Optical parameters of $Cd_{1-x}Fe_xSe$ and $Cd_{1-x}Fe_xTe$ by means of Kramers-Kronig analysis of reflectivity data

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The reflectivity spectra were measured in the energy range of $1.15\ \text{eV} \le h\nu \le 12\ \text{eV}$ for $Cd_{1-x}Fe_xTe$ and $Cd_{1-x}Fe_xSe$ semimagnetic semiconductor (SMSC) crystals, and a Kramers-Kronig analysis was used to determine the $\text{Re}\epsilon$ and $\text{Im}\epsilon$ spectral dependences from the results obtained. The spectra were interpreted on the basis of the binary-II-VI-compound band structures. The observed dependence of the positions and intensities of the spectral peaks on the Fe content allows us to estimate the contribution of Fe-derived states to the band structures of two SMSC with different crystalline structures: zinc blende for $Cd_{1-x}Fe_xTe$ and wurtzite for $Cd_{1-x}Fe_xSe$.

I. INTRODUCTION

The term "semimagnetic semiconductors" (SMSC's) or "diluted magnetic semiconductors" (DMS's) nowadays describes a fairly large group of ternary alloys of a semiconductor (commonly, a II-VI compound) and a magnetic transition-metal material (containing cations with high net spins, such as Mn, Fe, and Co). The first group of these materials to be obtained, and investigated for the longest time, were the Mn-based materials. The high spin ($\frac{5}{2}$) of Mn, and the relatively high solubility of manganese in II-VI compounds, made these systems the easiest ones on which to investigate the peculiar properties of SMSC's.[1] Results of these investigations led to the conclusion that interaction between the delocalized band electrons and the *d* electrons of Mn is responsible for the observed strong modification of the electrical and optical properties of SMSC's with respect to their parent II-VI compounds.[1]

Some of the conclusions seem to be general enough to justify a comparison and some analogies with the compounds containing different transition-metal ions. However, when some data on Fe-based semimagnetic semiconductors were collected, it became evident that these materials have similarities as well as differences with respect to the Mn-based compounds studied.[2-4] Standard optical and electrical methods applied to the new crystals containing Fe give results that often call for quite new interpretations, principally as a consequence of the presence of one more electron in the *d* shell in an Fe ion. This is the case when we are dealing with the reflectivity spectra of Fe-based semimagnetic semiconductors.

The reflectivity spectra of Mn-containing SMSC's [$Cd_{1-x}Mn_xTe$, $Hg_{1-x}Mn_xTe$, and $Cd_{1-x}Mn_xSe$ (Refs. 5-7)] show that an increase of Mn content leads to a slight change of the valence-band structure parameters, and to marked blurring of the spectra, especially for $x \ge 0.15$. The latter effect is a peculiar property of Mn-based materials, not seen for the other II-VI ternary alloys [e.g., $Zn_{1-x}Cd_xTe$ (Ref. 8)]. Such strong modification of the optical spectra confirms the important role played by the Mn ions in the formation of the band structure of the alloy, although, on the other hand, it makes it impossible to observe changes of the band-structure parameters in the whole range of Mn contents. These phenomena can occur for Fe-based materials, too. However, they must be weaker, since the solubility of Fe is much lower than that of Mn (maximum x is equal to about 0.1).

The purpose of this paper is to use the reflectivity data for two SMSC materials with different crystalline structure, $Cd_{1-x}Fe_xTe$ (zinc-blende structure) and $Cd_{1-x}Fe_xSe$ (wurtzite structure), to study their electronic structures, as well as to recognize the regions in the Brillouin zone for which the Fe contribution leads to changes of the electronic structure. The reflectivity spectra, and the experimental procedure by which they have been obtained, are presented in Sec. II. The curves obtained for the ternary crystals were compared with those obtained for CdTe or CdSe, respectively. The $\epsilon(h\nu)$ dependence, a result of a Kramers-Kronig analysis (Sec. III), enabled us to consider changes of the band structure caused by the presence of Fe ions in $Cd_{1-x}Fe_xTe$ and $Cd_{1-x}Fe_xSe$ crystals. These are then discussed, on the basis of published interpretations of the spectra of parent binary compounds, in Secs. IV and V.

