2 - \omega^2} - \frac{\Delta N_{IR} f_{IR}}{\omega^2}\right), \tag{4}$$

where ΔN is the change of the concentration of the centers giving rise to VIS or IR absorption, f is the value of the oscillator strength of the respective optical absorption, ω_{VIS} is the effective frequency for the VIS absorption, ω is the laser frequency, m is the electron mass, and n is the refractive index of the host. According to the "negative-U" model of metastable donors in CdF_2 $\Delta N_{IR} = -2\Delta N_{VIS}$. Therefore, we may further estimate the changes of the refractive index as equal to

$$\Delta n = -\frac{2\pi e^2}{mn}\Delta N_{VIS}\left(\frac{f_{VIS}}{\omega_{VIS}^2 - \omega^2} + \frac{2f_{IR}}{\omega^2}\right). \tag{5}$$

It is clear from the above equation that both absorption bands add in phase to the change of refractive index. The same would apply if some of the photogenerated carriers were in the conduction band (persistent photoconductivity phenomenon).

As the oscillator strength f_{IR} is close to unity (it is the photoionization spectrum of a quasi hydrogenic impurity state) [18], the IR term generally dominates, unless f_{VIS} is much larger than unity.

In CdF_2 crystals the refractive index n for the visible range is close to 1.5. Taking the values of both oscillator strengths as equal to unity and estimating $\lambda_{VIS} = 2\pi/\omega_{VIS} = 0.3$ μm, we may expect a change of the refractive index Δn of about -10^{-4} for the probe beam at the wavelength of $\lambda = 0.5$ μm and the concentration of In donors of 10^{18} cm^{-3}. Just this value was measured by us for CdF_2:In crystals [9], confirming validity of the above estimation.

3. Four-wave mixing experiments in CdF_2:In and Ga crystals

The changes of the refractive index Δn in CdF_2:In semiconducting crystals are very large that should result in high scattering efficiency of the four-wave mixing (FWM) performed in these crystals. The scattering efficiency η of the FWM is described by the Kogelnik formula [25]

$$\eta = \exp\left(-\frac{ad}{\cos\alpha}\right)\left(\sin^2\frac{\pi\Delta n d}{\lambda\cos\alpha} + \sinh^2\frac{\Delta a d}{4\cos\alpha}\right), \tag{6}$$

where a is the absorption coefficient, Δa and Δn are the induced changes of the absorption coefficient and the refractive index, respectively, between the peaks and the valleys of the gratings, d is the thickness of the hologram and α is the Bragg angle.

The first term in Eq. (6) describes the contribution to the scattering efficiency from the phase gratings and the second one — from the amplitude (absorption) grating. Theoretically, the scattering efficiency from the phase grating could be equal even up to 100%. The maximum scattering efficiency from the absorption grating cannot exceed 4%. This provides a convenient test for distinguishing the dominating mechanism of grating formation.

We performed a standard FWM experiment of writing a grating and measuring the light diffraction from a photoinduced grating in a set-up consisting of a CW Innova Ar-laser as a writing medium and a He–Ne laser as a source of the probe beam. CdF_2 crystals were placed in a continuous flow cryostat. The temperature dependence of the FWM scattering efficiency is presented in Fig. 2. The efficiency critically depends on temperature. The maximum of the efficiency occurs for an In doped sample at about 120 K. In contrast to that the scattering efficiency for CdF_2:Ga crystal peaks at about 280 K, very close to the room temperature. The bell-like shapes of the scattering efficiency curves are related to the processes responsible for the grating creation and destruction. At high temperatures the thermal recovery from the shallow to the deep state is responsible for the grating destruction. At low temperatures the thermal recovery is very slow and any scattered light from the cryostat windows or nonequal beam intensity causes phototransformation of the active centers not only in the peak of the grating but also in its valleys. This ultimately causes bleaching of all the illuminated regions and causes a lack of difference of the refractive indices between the grating peaks and valleys. Therefore the grating disappears. A maximum in the diffraction effi-

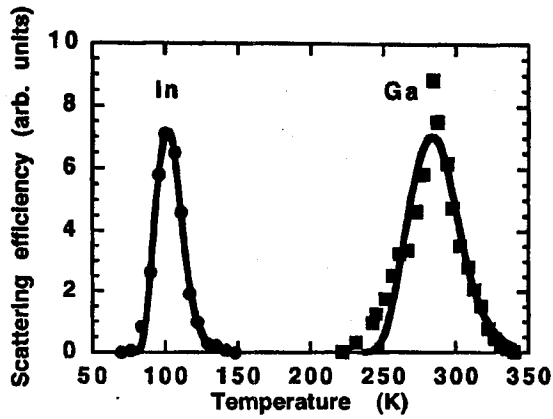

Fig. 2. The temperature dependence of the c.w. four-wave mixing scattering efficiency of the CdF_2:In and CdF_2:Ga crystals.

