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Te and Cd nuclear-magnetic-resonance study of local structure and bonding in $Cd_{1-x}Zn_xTe$

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Magic-angle sample-spinning NMR spectra of ^{111}Cd and ^{125}Te in $Cd_{1-x}Zn_xTe$ semiconductor alloys have been recorded for $0 \leq x \leq 1$. The ^{111}Cd NMR spectra exhibit a single line whose chemical shift and linewidth depend on x. The linear dependence of the ^{111}Cd chemical shift on x is assumed to be the result of charge transfer, while the linewidth dependence is shown to be the result of a random distribution of Zn atoms which are next-nearest neighbors to Cd. The ^{125}Te spectra exhibit multiple lines, their number and chemical shift depending on x. The different chemical shifts of the Te lines are assigned to the five different cation configurations—Cd_nZn_{4-n}, $n=0,\ldots,4$—around the Te. The intensities of the experimental Te lines are compared to those in spectral simulations obtained by assuming a random distribution of the cations. The comparison reveals small deviations from a random distribution of the cations. This clustering phenomenon and the effect of the bond-length changes with x on the ^{125}Te chemical shift are discussed.

I. INTRODUCTION

$Cd_{1-x}Zn_xTe$ belongs to a group of semiconductor alloys of the form $A_{1-x}B_xC$, which are of considerable technological and scientific interest. In order to understand the properties of these materials, much effort has been devoted to calculating their electronic structure and bonding properties,[1] especially their dependence on composition. However, theoretical predictions of these properties are more difficult than usual because of the disordered nature of these systems. For example, in $Cd_{1-x}Zn_xTe$, the Te anions are surrounded in the lattice by different numbers of cations, Cd and Zn, the exact configuration depending on the alloy composition x. It is this disorder which leads to the difficulties alluded to above. The discovery of a bimodal distribution of nearest-neighbor bond lengths in $Ga_{1-x}In_xAs$ by extended x-ray-absorption fine structure (EXAFS) (Ref. 2) has proved that the *local* features in these alloy systems play a decisive role in determining their properties. It is therefore clear that any new experimental technique that can yield information on the local structure and bonding of these materials will add to our understanding of their properties.

NMR is one of a limited number of approaches that can be used to study local properties of solids. The information that can be derived from NMR spectra is due to the sensitivity of nuclear-resonance frequencies to electronic structure. There are several forms of this interaction (Knight shift, chemical shift, and quadrupole coupling) but in the present study we focus primarily on the chemical shift. This parameter is easily observed in the spectra of $I=\frac{1}{2}$ nuclei (Cd, Te) and is sensitive to the electronic distribution near the nuclear spin. It therefore provides information on structure and bonding properties. A considerable body of information has been accumulated on chemical shifts and, although it is not yet possible to perform *ab initio* calculations of these parameters for any but the simplest molecules, many of the factors affecting chemical shifts are well known and predictions for new systems are often possible.

In the present work we study *changes* in Cd and Te chemical shifts as a function of the composition of the alloy. Willig, Sapoval, Leiber, and Verie[3] were the first to employ this approach in studies of $Hg_{1-x}Cd_xTe$. However, due to unfavorable experimental conditions, these workers were not able to detect changes in Cd and Te shifts and instead concerned themselves exclusively with the behavior of the much larger variations present in the spectra of Hg. Nevertheless, from such data they were able to reach several interesting conclusions about the bonding properties of $Hg_{1-x}Cd_xTe$. Willig *et al.*[3] also calculated the Te chemical shift for several binary II-VI compounds, and showed that the main contribution to both the cation and the anion chemical shifts is the so-called paramagnetic term. This term in the chemical shift is the result of a partial dequenching of the orbital contribution, and it arises from p electrons in unfilled orbitals.

