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X-ray scattering studies of $2H$-$NbSe_2$, a superconductor and charge density wave material, under high external magnetic fields

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Abstract. Using x-ray scattering we have measured the response of the incommensurate charge-density wave (CDW) modulation in the quasi-low-dimensional material $2H$-$NbSe_2$ to applied magnetic fields at low temperatures. The application of a magnetic field, either perpendicular or parallel to the layers of a single crystal of $2H$-$NbSe_2$, caused no significant change to either the correlation length or the intensity of the CDW satellites for magnetic fields up to 10 T. These results suggest that the enhancement of the resistance observed in low-dimensional CDW materials exposed to the applied magnetic field does not result from an appreciable conversion of carriers from the normal state to the CDW state. In addition $2H$-$NbSe_2$ is a superconductor at low temperatures ($T_c = 7.2$ K), whilst still within the incommensurate CDW state. This material therefore affords an opportunity to study any interaction between the CDW and superconducting condensates. High magnetic fields can suppress the superconducting state yet no change in the incommensurate CDW satellite correlation length or intensity was observed at high applied magnetic fields. These results conflict with spectroscopic measurements, which suggest a coupling between the superconducting gap excitations and the CDW.

1. Introduction

In a one-dimensional metal with a partly filled band, the lattice will never be stable at sufficiently low temperatures because the presence of the periodic potential would break the Fermi surface distribution, resulting in a Peierls distortion [1]. Such a distortion induces a corresponding spatial modulation of the electron density called a charge-density wave (CDW) throughout the crystal [2]: $\rho(\boldsymbol{r}) = \rho_n + P\cos(\boldsymbol{Q}\cdot\boldsymbol{r} + \phi)$, where ρ_n is the average electron density, P is the amplitude of the electronic density wave, $Q = 2k_f$ is the CDW wavevector and ϕ is the CDW phase which is, in a real system, pinned by impurities. Such a pinning effect dominates the transport behaviour in CDW materials. By applying an electric field exceeding the threshold field, the CDW becomes depinned and it carries additional current, $I_{CDW} \approx \rho_{CDW}\partial\phi/\partial t$. Although not observed in many CDW materials this nonlinear behaviour has been demonstrated in low-dimensional CDW materials such as $NbSe_3$ and the blue bronze $K_{0.3}MoO_3$ [3].

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Another fascinating aspect of the unusual dynamic and static phenomena caused by the CDW modulation is the response of the density wave to an applied magnetic field. Considering a two-dimensional electron gas exposed to an external magnetic field, Fukuyama *et al* [4] proposed that the system has a tendency towards the formation of a charge-density wave state. Further studies by Normand *et al* and Fujita *et al* constructed theoretical models showing the magnetic field could cause the formation of a variety of CDW states [5]. Balseiro and Falicov also reported that the response of the Fermi surface is related to field-enhanced CDW formation through an improvement of a nested Fermi surface [6]. This magnetic-field-enhanced gap would lead to a direct conversion of carriers from the normal to the CDW state. For an anisotropic metal with two open Fermi-surface sheets, Gor'kov and Lebed also showed that a SDW could be enhanced by a magnetic field applied parallel to the sheets [7].