II. EXPERIMENTAL CONDITIONS AND RESULTS

The reflectivity of $Cd_{1-x}Fe_xTe$ and $Cd_{1-x}Fe_xSe$, the experimental basis for our discussion, has been measured for light energy of $1.15\ \text{eV} \le h\nu \le 12\ \text{eV}$. An experimental setup with a prism monochromator was used for measurements covering the infrared, visible, and near-ultraviolet ranges.[9] A hydrogen lamp and a vacuum monochromator (based on the Seya-Namioka scheme) were used as the source of vacuum ultraviolet radiation. The radiation was unpolarized in the whole energy range. The energy resolution was kept at between 5 and 20 meV for light energies of 5 and 12 eV, respectively.

The $Cd_{1-x}Fe_xTe$ ($x=0.0$, 0.02, and 0.06) and $Cd_{1-x}Fe_xSe$ ($x=0.0$, 0.03, and 0.1) crystals investigated

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were grown by the modified Bridgman method at the Institute of Physics, Polish Academy of Sciences. Their surfaces were mechanically polished and etched just before the experiment.

In order to ensure correct extrapolation of the unknown low- and high-energy parts of the reflectivity curves in the Kramers-Kronig analysis, absorption spectra within some restricted region were necessary. Such data, for the absorption edge region, were taken from the already published results [for CdSe (Ref. 10) and $Cd_{0.97}Fe_{0.03}Se$ (Ref. 11)], or were obtained (for other materials) using a Perkin-Elmer-Hitachi spectrophotometer.

Results of the reflectivity experiments are shown in Figs. 1(a) and 1(b). The curves corresponding to the binary compounds (CdTe and CdSe) are consistent with published results,[12–15] and are a convenient background for analysis of the spectra of the semimagnetic semiconductors.

A. $Cd_{1-x}Fe_xTe$

The reflectivity spectrum of CdTe has been carefully analyzed by several authors, and the features observed have been ascribed to optical transitions at some points (or regions) in the Brillouin zone,[12,15,16] (see Table I).

A comparison of the spectra obtained for three samples with different Fe contents [Fig. 1(a)] suggests that introduction of some Fe ions into the cationic sublattice does not influence all the features in the same way. Although the principal peaks and shoulders can be discerned in the spectra of $Cd_{0.98}Fe_{0.02}Te$ and $Cd_{0.94}Fe_{0.06}Se$, the relations between the heights of the peaks change significantly. Since the changes are clearly correlated with an increase of Fe content, they can be regarded as a manifestation of the modifications arising in the band structure of the crystal. The assignments of the spectral features to optical transitions at various points in the **k** space enable us to identify the regions in the Brillouin zone where changes in the band structure are most pronounced. This observation will be supported and elaborated in the discussion of results of the Kramers-Kronig analysis of the spectra.

B. $Cd_{1-x}Fe_xSe$

Just like CdTe, CdSe has been carefully investigated, and most of the features discerned in the reflectivity spectra are ascribed to optical transitions between the electronic bands[14,17] (see Table II). Due to the hexagonal structure of CdSe, the optical transitions observed have been divided into two groups, according to the polarization of the light in which they can be observed. Since our spectra have been obtained for unpolarized incoming light, the structure shown in Fig. 1(b) is a mixture of the peaks and shoulders caused by transitions allowed for both perpendicular ($\mathbf{E}\perp\hat{\mathbf{c}}$), and parallel ($\mathbf{E}\parallel\hat{\mathbf{c}}$) polarizations, where **E** is the electric vector of the incident light.

The influence of the introduction of Fe on the spectra of $Cd_{1-x}Fe_xSe$ is qualitatively similar to that for $Cd_{1-x}Fe_xTe$. The selective change of some maxima shall be the basis for our conclusions about the modifications of the band structure, as was described for $Cd_{1-x}Fe_xTe$ in Sec. II A.

TABLE I. The energies of the peaks and shoulders discerned in the spectra of $Cd_{1-x}Fe_xTe$, together with their assignment to interband transitions.