While the chemical-shift calculations for the binary compounds are in relatively good agreement with the experimental results,[3] the calculations for the alloys were much more difficult to perform. In particular, a detailed knowledge of the electronic structure of the alloy, including wave functions and energies of the excited states, is required. Because an *ab initio* calculation of chemical shift was not possible, Willig *et al.*[3] employed the average-energy approximation proposed by Jameson and Gutowsky.[4] For tetrahedral symmetry the paramagnetic chemical shift is

$$\sigma_p = \frac{C}{\Delta}\langle r_p^{-3}\rangle N_p(1-N_p/6) . \tag{1}$$

$\langle r_p^{-3}\rangle$ is the inverse cube of the p-electron radius, N_p is the number of valence p electrons per atom, and Δ is an average excitation energy between occupied and empty states. N_p can vary for two reasons: (a) charge transfer and (b) distortion of the local symmetry. Willig *et al.*[3] concluded that the large change in Hg chemical shift with Cd alloying is due to a change in local symmetry around the Hg which drives a more *p*-type electron around Hg atoms. In the $Cd_{1-x}Zn_xTe$ system there is a relatively large difference between the Cd—Te and Zn—Te bond lengths, causing a distortion of the cubic symmetry at the Te site. We expect this distortion to affect the chemical shift.

The failure by Willig *et al.*[3] to detect changes in Cd and Te shifts was due in large part to the fact that chemical shifts in solids are masked by dipolar interactions. In the present work we circumvent this problem by employing magic-angle sample spinning (MASS), which averages all anisotropic interactions describable as second-rank tensors, including the dipolar, first-order quadrupolar, and chemical shift. The improved resolution in the spectra enables us to detect small changes in ^{125}Te and ^{111}Cd chemical shifts, and thus to examine changes of structure and bonding with alloy composition.

Of special interest in the semiconductor alloy systems is the phenomena of cation clustering. Clustering is defined in this case as a deviation from a random distribution of the two cations in their lattice sites. Several authors have predicted[5] clustering and claimed that it will be an important factor in understanding the properties of semiconductor alloys. The MASS NMR technique can, in principle, be employed to detect clustering, since the chemical shift of a particular Te is determined by the number of Cd and Zn nearest neighbors, and the intensity of a spectral line is directly related to that number. The MASS NMR line shapes therefore directly provide information on clustering. This feature of MASS NMR spectra has recently been employed in investigations of $Hg_{1-x}Cd_xTe$,[6] and here we study $Cd_{1-x}Zn_xTe$. In particular, we present results of ^{111}Cd and ^{125}Te NMR spectra and a model for explaining the chemical shifts and spectral line shapes. Conclusions with regard to clustering and charge transfer will also be discussed.

II. EXPERIMENTAL PROCEDURE

A. Sample preparation

The samples used in this study were in the form of fine powders, obtained by grinding pieces of single crystals grown by the vertical Bridgman technique. Prior to the synthesis of the alloys, pure elements—Cd, Te, and Zn (99.9999%)—were subjected to vacuum sublimation. Weighed amounts of the three elements for each $Cd_{1-x}Zn_xTe$ sample were sealed under vacuum ($\sim 10^{-6}$ Torr) in a 16-mm-i.d., graphitized quartz ampoule with a tapered end. The ampoule was placed in the Bridgman furnace at 500 °C and the reaction temperature was slowly raised to 1120 °C, where it was held for 24 h to homogenize the melt. Next, the ampoule was lowered through the furnace at the rate of 1.5 mm/h, the solidification gradient in the furnace being 15 °C cm^{-1}. To diminish radial heterogeneity the sample was rotated at the rate of about 2 rpm. Ingots containing crystals with no more than two grains were selected and ground to a fine powder. The concentration of Zn and Cd was determined by atomic absorption spectroscopy, and in most of the samples the cation concentration was very close to the intended value. In the few cases where large deviations were found, the samples were discarded. As we will see below, the ^{111}C resonance was also a good indicator of the cation concentrations.

