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^{93}Nb NMR study of the charge density wave state in $NbSe_2$

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Abstract

^{93}Nb NMR studies were carried out for a single crystal of $NbSe_2$ at 73.328 MHz in the temperature range 9–300 K to investigate the normal and charge density wave (CDW) states. Detailed analysis of the NMR line shape of the central transition using a classical incommensurate model reveals the change in the conduction electron spin dynamics from above T_{CDW}. An increase of the Knight shift below T_{CDW} reflects modification to the uniform part of the conduction electron density of states. As suggested theoretically, the Knight shift distribution is found to be directly proportional to the square of the amplitude of the CDW. The results further indicate an incommensurate CDW state in 2H-$NbSe_2$. Analysis of the NMR spectra using the McMillan incommensurate model suggests a large value of the discommensuration parameter (γ) which is almost temperature independent, in contrast to the much smaller value previously reported in the case of 2H-$TaSe_2$.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Various types of charge density waves (CDWs) have been observed in different layered transition metal dichalcogenides [1–3]. Among them 2H-$NbSe_2$, 2H-$TaSe_2$ and 2H-TaS_2 have attracted particular interest as they exhibit the coexistence of a CDW with superconductivity. Furthermore, in the case of these dichalcogenides the in-plane resistivity decreases below the CDW transition temperature (T_{CDW}) in contrast to the resistivity increase in other larger amplitude CDW materials [1]. Using x-ray scattering to measure the response of the incommensurate CDW modulation in 2H-$NbSe_2$ to applied magnetic fields up to 10 T, Du *et al* [4] observed no significant change in either the correlation length or the intensity of the CDW satellites. This finding suggests that the increased resistance observed in low-dimensional CDW materials exposed to applied magnetic fields does not result from an appreciable conversion of carriers from the normal to the CDW state.

Angle resolved photoemission (ARPES) studies [5] have suggested that the CDW instability in 2H-$NbSe_2$ is driven by Fermi surface nesting and not by saddle point singularities. However, this nesting mechanism is unable to explain the observed decrease in resistivity at the CDW transition. Recent high resolution ARPES studies [6] indicate that CDW instability in this family of materials originates from a combination of the partial nesting of the Fermi surface around K and the saddle band. However, this cannot also explain the observed CDW wavevector. Therefore it has emerged that in spite of all the various experimental investigations, the actual mechanism of the CDW transitions in these types of materials is not yet fully understood.

For 2H-$NbSe_2$, the difference between the transition temperatures T_{CDW} (33 K) and T_c (7.4 K) is smallest when compared to those of 2H-$TaSe_2$ and 2H-TaS_2. This indicates that competition between these two transitions is much stronger in the case of 2H-$NbSe_2$ compared to the other transition metal dichalcogenides. In 2H-$NbSe_2$, neutron scattering results [7, 8] indicated a second order

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transition with $T_{CDW} = 33$ K and a superlattice below T_{CDW}, which is incommensurate down to 5 K. Measurements of Young's modulus and internal friction suggest that the incommensurability parameter (δ) decreases continuously on cooling below T_{CDW}, but the material remains incommensurate down to 1.3 K [9]. On the other hand, in the case of 2H-$TaSe_2$ the system undergoes a transition from the normal to the incommensurate state at 122 K and remains in this state in the range 90–122 K. Below 90 K, it undergoes a transition to a commensurate CDW state.