Energies of the peaks						
Reflectivity Fe content			Imε Fe content			
0.0	0.02	0.06	0.0	0.02	0.06	Assignment
1.60	1.55	1.55	1.70	1.75	1.65	Γ_8-Γ_6 (Ref. 16)
2.35			2.45			Γ_7-Γ_6
3.25	3.25	3.25	3.30	3.25	3.30	$L_{4,5}$-L_6 (Ref. 12) Λ (0.43,0.43,0.43) (Ref. 16)
3.85	3.85	3.85	3.85	3.85	3.80	L_6-L_6 (Ref. 16) Λ (0.43,0.43,0.43) (Ref. 12)
5.35	5.40	5.40	5.20	5.35	5.20	Δ_5-Δ_5 (0.5,0,0), (0.75,0.25, 0.25), Δ_5-Δ_5 (0.75,0,0) (Ref. 16); Δ(0.7,0,0), (0.9,0.2,0.2), Σ(0.7,0.7,0), Δ(0.7,0,0), (0.35,0.07,0.7), Δ(0.2,0,0) (Ref. 12)
6.70	6.75	6.75	6.45	6.60	6.60	$L_{4,5}$-L_6 (Ref. 16); (0.6,0.5,0.2) (Ref. 12)
7.80	7.80	7.75	7.30	7.55	7.40	L_6-$L_{4,5}$ (Ref. 16); (0.6,0.2,0.1) (Ref. 12)
	8.70	8.50		8.45	8.30	VB-CB (at point U?)
9.10			8.75			
	9.35	9.35		9.25	9.20	
		10.35				
10.20	10.35		10.35	10.15	10.15	
		10.75				

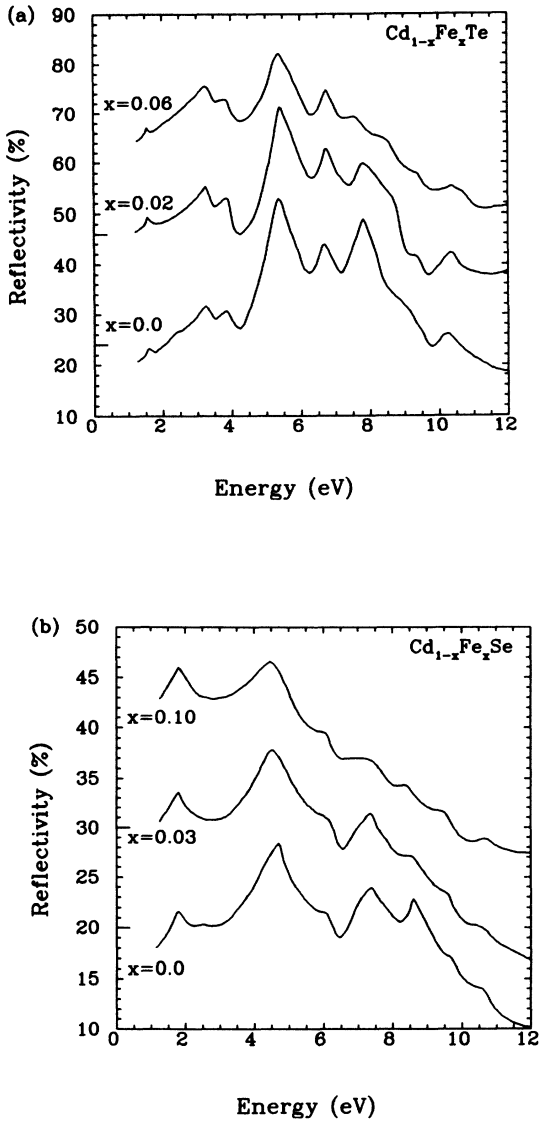


FIG. 1. The reflectivity spectra for (a) $Cd_{1-x}Fe_xTe$ and (b) $Cd_{1-x}Fe_xSe$.