ciency occurs at the temperature at which the speed of incoherent erasure equals the thermal recovery rate via the barrier separating the two states. Therefore the temperature, at which the maximum of the FWM scattering efficiency occurs, is proportional to the value of the energy barrier. By solving the rate equations (1) at the equilibrium condition for the peaks and for the valleys of the grating it is possible to calculate a theoretical temperature dependence of the FWM scattering efficiency, using the Kogelnik formula. The computer fits of these dependences are marked by solid lines in Fig. 2. Here, the values of the energy barrier were treated as a fitting parameter. Their values, obtained from the fits, are equal to 240 ± 25 meV and 920 ± 260 meV for In and Ga doped samples, respectively. The larger discrepancy between the theory and experiment for CdF_2:Ga crystal are probably related to the larger inhomogeneity of the CdF_2:Ga crystal.

A similar fit has been done by us previously, but for a standard $U > 0$ model. As expected, energies are smaller by about a factor of 2, coming from the difference in the number of electrons in the ground state [21].

The changes of the refractive index of the CdF_2 crystals doped with metastable centers depend on the wavelength, according to the double oscillator model, described by Eqs. (4) and (5). Its validity has been checked also using the FWM experiments, using the Kogelnik formula (Eq. (6)). Since the maximum value of scattering efficiencies, obtained in our previous experiments, have been higher than 10% for both CdF_2:In and CdF_2:Ga, we assume that refractive index changes are responsible for the grating formation in CdF_2 crystals [9, 21]. This allows us to neglect the contribution to the scattering efficiency from the amplitude grating (the second term in Eq. (6)). The scattering efficiency at various wavelengths of the read beam was measured, keeping the wavelength and the power of the write beams constant during the experiment. This is an indirect measure of the refractive index changes at various wavelengths. The results of the experiment are presented in Fig. 3. The value of the refractive index changes increases with increasing wavelength for both CdF_2:In and CdF_2:Ga crystals. They are in very

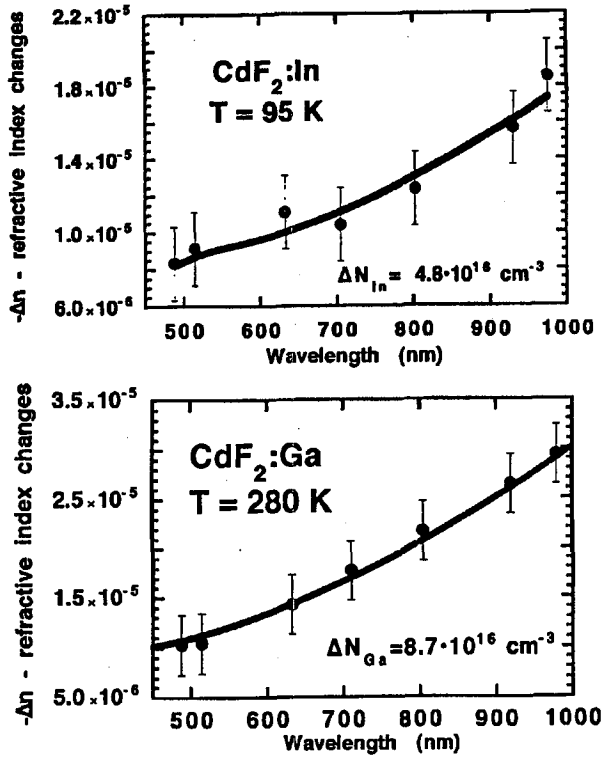

Fig. 3. The spectral dependence of the photoinduced refractive index changes in the CdF_2:In and CdF_2:Ga crystals, measured with the use of the FWM technique. The wavelength and the power of the write beams were kept constant during the experiment ($\lambda_{write}(CdF_2{:}In) = 514.5$ nm, $\lambda_{write}(CdF_2{:}Ga) = 457.9$ nm). The number of phototransformed centers ΔN is shown on the graph.

good agreement with the double-oscillator model, marked by the solid lines in Fig. 3, which confirms its validity for metastable centers in the CdF_2 crystals.