Experimentally, Coleman *et al* measured the magnetotransport properties of $NbSe_3$ below 59 K being the onset temperature of a second CDW by applying magnetic field up to 23 T perpendicular to the high conductivity axis, and found an enhancement of resistivity at high fields [8]. They stated that the extra resistance could result from a decrease in the number of normal carriers, caused by the transfer of electrons into the CDW. From the noise and resistance measurements on $NbSe_3$ up to 7.5 T and at low temperatures, Parilla and co-authors also reported an enhancement of CDW carriers by an applied magnetic field [9]. Thus it became widely believed that the unusual transport behaviour observed in most CDW materials under applied magnetic fields resulted from an enhancement in the number of CDW carriers. However, in some other studies, a different conclusion was reached. Tritt *et al* [10] reported that the applied magnetic field had an effect of less than 5% on the CDW carrier concentration from measurements of the resistance and narrow-band noise in $NbSe_3$ for magnetic fields up to 10 T and temperatures in the range from 30 to 50 K implying that the increase in the resistance with magnetic field was not related to an increase in the CDW carrier concentration. They also pointed out that the opposite conclusion was reached only due to neglect of the contact resistance induced by the electrical leads. An x-ray scattering study of $NbSe_3$ reported no change of the CDW order parameter or wavevector with applied magnetic fields up to 10 T [11]. More recently, deformation of the CDW in $NbSe_3$ has been observed in the sliding state by x-ray diffraction. Spatially resolved high-resolution x-ray measurements have shown a shift of the CDW wavevector close to electrical contacts upon applying an electric field. The steep variation of the wavevector near the contact was modelled in terms of dislocation loops nucleated at host defects. A small constant gradient in the central part of the sample indicated incomplete carrier conversion consistent with dislocation loop pinning [12].

X-ray scattering affords a direct method observation of possible field-induced effects on the CDW state. Information obtained from x-ray scattering studies is straightforward to link to the changes of the CDW condensate, and the contact effects induced by leads are excluded. The integrated intensity of CDW satellite reflections is proportional to the square of the amplitude of the structural distortion associated with the CDW. This is related to the number of electrons in the CDW, which is a measure of the energy gap. The peak width provides information on the interplay between the CDW condensate and the applied field and the level of impurities which might pin the CDW. If the unusual magnetotransport phenomena observed in those CDW or SDW materials result mainly from the changes of CDWs by the applied magnetic field, such changes would be expected to be observed in the peak profile of CDW reflections. In addition, there has been speculation about the nature of any interaction between the CDW condensate and superconductivity. Dramatic changes of the density of states around the Fermi surface are caused by the transition to both a CDW state and a superconducting state. The application of a magnetic field was observed to suppress the intensity of Raman active superconducting-gap excitations, and simultaneously increase those of CDW-induced modes. This led Sooryakumar and Klein [13] to claim evidence of coupling between the CDW state and superconductivity.

The layered transition metal dichalcogenide $2H$-NbSe$_2$ provides a unique system for the study of CDW and superconducting state interactions. $2H$-NbSe$_2$ is a quasi-two-dimensional material and possesses a complex hexagonal shape of the Fermi surface being cut up by the introduction of CDW gaps with an incommensurate wavevector $q = (1/3)(1 - \delta)a^*$, at $T_{CDW} = 32.5$ K. Upon cooling the CDW remains incommensurate at least down to 5 K. In addition to exhibiting a CDW, $2H$-NbSe$_2$ is a high field anisotropic superconductor ($T_c = 7.2$ K) with upper critical fields as high as $H_{c2\parallel} = 130$ kG and $H_{c2\perp} = 32$ kG. This state displays both the flux line lattice and De Haas–van Alphen oscillations. There remains debate about the nature of the origin of the CDW state. Some authors have argued that the CDW state is caused by Fermi surface nesting [14, 15] with electron-phonon matrix elements playing a role [16]. In this case the CDW wavevector would be given by the nesting vector. Alternatively, Rice and Scott [17] have shown that a two-dimensional conduction band containing saddle points close to the Fermi level is unstable against a CDW distortion. In this case the CDW wavevector would be determined by the $\boldsymbol{k}$-space separation of the saddle points. Very recently angle resolved photoelectron spectroscopy measurements have strongly favoured the Fermi surface nesting mechanism [18].

In this paper we report detailed high-resolution x-ray scattering studies on $2H$-NbSe$_2$ at low temperatures and simultaneous high magnetic fields up to 10 T. Our results clarify the nature of the CDW modulation, the effects of the magnetic field on the CDW and the interaction between the CDW and the superconducting state.