III. KRAMERS-KRONIG ANALYSIS

The Kramers-Kronig analysis of the reflectivity spectra was performed by means of the formula[18]

$$\Theta(\omega_0)=\frac{\omega_0}{\pi}\int_0^{\infty}\frac{\ln[R(\omega)/R(\omega_0)]}{\omega_0^2-\omega^2}d\omega \ . \tag{1}$$

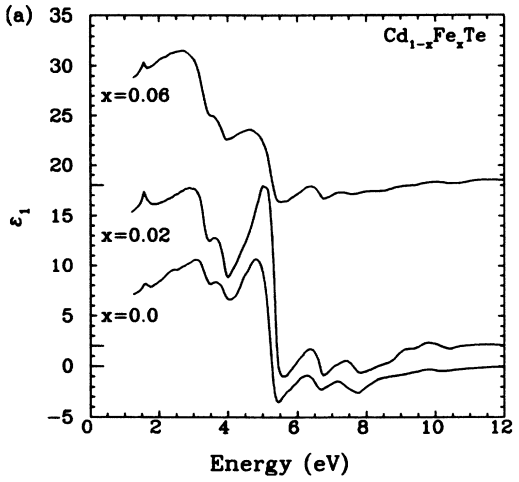
Since the spectra have been measured within a restricted energy region, the remainder of the $R(\omega)$ function must be extrapolated in order to perform the integration in Eq. (1). In the low-energy region, the reflectivity was assumed to vary as $a\omega^2+b\omega+c$ down to the minimum of the polynomial, and then taken to be constant. The parameters of the polynomial must fulfill two equations describing the continuity of the reflectivity curve and of its derivative. a was chosen as the free parameter. In the high-energy region, the reflectivity was assumed to be given by[18]

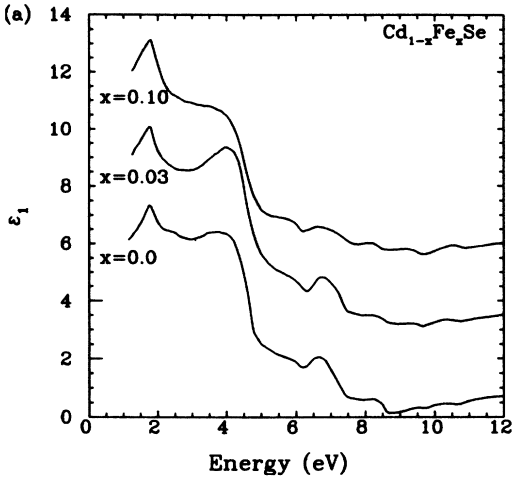
$$R(\omega)=\begin{cases} R_N(\omega_N/\omega)^s & \text{with } s>0, \text{ for } \omega_N\le\omega\le\omega_x \ , \quad (2a) \\ R_x(\omega_x/\omega)^4, & \text{for } \omega>\omega_x \ , \quad (2b) \end{cases}$$

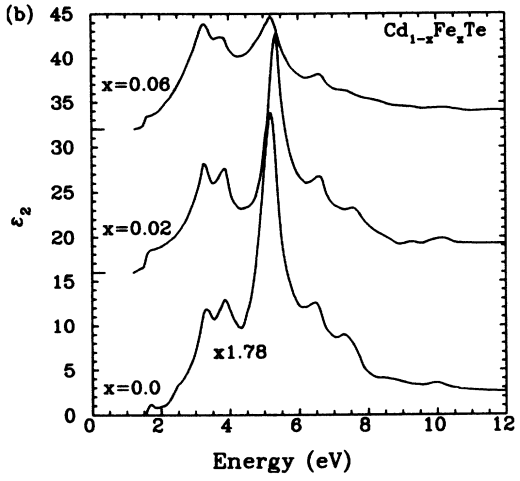
where R_N and ω_N are the reflectivity coefficient and the frequency at the end of the measured spectrum, respectively. R_x, ω_x, and s are free parameters. One of them (R_x) was removed with the help of the condition of continuity at ω_N. The integration was performed numerically for the low-energy and the experimentally investigated regions, and by means of a series expansion for the high-energy region. The best set of free parameters was chosen by following a procedure which arises from the concept, proved to be true by Velický,[19] that an optical experiment in a limited range is complete if it gives two independent optical constants. In practice, this means that a limited reflectivity spectrum and some other optical data (e.g., the absorption coefficient) for some part of