B. NMR measurements

All spectra were taken at room temperature on a home-built Fourier-transform NMR spectrometer using a 9.3-T magnet, yielding ^{111}Cd and ^{125}Te frequencies of 84.348 and 125.35 MHz, respectively. ^{113}Cd NMR would give essentially the same result as ^{111}Cd. Sufficiently short 90° and 180° pulses (3 and 6 μsec) were used for Hahn echo sequences to effectively excite the large dispersion of the spectra. Recycle delays varying from 10 to 20 min were used to avoid spectral distortions due to different T_1's of the various resonance lines. We have found that doping with Mn^{2+} did shorten T_1 considerably, but at the same time the dopant induced subtle changes in the line shapes. The spectra in this initial study were therefore obtained from Mn^{2+}-free samples.

MASS NMR experiments were performed with rotors and stators obtained from Doty Scientific Company (Columbia, SC). The rotor volume is 360 μl and typical spinning speeds were 1.5–2.8 kHz. Since Te and Cd occupy nearly cubic sites in the alloys, the shift anisotropy exhibited by these species is small and therefore minimal rotational sidebands were apparent in the spectra. In addition, dipole-dipole couplings in these systems are attenuated because of the low abundance wt. % of ^{111}Cd (12%), ^{125}Te (7%), and ^{67}Zn (4%), and the low gyromagnetic ratios of the nuclei. Thus these spinning speeds were sufficient to attenuate the residual dipolar couplings. ^{125}Te and ^{111}Cd chemical shifts are reported relative to CdTe.

III. RESULTS AND DISCUSSION

We have performed MASS NMR measurements on both ^{111}Cd and ^{125}Te nuclei in $Cd_{1-x}Zn_xTe$ for a wide range of x. There is a dramatic difference between the spectra of Cd and Te, whose origin is primarily the nearest-neighbor (NN) environment of the two atoms. Cd occupies a lattice site which is always surrounded by four Te NN for all x, whereas Te sites are surrounded by two types of cations, Zn and Cd, the number of each varying between 0 and 4. Their distribution and therefore the chemical shifts depend on the alloy composition.

A. ^{111}Cd NMR

The ^{111}Cd NMR spectrum of the binary CdTe exhibits a narrow center resonance line of 150-Hz width flanked by two small lines due to J coupling between ^{111}Cd and ^{125}Te. With increasing Zn concentration the ^{111}Cd spectrum remains a single line but the chemical shift and width of the resonance change with x. This is illustrated in Fig. 1, where we present the relative chemical shift and width of ^{111}Cd line as a function of composition. As shown in the figure, the experimental ^{111}Cd chemical shift varies linearly with Zn concentration, while the linewidth reaches a maximum of 33.2 ppm at $x=0.5$ and decreases symmetrically about this concentration.

The chemical shift and linewidth behavior observed for ^{111}Cd can be explained by assuming the Zn atoms are distributed randomly among the 12 next-nearest-neighbor (NNN) sites of the Cd. The probability $P(n,x)$ of finding all possible combinations of $\mathrm{Cd}_n\mathrm{Zn}_{(12-n)}$ in the NNN shell of Cd for a given concentration $\mathrm{Cd}_{1-x}\mathrm{Zn}_x\mathrm{Te}$ can be calculated using the Bernoulli function:

$$P(n,x)=\begin{bmatrix}12\\ n\end{bmatrix} x^{12-n}(1-x)^n, \quad n=0,1,\ldots,12\,. \tag{2}$$

The result is plotted in Fig. 2, where for simplicity we have chosen x values for the calculations which correspond to samples employed in our experiments.

The maximum value of each curve is the most probable number of Zn atoms to be found among the 12 NNN sites to Cd for a given x, and is also linearly dependent on x. Combining this result with the experimentally observed chemical shift, we find there is an 8.2-ppm frequency shift of the ^{111}Cd resonance line for each neighboring Zn atom.