The most important parameter of the metastable center for the application is the metastability temperature, which is directly related to the value of the energy barrier separating shallow and the deep states of the metastable center. The value of the energy barrier can be established from the grating decay kinetics. For weak excitation conditions the FWM scattering efficiency is proportional to the square of the refractive index changes (see Eq. (6)), which are, in turn, proportional to the square of the population of the shallow states, described by Eq. (2). Therefore the FWM decay kinetics can be expressed as

$$\eta(t) = \left(\frac{A}{Act+1}\right)^2 . \tag{7}$$

The FWM grating decay kinetics have been measured in a set-up with a He–Ne laser as a reading beam. The decay kinetics both of CdF_2:In and CdF_2:Ga are strongly nonexponential. The decay kinetics can be fitted by Eq. (7) with good accuracy. The decay kinetics depend on the temperature and the FWM signal decays slower with a decrease in temperature. For CdF_2:In crystal the rate at which the signal decays is approximately the same for all temperatures below 90 K. This behavior is caused most probably by the influence of the read He–Ne beam on the FWM decay kinetics. The He–Ne beam partially erases the grating for the In doped sample since the 632.8 nm wavelength is at the edge of the deep state absorption band (see Fig. 1). This behavior is not observed for Ga-doped samples since the deep-state photoionization band is shifted towards the UV in these crystals. The temperature dependences of the kinetics parameters $c(T)$ for crystals doped with In and Ga have been obtained from the computer fits of the FWM decay kinetics. They are shown in Fig. 4. The activation energies, estimated

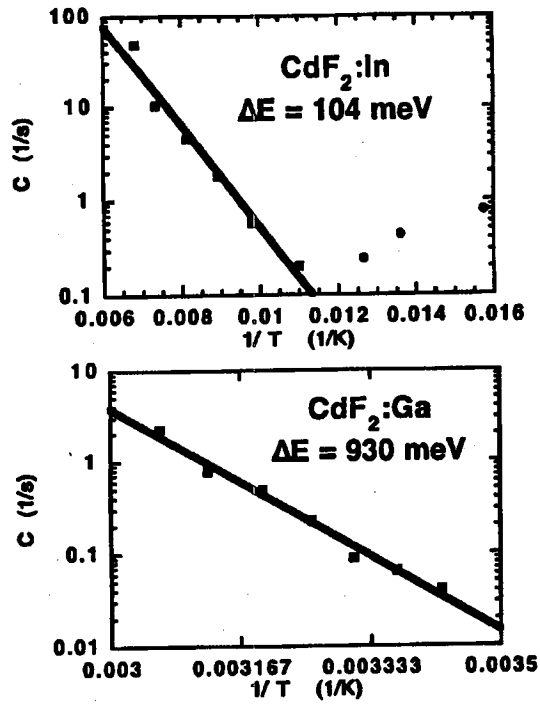

Fig. 4. The temperature dependence of the kinetics parameter, calculated from the decay kinetics of the FWM signal for the CdF_2:In and CdF_2:Ga crystals.

from the temperature dependences, are equal to about 100 meV and 920 meV for In and Ga doped crystals, respectively. For the CdF_2:Ga it is in very good agreement with the value obtained from the fits of the temperature dependence of

the scattering efficiency (see Fig. 2). The activation energy for In-doped sample is about two times smaller than that obtained from the temperature dependence of the scattering efficiency. This discrepancy could be also related to the partial erasing of the grating by the He–Ne laser.

It should be stressed once more that the same quality fits of the FWM decays could be obtained using a two-exponential formula, which can be a consequence of the crystal inhomogeneity or existence of two types of metastable centers in the crystals). Therefore the shape of the decay kinetics alone cannot be a test of "negative-U" properties of the metastable centers in the CdF_2 crystals. Similar as in the case of the diffraction efficiency, the barrier energies are larger by a factor of 2 than those obtained in a one-electron model [9, 21].

4. Conclusions

It has been shown that the metastable centers in semiconductors could be used in efficient hologram recording. The dynamic range of refractive index changes of such materials can be very high, that is directly related to the possible high concentration of the metastable centers in semiconductors and large oscillator strength of the photoionization transitions involved in the process of hologram recording. Low metastability temperature, usually below 140 K, is the main obstacle for possible applications of the metastable centers. It comes from relatively low value of the energy barrier separating shallow and deep states of the metastable centers. This problem could be circumvented by using the materials with metastable centers having higher metastability temperature. The CdF_2:Ga crystals offer such a possibility. The energy barrier separating metastable and ground states of the main defect in the CdF_2:Ga crystals is much higher than in the CdF_2:In crystals, as well as in the other metastable centers in other compound semiconductors. Therefore this center is metastable below 240 K. Additionally, bulk CdF_2 crystals can be easily grown. Thus, CdF_2 crystals are more suitable for writing thick and multiple holograms. Moreover, CdF_2 offers also a much broader spectral range for non-destructive scanning of the hologram, as compared to other semiconductors due to a large "optical window" — a spectral separation between the photoionization absorption of deep and shallow states of the metastable centers. Additionally, the spatial resolution is likely to be better due to very small carrier mobility in CdF_2. Further progress in increasing the metastability temperature above the room temperature can make semiconductors with metastable centers very attractive for applications.

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