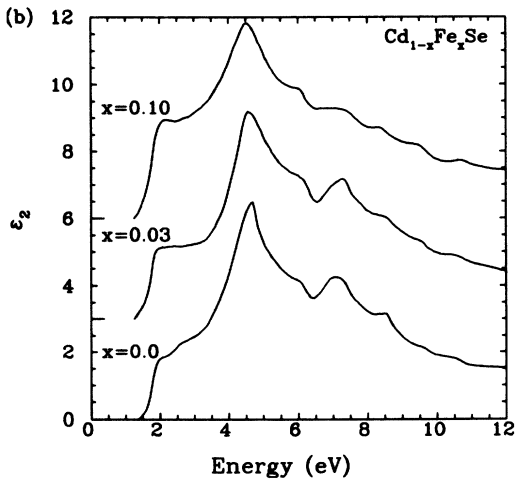
TABLE II. The energies of the peaks and shoulders discerned in the spectra of $Cd_{1-x}Fe_xSe$, together with their assignment to interband transitions.

Energies of the peaks						
Reflectivity Fe content			Imε Fe content			
0.0	0.03	0.1	0.0	0.03	0.1	Assignment
1.80	1.80	1.80	1.60	1.60	1.60	(for Imε: position of the edge) (Γ_6,Γ_1) -Γ_1 (Ref.17)
2.50			2.60	2.45	2.70	Γ_5-Γ_1
4.70	4.55	4.45	4.70	4.55	4.50	U_4-U_3,U_3-U_3-, Γ_5-Γ_3 (Ref. 17)
6.10	6.05	6.10	6.05	6.15	6.05	H_3-H_3 (and M_2-M_1?) (Ref. 17)
7.40	7.35	7.35	7.10	7.25	7.25	K_2-K_2,K_3-K_2 (Ref. 17)
8.60	8.55	8.40	8.50	8.55	8.40	Γ_1-Γ_1,Γ_5-Γ_6,Δ, L (Ref. 17)
9.70	9.70	9.50	9.70	9.60	9.45	region of M and the B face (Ref. 17)
10.60	10.55	10.65	10.60	10.50	10.65	VB-CB









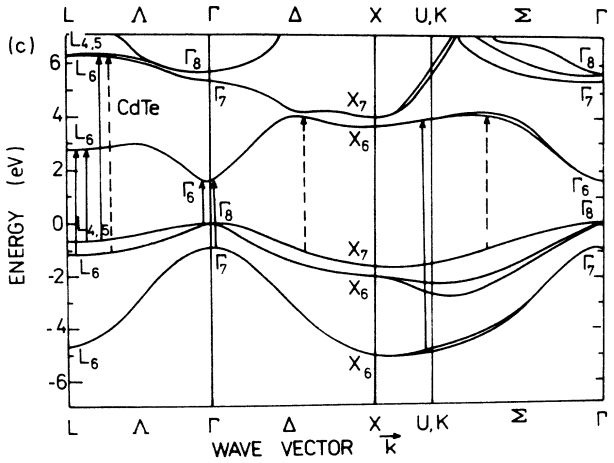


FIG. 2. The (a) $\epsilon_1(h\nu)$ and (b) $\epsilon_2(h\nu)$ curves for $Cd_{1-x}Fe_xTe$ obtained by means of a Kramers-Kronig analysis of the reflectivity spectra shown in Fig. 1(a). (c) The optical transitions observed for $Cd_{1-x}Fe_xTe$, drawn together with the calculated band structure of CdTe (Ref. 16). The dashed arrows correspond to the transitions that decrease in intensity with increasing Fe content, while the solid arrows correspond to the transitions with constant intensities.