In addition, Fig. 2 also shows the width of the distribution function, reaching a maximum at $x=0.5$. This relationship has been calculated for many values of x and the result is also plotted in Fig. 1. Again, the agreement between experimental linewidth, shown by triangles, and a random distribution width—the solid line—is very good. Hence, we conclude that the ^{111}Cd MASS NMR spectral linewidths of $\mathrm{Cd}_{1-x}\mathrm{Zn}_x\mathrm{Te}$ are due to a distribution of isotropic chemical shifts arising from the random distribution of Zn atoms in the NNN shells. Unfortunately, these spectra are not resolved for each distribution of $\mathrm{Cd}_n\mathrm{Zn}_{(12-n)}$ NNN due to some or all of the following reasons.

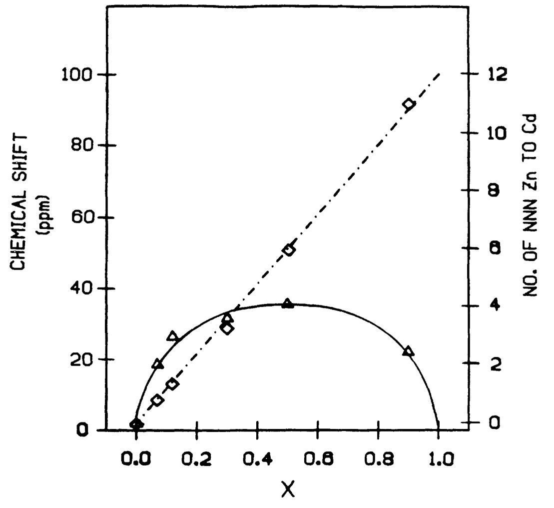

FIG. 1. ^{111}Cd NMR experimental relative chemical shifts (◇) and linewidths (△) of $\mathrm{Cd}_{1-x}\mathrm{Zn}_x\mathrm{Te}$ as a function of x. The dash-dotted straight line presents the most probable number of NNN Zn atoms, and the curved solid line is the width as calculated using the probability curves in Fig. 2. (See text for explanation.)

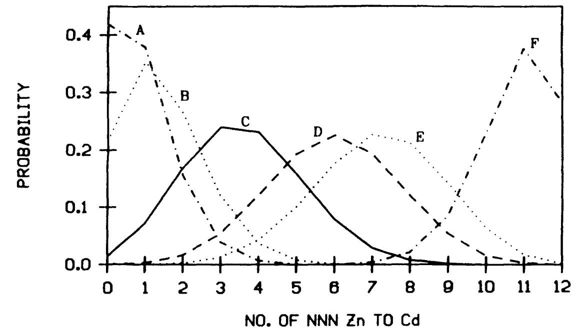

FIG. 2. Probability of number of NNN Zn to Cd assuming Zn-Cd random distribution for concentrations of $\mathrm{Cd}_{0.93}\mathrm{Zn}_{0.07}\mathrm{Te}$ (curve A), $\mathrm{Cd}_{0.88}\mathrm{Zn}_{0.12}\mathrm{Te}$ (curve B), $\mathrm{Cd}_{0.7}\mathrm{Zn}_{0.3}\mathrm{Te}$ (curve C), $\mathrm{Cd}_{0.5}\mathrm{Zn}_{0.5}\mathrm{Te}$ (curve D), $\mathrm{Cd}_{0.4}\mathrm{Zn}_{0.6}\mathrm{Te}$ (curve E), and $\mathrm{Cd}_{0.1}\mathrm{Zn}_{0.9}\mathrm{Te}$ (curve F).

(a) Apart from the $\mathrm{Cd}_n\mathrm{Zn}_{(12-n)}$ discussed above, each configuration, for example $\mathrm{Cd}_9\mathrm{Zn}_3$, will have 12×11×10 different permutations occupying the 12 NNN sites. Each of these permutations may yield slightly different Cd chemical shifts, thus inducing an additional line broadening.

(b) Fourth-nearest neighbors, which were ignored in our calculation, may affect the linewidth, although we expect this effect to be small.

(c) J coupling of ^{111}Cd to ^{125}Te, which was found to be 0.7 kHz, will also contribute to the linewidths.