The incommensurate state in 2H-$TaSe_2$ and 2H-$NbSe_2$ was described as a sum of three plane waves, each with a wavelength of almost, but not exactly equal to, three lattice spacings. If we consider this as the conventional or classical model for the incommensurate state, then there is another model, proposed by McMillan using Landau theory [10], where the incommensurate CDW can be better described as having regions which have the periodicity of the commensurate wave separated by phase slip regions or discommensurations. The difference in wavelength between the incommensurate and commensurate states is then related to the density of discommensurations. Direct visual evidence for such discommensurations was first reported from electron microscope images for 2H-$TaSe_2$ [11] and later from scanning tunneling microscopy of the $1T_2$ phase in 1T-$TaSe_2$ [12]. Previously Suits *et al* [13, 14] had used ^{77}Se NMR spectra in 2H-$TaSe_2$ to validate McMillan's hypothesis. On the other hand Berthier *et al* analyzed the ^{93}Nb NMR spectra from 2H-$NbSe_2$ below T_{CDW} by considering the spatially modulated conduction electron density as the basal superposition of triple conventional incommensurate CDW states with hexagonal symmetry [15, 16]. Later on Skirpov *et al* analyzed ^{93}Nb and ^{77}Se NMR spectra for 2H-$NbSe_2$ [17, 18] below T_{CDW} (31 K) as a superposition of three commensurate CDW states with orthorhombic symmetry. However, Eaglesham *et al* [19] presented electron microscope images where discommensuration structure can be seen in 2H-$NbSe_2$. They have interpreted these images, by analogy with 2H-$TaSe_2$, as being due to a double honeycomb array of discommensurations separating orthorhombic domains. The contrast between the fringes in 2H-$NbSe_2$ was much weaker than that observed in 2H-$TaSe_2$ by Fung *et al* [11]. Eaglesham *et al* explained this difference as being due to the broader discommensurations in 2H-$NbSe_2$. This finding creates enough interest to reinvestigate the nature of the CDW state in 2H-$NbSe_2$ using a microscopic tool such as NMR, in order to see whether McMillan's incommensurate state could give a better description of the CDW state.

Thus in the present work we have reinvestigated the CDW and normal state in 2H-$NbSe_2$ using the ^{93}Nb NMR line shape studies in aligned, high quality single crystals. The measurement was done at a much higher resonance frequency of 73.6 MHz than was used previously in order to obtain a greater spread in the spectrum due to the Knight shift distribution. The greater spread permits one to extract more accurate information about the nature of the CDW state. The resulting spectra are analyzed and discussed by considering (1) the conventional (classical) incommensurate CDW model and (2) McMillan's incommensurate model including discommensurations.

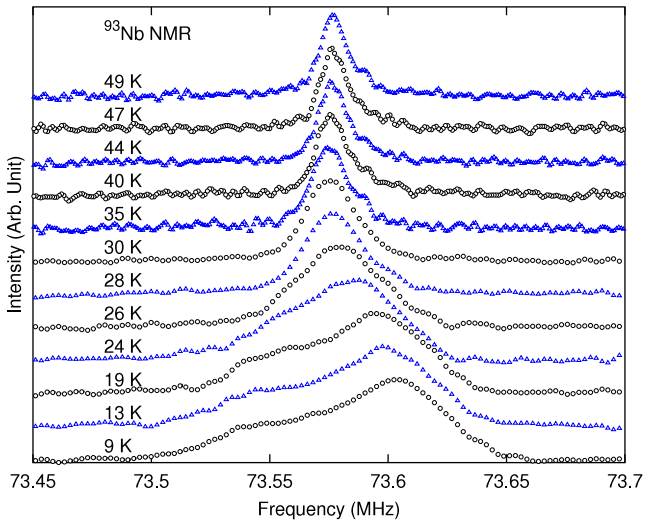

Figure 1. ^{93}Nb NMR spectra at some temperatures.

2. Experimental details

Single crystals of $NbSe_2$ were prepared from polycrystalline ingots (99.999% Nb and Se) by means of chemical vapor transport with an iodine carrier (0.75 atm) using mica tubes of length 25 cm under a thermal gradient 1000–900 °C. Magnetization and resistivity measurements revealed a sharp superconducting transition at $T_{SN} = 7.2$ K with a width not larger than 20 mK. Observation of quantum oscillations in the magnetostriction at temperatures as high as $T = 4.2$ and 8 K [20] confirmed the sample's extraordinary quality. Also the electrical resistivity ratio from ambient temperature to 10 K was higher than 30 ($\rho_{300\ K}/\rho_{10\ K} \geqslant 30$), typical of high quality $NbSe_2$ single crystals.