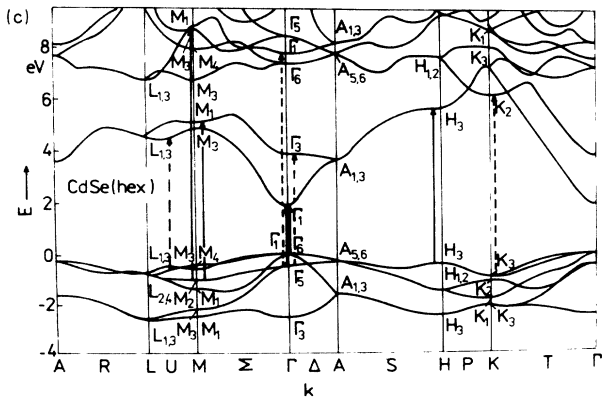


FIG. 3. The (a) $\epsilon_1(h\nu)$ and (b) $\epsilon_2(h\nu)$ curves for $Cd_{1-x}Fe_xSe$ obtained by means of a Kramers-Kronig analysis of the reflectivity spectra shown in Fig. 1(b). (c) The optical transitions observed for $Cd_{1-x}Fe_xSe$, drawn together with the calculated band structure of CdSe (Ref. 17). The dashed arrows correspond to the transitions that decrease in intensity with increasing Fe content, the solid arrows correspond to the transitions with constant intensities, and the thick solid arrow correspond to the transitions that increase in intensity.

the range in which the reflectivity is known, are enough for determining all the optical coefficients for the experimentally available photon energies. Equation (1) is applied, for given values of the three fitting parameters, to an analysis of the reflectivity spectrum, giving the phase angle of reflection as a function of the energy of light. For every point where R and Θ are known, other pairs of optical constants can be calculated [(n,k) and then (ϵ_1,ϵ_2)]. The absorption coefficient α can be easily obtained from the extinction coefficient k. A comparison of the calculated and measured values of this constant enables us to check the correctness of the assumed values of the extrapolation parameters. These may be modified until the correspondence is satisfactory.

The results obtained in this way are shown in Figs. 2 and 3.

IV. DISCUSSION

Since the character of the $\epsilon(h\nu)$ function is directly connected with the joint-density-of-states distribution, the maxima of $\epsilon_2(h\nu)$ [shown in Figs. 2(b) and 3(b)] can be ascribed to Van Hove singularities.[20] The energy positions and heights of the peaks correspond to the energies of optical excitations and their strengths, respectively. Thus, the changes in the shapes of the spectra caused by an increase of the Fe content reveal modifications of $E(\mathbf{k})$.

A. $Cd_{1-x}Fe_xTe$

The curves shown in Fig. 2(b) begin with sharp edges, corresponding to the fundamental gap, then ϵ_2 increases up to two distinct maxima, assigned to transitions at point L (3.30 and 3.85 eV). The next (highest) peak (5.20 eV) is ascribed to excitations at several points in the volume of the Brillouin zone (according to the reflectivity structure at 5.35 eV) (see Table I). In the high-energy parts of the spectra, several relatively weak maxima are discernible. Their physical origins are similar to those ascribed to the corresponding reflectivity features (Table I).

A comparison of the ϵ_2 curves obtained for crystals with different Fe contents enabled us to observe changes of positions and heights of the peaks. Table I shows that a shift of 0.15 eV occurs for the maxima at 6.45 and 10.0 eV (for CdTe), but it is close to the limit of accuracy of determination of the energy positions. The broad feature at 8.75 seems to split into two components, at 8.45 and 9.25 eV (for $Cd_{0.98}Fe_{0.02}Te$). The remaining maxima do not show any clear changes of position (within the limits of accuracy of determination of their energy positions). The changes in peak heights are much clearer. In order to obtain a consistent picture of the modifications, the spectrum of CdTe had to be scaled up to the other curves. It was multiplied by 1.78, so as to obtain the same height of the peak at 3.30 eV for all the crystals. The relatively low value of ϵ_2 for the CdTe sample is consistent with the lower sample reflectivity [Fig. 1(a)], probably caused by poorer quality of the crystal. Figure 2(b) shows that the maxima at 3.30, 3.85, and 6.45 eV are weakly influenced by changes in the Fe content. The apparent decrease and shift of the second and third of these (-0.05 and 0.15 eV, respectively) can be explained by a strong decrease of the pronounced peak at 5.20 eV. For energies higher than 7 eV, all the features are almost smeared out for $Cd_{0.94}Fe_{0.06}Te$. It is clear from the assignment of the peaks [listed in Table I and shown in Fig. 2(c)], that the features insensitive to the Fe content are ascribed to transitions at (or close to) the high-symmetry points in the Brillouin zone (BZ). In particular, the insensitivity of the edge of the spectrum to the presence of Fe suggest that the bands near the gap at point Γ are quite weakly influenced by the Fe-derived states that are expected to occur in both in the valence and conduction bands. In contrast, an increase of x strongly influences the peak observed at 5.20 eV, caused by the transitions at several points in the volume of the Brillouin zone, as well as the other excitations ascribed to regions away from the high-symmetry points. The maxima at 6.45 and 7.30 eV have been ascribed to the L point,[16] or to two different low-symmetry points.[12] The observed dissimilarity of their behavior seems to testify to their different physical origins, or to selective interaction of the Fe-derived states with the nearly degenerate states. Since the transitions at 3.30 and 3.85 eV (starting from the same bands at point L) are insensitive to an increase of the Fe content, the Fe states are expected to influence mainly the conduction band structure. An analogy with the $Cd_{1-x}Mn_xTe$ crystals[23] leads us to suggest that the empty Fe $3d$ states may occur somewhere between the first and second conduction bands, and modify strongly the structure of the final states involved in some of the optical transitions observed. A competitive process, influencing the structure of the conduction bands is replacement of the Cd $5s$ states by Fe $4s$, but this seems to be less important for the crystals with low Fe contents.