In general, changes in chemical shift due to the introduction of new atoms in a compound can be the result of charge redistribution around the atom of interest. In our case the ^{111}Cd resonance line is shifted to a higher frequency as a result of Zn alloying, a deshielding effect. This can be a result of either the addition of p electron character or a reduction of s electron character. Based on simple ionic considerations we expect that there is an inductive charge transfer via Te, causing a net charge of s type to transfer from Cd to Zn. Similar behavior has been observed in ^{29}Si NMR of silicates where the Si resonance line was shifted to higher frequency with the addition of more electronegative atoms in NNN positions.[7]

B. ^{125}Te NMR

Figure 3 presents the ^{125}Te NMR spectra for several different compositions, the solid lines being the experimental spectra, while the circles result from simulations to be explained below. For reference we show in the same figure the Te spectra of the binaries, CdTe and ZnTe (bottom trace). For all compositions, besides $x=0.5$, the spectra consist of two or more lines and the shape changes with composition.

In order to explain these results we first examine the NN environment of Te. Te is surrounded by four cations consisting of Zn and Cd, the five possible configurations being Cd_nZn_{4-n}, $n=0,\ldots,4$. In addition, the Cd—Te and Zn—Te bonds have different lengths and ionicity. Since the Te chemical shift is sensitive to its local environment, each cation configuration will result in a different chemical shift for its associated Te. Thus each of the lines in the spectra corresponds to a different cation configuration. The reason for the improved resolution observed at low and high x is the same as we found for the ^{111}Cd linewidth. In particular, the ^{125}Te shift is determined primarily by its first NN cation distribution, and each resonance line is broadened by interactions with third NN atoms. As was found in Figs. 1 and 2 for ^{111}Cd, the distribution function and spectral width reach a maximum for $x=0.5$ and are quite broad for $0.15<x<0.85$. Thus in this composition region the ^{125}Te linewidths are large and therefore the resolution of the spectra is degraded. Nevertheless, the resonance-line positions and the line shapes observed in the Te spectra can be understood by combining the following arguments.

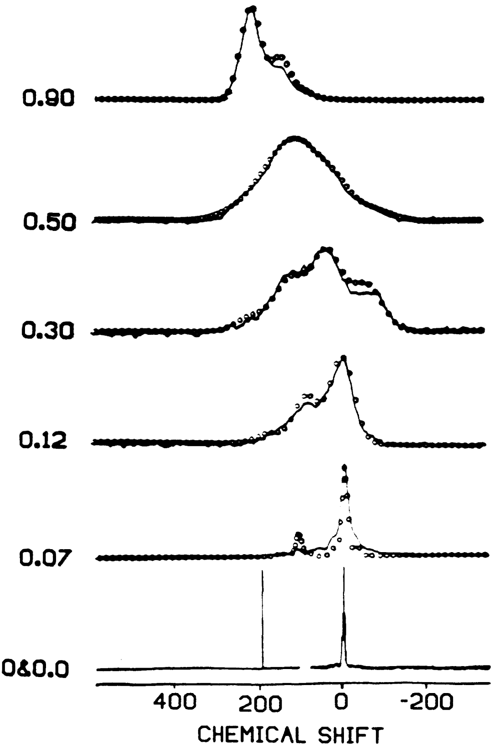

FIG. 3. ^{125}Te NMR spectra of various $Cd_{1-x}Zn_xTe$ alloys with indicated x values. The bottom spectra are of ZnTe at 200 ppm and CdTe at 0 ppm (experimental reference). The superimposed circles are calculated points assuming a random distribution of the cations. The lines were assigned using method described in the text.