NMR studies were performed using a single crystal obtained from this batch, oriented with $c \| H_0$, at a frequency of 73.6 MHz in a Bruker MSL100 pulse spectrometer with a 7.04 T superconducting magnet. A home built NMR probe with a rf coil made of silver was used to avoid spurious ^{63,65}Cu signals. The spectrum was recorded by applying a $\pi/2$–τ–$\pi/2$ solid echo sequence. The length of the $\pi/2$ pulse was 2 μs and $\tau = 40$ μs. Temperature variation was performed in an Oxford continuous flow He cryostat with an ITC503 controller.

3. Results and discussion

The ^{93}Nb central transition was recorded at different temperatures for a single crystal of 2H-$NbSe_2$ oriented with $c \parallel H_0$. In this orientation the central transition ($1/2 \rightarrow -1/2$) is not affected by the quadrupolar interaction. Therefore one can derive the local properties simply from the distribution of the Knight shift, independently from the shape of the central transition. First the crystal was cooled slowly from 300 to 9 K and then the spectra were recorded at different temperatures during the heating cycle. Figure 1 shows some typical spectra

in the range 9–50 K. From the present experiment it is seen that there is substantial difference in the spectral features compared to those reported previously [15, 17]. For example, the overall line width remains unchanged in the range 75–300 K.

Below 75 K, though still in the 'normal' state, a gradual increase in the width of the resonance line is observed. Finally, by 30 K, there is a considerable increase in the overall width of the resonance line. The spectrum broadens further at lower temperatures to resemble a double peak structure below 24 K. The separation between the two peaks gradually increases down to 9 K. Previous authors [16] did not report any pretransitional broadening of the $(1/2 \rightarrow -1/2)$ transition. They did, however, observe this effect on the satellite transition $(-1/2 \rightarrow -3/2)$, which they attributed to the presence of impurities. Moreover, note the width (δH) of the central transition obtained in the present crystal at 300 K is 7 G (7 kHz) which is considerably less than that reported previously ($\delta H \sim$ 10 G). This suggests that the purity of the crystal in the present case is higher.

3.1. Classical incommensurate model

Below the CDW transition the conduction electron density is spatially modulated, which in turn produces a local modulation of the Knight shift $K(\mathbf{R})$. Assuming a linear and a local relationship [16, 17] between the CDW amplitude and the change in the resonance frequency ν at a nuclear site $\mathbf{R}_i$, one can write

$$\nu = \nu_{\text{ref}}[1 + K_{\text{iso}} + 2K_{\text{ax}}] + \nu_1 \Sigma_j[\cos(\mathbf{q}_j \cdot \mathbf{R}_i + \phi_j)]. \quad (1)$$

This can also be written as

$$\nu = \nu_{\text{ref}}[1 + K_{\parallel}] + \nu_1 \Sigma_j[\cos(\mathbf{q}_j \cdot \mathbf{R}_i + \phi_j)]. \quad (2)$$

Hence

$$\nu = \nu_0 + \nu_1 \Sigma_j[\cos(\mathbf{q}_j \cdot \mathbf{R}_i + \phi_j)] \quad (3)$$

where ν_0 is the NMR frequency corresponding to uniform electron density, $K_{\parallel}$ is the Knight shift in the normal state (i.e. for a uniform conduction electron density of states) for the orientation $c \| H_0$ of the crystal. ν_1 is the modulation amplitude of the conduction electron density of states which was assumed to be proportional to the CDW amplitude. $\mathbf{q}_{\text{j}}$ are the CDW wavevectors along the three equivalent directions of the parent Brillouin zone, and ϕ_{j} are the CDW phases. Here

$$\mathbf{q}_1 = \tfrac{1}{3}\mathbf{a}_1^{\star}(1 - \delta_1)$$

$$\mathbf{q}_2 = \tfrac{1}{3}\mathbf{a}_2^{\star}(1 - \delta_2) \qquad \mathbf{q}_3 = \tfrac{1}{3}\mathbf{a}_3^{\star}(1 - \delta_3)$$

which with $\delta_1 = \delta_2 = \delta_3 = \delta$ makes

$$\mathbf{q}_1 + \mathbf{q}_2 + \mathbf{q}_3 = 0. \quad (4)$$

Here $\mathbf{a}_{123}^{\star}$ are the reciprocal lattice vectors for a single layer and δ constitutes the (presumed hexagonal) incommensurability parameter of the charge density waves. The simulated spectra have been obtained from the theoretical equation