The behavior of the spectra described above can also be connected with the symmetry properties of the band structure. At points of low symmetry, Van Hove singularities, connected with the particular shape of the crystal potential, and not determined by the point group of the crystal, can be easily removed by the absence of any long-range order. Similar behavior was observed by Kendelewicz for $Cd_{1-x}Mn_xTe$.[6] On the other hand, at the high-symmetry points, the sharp bands may become somewhat broader as a result of disorder, but the high density of states (occurring due to the condition $\nabla_{\mathbf{k}}\varepsilon_v=\nabla_{\mathbf{k}}\varepsilon_c=0$) does not disappear, and strong optical transitions may still take place. Thus, we can observe that the presence of Fe ions influences the optical properties of $Cd_{1-x}Fe_xTe$ mainly by means of the disorder occurring in the lattice. No particular feature has been discerned that may be connected with the appearance of new maxima of the Fe-derived density of states.

B. $Cd_{1-x}Fe_xSe$

An analysis of the spectra of $Cd_{1-x}Fe_xSe$ is more complicated, because every feature is a superposition of the peaks and shoulders occurring for different light polarizations. A discussion of the influence of the Fe content on the reflectivity spectra obtained for perpendicular and parallel polarizations has recently been given by Kisiel

et al.[21] Unfortunately, the corresponding $\epsilon_2(h\nu)$ curves are not available.