First a comparison of the spectra of ZnTe and CdTe and the Te spectra of CdZnTe at low and high Zn concentrations clearly show that the chemical shift of the new lines increases with the addition of Zn. Therefore, the larger the number of Zn atoms in a configuration (the smaller n), the higher the shift of the associated ^{125}Te NMR line. In Fig. 4 we plot the relative chemical shift for each of the above configurations as obtained from the experimental spectra, and the few points from the simulated spectra as described below. Assuming a random distribution of the cations, we used the Bernouli function

$$P(x,n)=\begin{bmatrix}4\\n\end{bmatrix}x^n(1-x)^{4-n},\quad n=1,\ldots,4 \qquad (3)$$

to calculate the relative intensities of each cation configuration for the various compositions used in this work. The intensities of the simulated spectra were scaled such that the intensities of the dominant lines in the simulated and experimental spectra coincide. The width of each resonance line was obtained using a distribution function for the sites in the third-nearest neighbor for each concentration x, similar to the procedure employed for the case of ^{111}Cd (Fig. 2). The assignments of the chemical shifts to the different configuration for cases where there were no resolved spectra were determined by iteratively adjusting the line position to fit the experimental Te spectra. This leads to the simulated line shapes (open circles) shown in Fig. 3.

Second, we find that the chemical shift for a given Cd_4Zn_{n-4} configuration also depends slightly on composition x. In order to understand this dependence we

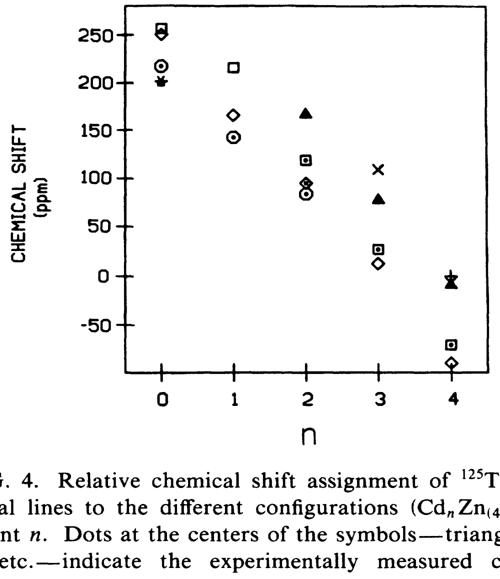

FIG. 4. Relative chemical shift assignment of ^{125}Te NMR spectral lines to the different configurations ($Cd_nZn_{(4-n)}$) for different n. Dots at the centers of the symbols—triangles, circles, etc.—indicate the experimentally measured chemical shifts while the remainder were extracted from best-fit simulations of the experimental line shapes. The alloys studied are +, CdTe; ×, $Cd_{0.93}Zn_{0.07}Te$; △, $Cd_{0.88}Zn_{0.12}Te$; □, $Cd_{0.7}Zn_{0.3}Te$; ◇, $Cd_{0.5}Zn_{0.5}Te$; ○, $Cd_{0.1}Zn_{0.9}Te$; *, $Cd_{0.01}Zn_{0.99}Te$; ●, ZnTe (at 200 ppm).

focus our discussion on the Cd_4Zn_0 and Cd_0Zn_4 configuration, which is the only case where the Te is in symmetric site. For each one of these configurations we find that the higher the Cd concentration of the sample, the higher the chemical shift of these configurations. These results can be correlated with changes in Cd—Te and Zn—Te bond lengths as a function of x, as measured by EXAFS.[8] When Zn is added to the alloy the Cd—Te bond length decreases and the Cd_4Te tetrahedron contracts. In contrast, the Zn—Te bond length increases with Cd concentration, and the Zn_4Te tetrahedron expands. Hence, we find that when the bond length increases, the ^{125}Te resonance line moves to a higher chemical shift. A similar correlation has been observed for ^{29}Si, where the chemical shifts of a variety of zeolites were found to be linearly dependent on the Si—O bond length as determined by x-ray diffraction. The dependence of chemical shift on bond length can be rationalized as follows: It was assumed by Willig *et al.*[3] that the overall number of electrons around the Te atom is nearly constant with the alloy composition. However, an increase in bond length decreases the charge density around Te, causing a decrease in shielding and thus an increase in chemical shift.