$$I(\nu) = \int g(\nu') \exp\left[-\frac{(\nu - \nu')^2}{2\beta^2}\right] \mathrm{d}\nu' \quad (5)$$

where $g(\nu)$ is the line shape function and ν is given by equation (2). Previously only the distribution function $g(\nu)\,\mathrm{d}\nu$ was calculated in case of 2H-$NbSe_2$. To obtain more accurate quantitative information we have calculated the total intensity pattern $I(\nu)$ to better fit the experimental spectra to theory.

We have analyzed the spectra using the conventional CDW model for three cases: (1) hexagonal symmetry of the incommensurate CDW, i.e. $\phi_1 = \phi_2 = \phi_3 = 2\pi/3$, with $\delta \neq 0$, as was done by Berthier *et al*; (2) orthorhombic symmetry of the commensurate CDW i.e. $\varphi_1 = 140°$, and $\varphi_2 = \varphi_3 = 2°$ with $\delta = 0$, as considered by Skirpov *et al*; and (3) when the effect of a non-zero value of δ is added in case (2). The experimental spectra were fitted by convoluting the line shape with a Gaussian shape function of width 2β. Such broadening models the contribution due to the nuclear–nuclear dipolar interaction and the electron–nuclear dipolar and hyperfine interactions. The former is temperature independent, as long as there is no mobility of the atoms whose nuclear resonance is observed. The latter contribution may be temperature dependent if the electron dynamics are affected by the change in temperature.

It was seen that the experimental spectra above 28 K could be fitted satisfactorily by considering just the first term of equation (2). From 28 K down, however, both the terms contribute to the resonance line shape.

Figure 2 shows some experimental spectra in the CDW state with superimposed theoretical spectra for both orthorhombic and hexagonal symmetry of the CDW. It is to be pointed out that a non-zero value of the incommensurability parameter (typically $\delta = 0.01$–0.015) proves essential for fitting the experimental spectra. However, the temperature dependence of δ, as observed in the neutron and x-ray studies, was not observed in the present NMR results. Furthermore, the disagreement between the theoretical and the experimental spectra is about the same for the hexagonal and orthorhombic models.

The behavior of the fitted parameters (the Knight shift $K_{\parallel}$, the CDW amplitude ν_1 and the intrinsic width 2β) is shown as a function of temperature in figures 3 (a)–(c) respectively. $K_{\parallel}$ (figure 3(a)) decreases continuously in the range 50–300 K and then remains almost unchanged in the range 30–50 K, before it finally increases again in the range 9–30 K. The increase of $K_{\parallel}$ below 30 K has not been reported previously. In fact Berthier *et al* gave the values of $K_{\parallel}$ at three temperatures in the range 77–273 K, showing a decreasing trend as we have found over the range 50–300 K. The subsequent increase in the contribution to $K_{\parallel}$ due to the uniform part of the conduction electron density of states below 30 K has not been reported before. A possible reason for the detection of this change could be the considerably higher resonance frequency being used in the present study. Although the magnitude of the increase is not very large, it is clearly seen by theoretically fitting the present single crystal NMR spectra. This finding indicates there is a small modification to the uniform part of the conduction electron density of state during the transition. That a relatively small portion of the Fermi surface is affected during the transition agrees with the observation from ARPES [5].

Figure 3(b) shows the variation of the modulation amplitude ν_1 with T. Below 28 K non-zero values of ν_1 and