The spectra obtained by us [shown in Fig. 3(b)] can still be interpreted on the basis of the published results of band-structure calculations.[17] The edge at 1.6 eV and the weak structure at 2.60 eV correspond to transitions at point Γ (the edge is connected with the fundamental gap). The highest maximum (at 4.70 eV) and each of the weaker peaks and shoulders observed for higher energies, are connected with excitations at several points in the Brillouin zone (Table II). Due to the introduction of Fe ions into the crystal, the peaks at 4.70 and at 9.70 eV (for CdSe) shift towards lower energies (0.20 and 0.25 eV, respectively), while the maximum at 7.10 eV shifts towards higher energies (0.15 eV). The heights of the peaks at 4.70, 7.10, and 8.50 eV decrease, while the edge corresponding to the fundamental gap increases markedly. Figure 3(c) shows all these transitions, drawn against the background of the band structure of CdSe.[17] Their dependence on the Fe content is, in principle, consistent with the conclusions of Kisiel[21] that Fe ions influence the band structure more strongly at point Γ and the high-symmetry directions, than in other parts of the volume or on the edges of the Brillouin zone (due to the fact that only near point Γ is the valence-band structure constructed predominantly from *p*-like anion orbitals). Such behavior indicates a stronger interaction between the Fe 3*d* and Se 4*p* orbitals than between Fe 3*d* and Cd 5*p*. The influence of Fe on the transitions at point Γ manifests itself in various ways. The height of a structure increases $[(\Gamma_6,\Gamma_1)\text{-}\Gamma_1]$ or decreases $(\Gamma_5\text{-}\Gamma_3, \Gamma_1\text{-}\Gamma_1, \Gamma_5\text{-}\Gamma_6)$, and some shifts in different directions with respect to the energy scale can be seen (at least for the reflectivity spectra). Since the initial states of these transitions are markedly concentrated both in **k** space and in energy, we ascribe the observed changes of the spectral features mainly to different influence of the Fe states on the separated conduction bands (final states of the transitions). Two phenomena which occur in the alloys may be responsible for this: (i) Fe 4*s* states replace some of the Cd 5*s* states, and contribute to conduction band formation, and (ii) empty Fe 3*d* states appear at energies corresponding to the empty (conduction) bands of the alloy. The result of the competition between these two processes determines the change of the conduction band structure, and influences the energies and intensities of the optical excitations observed. Nevertheless, the increase of the edge at 1.6 eV may be connected with the appearance of additional states at the edge of the valence band, or with some change of the matrix elements governing the optical transitions. This is consistent with results of a photoemission study.[22] The transitions ascribed to points at the BZ borders do not testify to any changes of their initial and/or final states, with the sole exception being the excitations to the K_2 states. These states lie at the energy of 6 eV, at which forbidden gaps are observed for other high-symmetry points and lines. This is an argument for locating the empty Fe 3*d* states between the first and second conduction bands, at about 6 eV. This conclusion is similar to that drawn for $Cd_{1-x}Fe_xTe$ in the preceding section.

V. SUMMARY

The foregoing discussion of the optical properties of $Cd_{1-x}Fe_xTe$ and $Cd_{1-x}Fe_xSe$ shows that in spite of their close similarity to the corresponding properties of the parent II-VI materials, the presence of Fe ions in these crystals causes both parallel and dissimilar changes in the two materials in question.

An analysis of the ϵ_2 spectra of $Cd_{1-x}Fe_xTe$ enables us to state that at points where Van Hove singularities are caused by the symmetry conditions (high-symmetry points), the band structure is weakly influenced by the presence of the Fe ions, in contrast with the regions where singularities occur by chance (due to the particular shape of the crystal potential). The presence of the Fe 3*d* states, and the disorder arising in the alloy, lead to changes in the energies of optical transitions, to a removal of some degeneracy, and, accordingly, to changes in the shapes of the spectra. Some indirect arguments enable us to locate the Fe 3*d* empty states between the first and second conduction bands of CdTe.

For $Cd_{1-x}Fe_xSe$, marked influence of the Fe-derived states on the band structure in the center of the Brillouin zone was revealed, in contrast with the situation for the states at the edges of the zone. In particular, the increase of the edge of $\epsilon_2(h\nu)$ shows that the presence of Fe increases the strength of the transitions at point Γ by means of a contribution to the density of states, or by an increase of the matrix element. Besides, as for $Cd_{1-x}Fe_xTe$, the presence of the Fe 3*d* empty states manifests itself in the range of energies corresponding to the lowest and second lowest conduction bands.

The above-listed noticeable differences between the optical properties of $Cd_{1-x}Fe_xTe$ and $Cd_{1-x}Fe_xSe$, show that a precise picture of Fe-based SMSC's cannot be drawn up regardless of the details of the iron-anion interaction, which is of great importance for the valence-band structure. Band-structure calculations that take into account the presence of a partially occupied Fe 3*d* shell, as well as the alloying effects, would be a useful tool for a further comprehensive analysis of the optical properties of Fe-based semimagnetic semiconductors.

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[23]Kisiel *et al.* (Ref. 7) suggest that some of the changes observed in reflectivity spectra of $Cd_{1-x}Mn_xTe$ with respect to those of CdTe, are caused by the presence of Mn 3*d* empty states between the lowest and second lowest conduction bands. This removes the near degeneracy of the states lying at 3–4 eV above the valence-band maximum (the final states of the transitions that are responsible for the peak at 5.2 eV), and pushes up or down the states that are more separated in energy, in particular, at point *L*.