In a recent NMR study of $Hg_{1-x}Cd_xTe$[6] it was found that the Cd_4Hg_0 resonance line is shifted to higher frequency with increasing Hg concentration. If the correlation between bond length and chemical shift is correct, then this behavior implies that the Cd—Te bond length increases with Hg concentration in agreement with theoretical calculations.[9] Thus it appears that in some cases chemical shift measurements can provide information on bond-length changes.

We can also employ the ^{125}Te spectra to determine if there is a deviation from random distribution of the cation configurations—i.e., do we have cation clustering in the $Cd_{1-x}Zn_xTe$ systems? In general, there is good agreement between the simulated and the experimental line shapes, indicating relatively small deviations from a random distribution. Nevertheless, there are consistently small deviations which we believe are beyond experimental error which indicate the possibility of clustering. For example, for $x=0.07$ we see that in the experimental spectra the higher-frequency resonance line assigned to the Cd_3Zn_1 configuration is less intense than in the simulation. This suggests that while the Cd_4Zn_0 configuration is very stable, the Cd_3Zn_1 is not. The Zn atoms missing from the unfavorable Cd_3Zn_1 configuration are probably distributed among other configurations such as Cd_2Zn_2 and Cd_1Zn_3. As these are "Zn-rich" configurations, only a small number of them is required to "consume" the missing Zn atoms, and therefore, the resonance lines of these configurations are too small to be observed in the spectra. For $x=0.12$ the Cd_3Zn_1 resonance intensity is also smaller than the simulated intensity. For $x=0.30$ the Cd_4Zn_0 component is less intense than the simulation; this indicates that for this composition Cd_4Zn_0 is no longer the most favorable configuration. Finally, for $x=0.90$ the Cd_1Zn_3 configuration is less favorable compared to Cd_0Zn_4. Nevertheless, the spectra indicate that the most probable configuration is certainly the most favorable and the amount of clustering is small.

We should remark here that the deviation from the random distribution found for the cations using the Te spectra does not affect the Cd spectra for the following reason: In the case of Te the spectra are affected by the distribution among the four NN sites, while for Cd the same clustering is divided among 12 NNN sites, thus diminishing the clustering effect. As shown in the present work, the Te spectra clearly demonstrate that the Te atom in the alloy can be in five different sites, each with a different charge distribution. This means that there are five different potentials randomly distributed, causing disorder among the anion sites. This observation supports the recent suggestion[10] that in a common anion semiconductor alloy, besides the cation disorder due to the cations randomly distributed, there is also anion disorder. It was suggested that this anion disorder could account for the low hole mobility observed in $Hg_{1-x}Cd_xTe$.[11]

Theoretical discussions of clustering in $A_{1-x}B_xC$ systems[5] suggest there are two competing effects: The enthalpy that includes chemical and strain interactions which tend to cause clustering, and the entropy term that derives the system towards randomness. An NMR study of materials prepared at lower temperature might show Te spectra which exhibit a larger deviation from random behavior.

IV. CONCLUSIONS

From the MASS NMR spectra of ^{111}Cd and ^{125}Te in $Cd_{1-x}Zn_xTe$ we arrive at the following conclusions.

(a) The ^{111}Cd chemical shift is linearly dependent on the number of Zn atoms in the NNN sites to Cd, and the change in ^{111}Cd chemical shift with x is due to charge transfer from Cd to Zn, probably of s-type electrons.

(b) The main contribution to the Te and Cd linewidths and their dependence on x is due to a distribution of isotropic chemical shifts resulting from a random distribution of Zn.

(c) The ^{125}Te spectral line shapes were found to depend strongly on the composition of the alloy and permitted a determination of the dependence of the Te chemical shift on NN cation configuration and bond length.

(d) Some clustering has been observed in the $Cd_{1-x}Zn_xTe$ system. In general we found that the most probable configuration appears to be the most stable.

The present work demonstrates the ability of the MASS NMR technique to provide valuable information on bonding and local structural properties in semiconductor alloys. The technique should be widely applicable to other systems where similar problems exist.

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