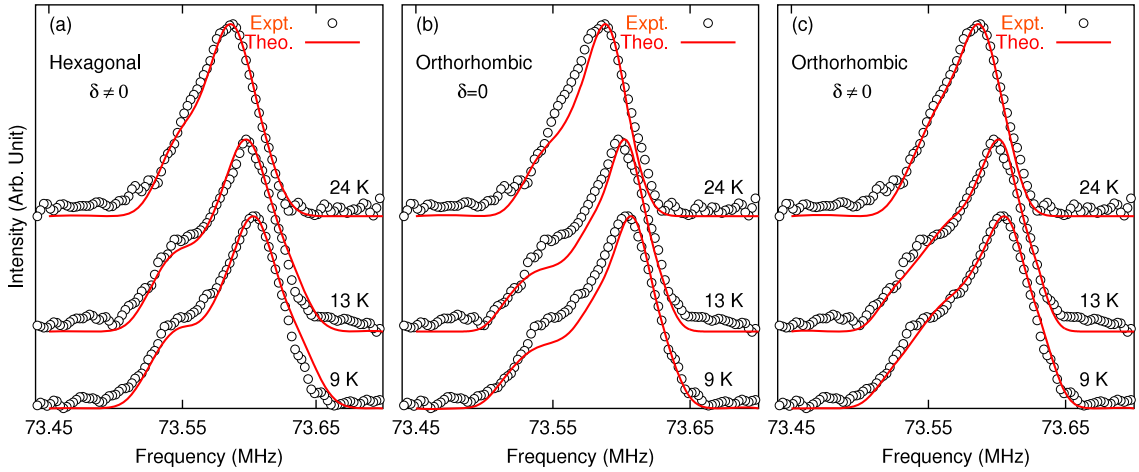

Figure 2. ^{93}Nb NMR spectra at 24, 13 and 9 K. The solid line represents the theoretical fitting, using equation (1) considering (a) case (1), (b) case (2) and (c) case (3).

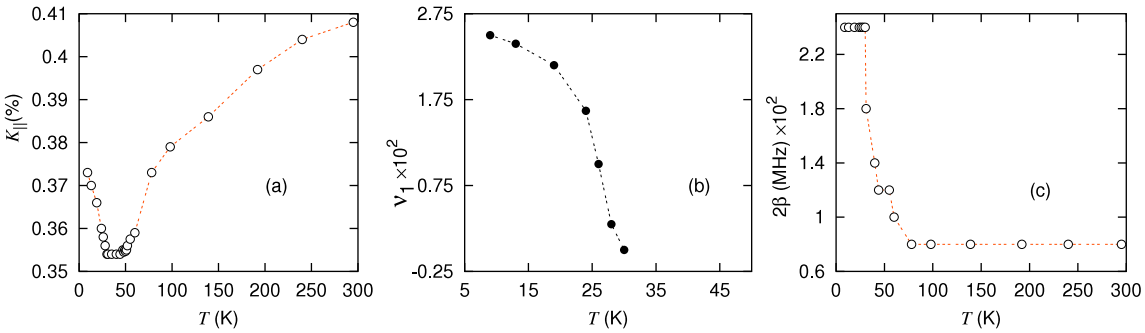

Figure 3. Temperature variation of (a) the Knight shift ($K_{\|}$), (b) the modulation amplitude (ν_1) and (c) the intrinsic line width (2β), respectively. Lines are a guide to the eye. The error in each case is the same as the size of the symbol.

δ appear. A continuous variation of ν_1 with T would suggest a second order nature for the phase transition, as indicated by the previous authors from the T dependence of the overall width of the NMR spectrum. The latter behavior also supports the prediction by McMillan [21, 22] using the Landau theory of phase transitions with the amplitude of the CDW as the appropriate order parameter. However, the NMR spectrum is not affected significantly with respect to variation of δ, as was reported by Berthier *et al*.

On the other hand it is seen that 2β (figure 3(c)) remains unchanged above 75 K, and then shows a continuous increment in the range 30 K $< T <$ 75 K. Below this range, 2β again becomes temperature independent. Such behavior of the intrinsic width of the central transition was not previously reported. It was only mentioned that an increment of the overall line width of the experimental spectrum occurred below T_{CDW}. The present observation of the enhancement of 2β in the range 28 K $\leqslant T <$ 75 K appears as a signature of change in the conduction electron spin dynamics, clearly starting above T_{CDW} and driving the system from a uniform into a modulated conduction electron density of states below T_{CDW}. In this connection it may be mentioned that Berthier *et al* [16] also reported a pretransitional broadening of the $(-1/2)$–$(-3/2)$ transition of the ^{93}Nb resonance in 2H-$NbSe_2$, by comparing the inverse of the measured spin–spin relaxation time (T_2) with the measured line width. They explained the excess line width as due to static impurity induced fluctuations. Stiles and Williams [23], however, explained the broadening of the same satellite transition as due to dynamical fluctuations of the CDW. Moreover, such pretransitional effects were also reported for 2H-$TaSe_2$ and 2H-TaS_2 from resistivity and specific heat measurements [24] as well as from thermal conductivity measurements [25] where the authors suggested that dynamical effects are responsible.

Finally, in order to check the prediction of Stiles and Williams [23] that the Knight shift distribution should be linearly related to the square of the CDW amplitude, we have plotted in figure 4 the normalized value of the total width of the experimental spectrum below T_{CDW} (after subtracting from it the high temperature line width) as a function of normalized $(\nu_1)^2$. The result agrees very satisfactorily with the prediction.

3.2. McMillan incommensurate model

To test the McMillan concept of discommensurations in 2H-$NbSe_2$, we have followed the procedure of Suits *et al* [13, 14]. This approach has the flexibility that by adjusting one parameter, γ, one obtains the NMR line shape corresponding to a conventional incommensurate ($\gamma \rightarrow \infty$), a McMillan

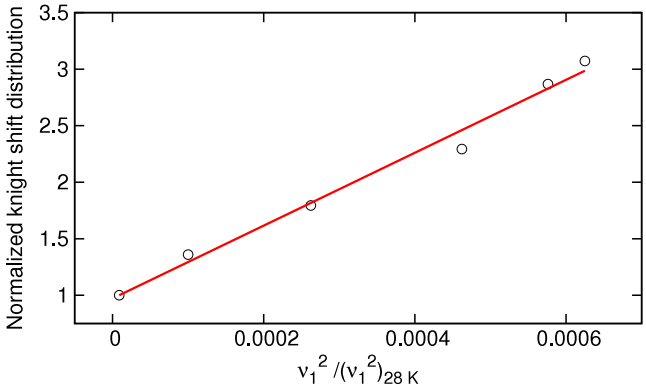

Figure 4. Variation below 28 K of the normalized Knight shift distribution with square of the CDW amplitude.

incommensurate or a commensurate ($\gamma \rightarrow 0$) line shape. In this case the modified form of the equation (3) is

$$\nu = \nu_0 + \nu_1 \Sigma_j \cos[\mathbf{q}_j \cdot \mathbf{R}_i + \theta_j(\mathbf{R}_i) - \theta_{j+1}(\mathbf{R}_i) + \theta_0]. \quad (6)$$

Different types of models are distinguished by the dependence of the θ_j s on the $\mathbf{R}_i$. The total NMR signal is calculated by fixing $\mathbf{R}_i$ and summing over the θ_j s with a weight function $\eta(\theta_j)d\theta_j$, which corresponds to how often a particular θ_j occurs throughout all the R_i s. $\eta(\theta)$ is given by [13, 14]

$$\eta(\theta) \propto \left[\sin^2\left(\frac{3\theta}{2}\right) + \gamma^2\right]^{-1/2}. \quad (7)$$

Note that γ gives a measure of the relative width of the phase slip region.

We have calculated the total NMR line shape from equation (6) by calculating the frequency of the NMR signal for each set of θ_j s and summing over all sets. We then broaden the resulting line shape with a Lorentzian function, which seemed a better choice in this case than the Gaussian model.

In figure 5(a) it is seen that the NMR spectra below T_{CDW} agree quite reasonably with the theoretical spectra with a fixed value of $\gamma = 1.0$. Figure 5(b) shows the theoretical spectra for $\gamma = 1.0$, and for different values of θ_0, superimposed on the experimental spectrum at 9 K. Figure 5(c) shows the experimental spectrum at 9 K superimposed on the theoretical spectra with γ lying in the range $0.1 \leqslant \gamma \leqslant 1.0$. With the amount of broadening necessary here, there is very little difference between a theoretical spectrum with $\gamma = 1$ and those with larger values of γ. The theoretical spectrum also depends on the value of θ_0. The agreement is best when $\theta_0 = 12°$. Moreover, from figure 5(c) it is seen that the extent of the mismatch also increases for values of $\gamma < 1.0$. In the case of 2H-$TaSe_2$ Suits *et al* [14] obtained the best agreement between the calculated and the experimental spectra of ^{77}Se NMR in the incommensurate CDW state for $\gamma \ll 1$. For these smaller values of γ the width of the phase slip region is not very broad, as may be seen from figure 4 of [14]. From this figure it is also seen that the phase slip region is quite broad for $\gamma = 1.0$, compared to the distance between the phase slips, so much so that a system with $\gamma = 1.0$ should closely resemble a conventional incommensurate state.

In the case of 2H-$NbSe_2$ the high value of γ suggests that the nature of the CDW state is very close to the conventional incommensurate state. The present NMR finding therefore suggests a much broader phase slip region in the case of 2H-$NbSe_2$ compared to that of 2H-$TaSe_2$. This also corroborates the results of electron microscope studies in 2H-$TaSe_2$ [11] and in 2H-$NbSe_2$ [19]. Moreover, the value of γ for 2H-$NbSe_2$ is found to be independent of temperature (figure 5(a)).

4. Conclusion

^{93}Nb NMR studies were carried out on a single crystal of 2H-$NbSe_2$ in the temperature range 9–300 K to reinvestigate the normal and CDW states at a higher resonance frequency than was done previously. The detailed analysis of the NMR line shape of the central transition monitors the change in the conduction electron spin dynamics starting from above T_{CDW}. The increase of the Knight shift once below T_{CDW} indicates a modification to the uniform part of the conduction electron density of states below the transition. Furthermore,

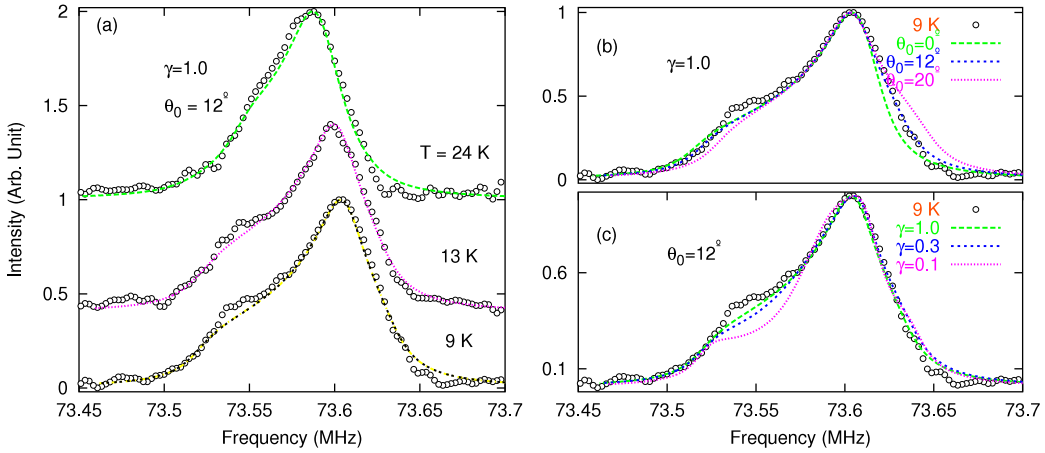

Figure 5. (a) ^{93}Nb NMR spectra at 24, 13 and 9 K. The solid line represents the theoretical fitting considering McMillan concept of discommensurations using equation (6). The effect of (b) θ_0 for $\gamma = 1.0$ and (c) γ for $\theta_0 = 12°$ on the line shape.

the present results demonstrate clearly that the Knight shift distribution is directly proportional to the square of the CDW amplitude. Variation of the incommensurability parameter (δ) could not be detected from the present study. The form of the temperature dependence of the amplitude of the CDW (which was theoretically suggested to be the appropriate order parameter [21]) clearly indicates the second order nature of the CDW transition. Application of McMillan's incommensurate model to analyze the spectra below T_{CDW} suggests there are much broader phase slip regions in 2H-$NbSe_2$ compared to those reported in case of 2H-$TaSe_2$ [14].

Acknowledgments

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