2. Experimental details and results

A thick, faceted $2H$-NbSe$_2$ crystal was grown by C S Oglesby using iodine vapour transport methods employing small ampoules [19]. This technique produces large, high quality crystals with very low mosaic widths. The sample crystal employed is of flat hexagonal shape 0.4 mm thick and 5.00 mm in diameter with very prominent (0001) faces. $2H$-NbSe$_2$ has a hexagonal ($P6_3/mmca = 3.45$ Å, $c = 12.54$ Å) layer structure that is composed of strongly bonded molecular layers stacked in a close-packed manner [20]. This structure is highly anisotropic (like graphite) in which the layers are highly correlated, but perpendicular to the layers the structure is less correlated. This leads to very different mosaic widths: $\sim 0.04^\circ$ and $\sim 0.007^\circ$ (as measured on the (200) and (008) Bragg peaks). A variety of x-ray experiments were undertaken. First, the crystal was characterized at zero magnetic field and at temperatures down to 10 K. We used a high-resolution multi-axis diffractometer (station 16.3) located on a high-energy Wiggler beamline at the Synchrotron Radiation Source, Daresbury Laboratory. Upon cooling down from room temperature the CDW satellites were observed at temperatures below 32 K. Upon cooling the CDW increased in intensity and shifted in wavevector (relative to neighbouring Bragg reflections) in a systematic fashion (see figure 1). As shown in the figure the CDW peak remained incommensurate down to 10 K. Although the peak moves towards the commensurate position of 2/3, it does not lock in over the temperature range 32–10 K. This is in accord with the neutron results of Moncton *et al* [21]. Measurements of both the incommensurate wavevector and the normalized intensity are in agreement with the earlier results.

Further measurements of possible magnetic field induced effects were carried out on X22B at the National Synchrotron Light Source, Brookhaven National Laboratory. This is a two-circle diffractometer operating on a focused beamline, with an x-ray flux approximately 100 times that of station 16.3. Mounted upon the diffractometer is a 13 T split coil vertical field superconducting magnet operating in a helium Dewar with a base temperature of 1.5 K.

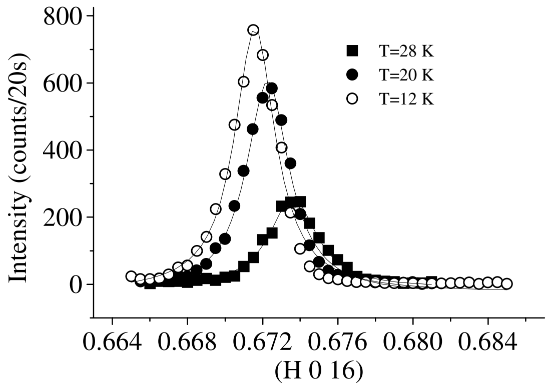


Figure 1. Variation of the CDW satellite (0.67 0 9) along the H-direction with temperature.

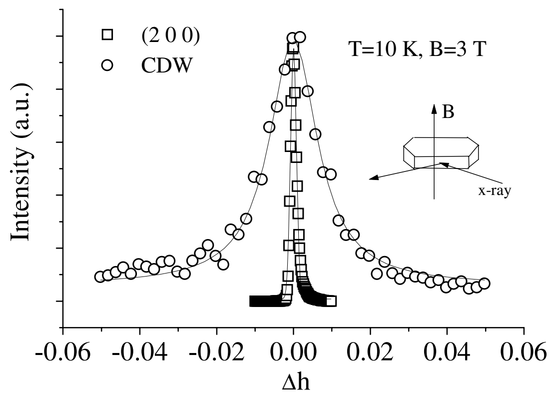


Figure 2. The widths of a Bragg reflection (200) and a CDW satellite (0.67 0 9) at $T = 10$ K and $B = 3$ T demonstrating the very different correlation lengths. The fitted lines are Lorentzian and Gaussian functions for the CDW satellites and Bragg reflection, respectively.

The crystal was pre-oriented on a four-circle diffractometer (X22A) and loaded into the magnet assembly. A horizontal axis sample rotator inside the magnet was used for *in situ* adjustments of the sample orientation. The magnetic field was applied perpendicular to the surface of the sample, in such a way that scattering plane was in the $a^* \times b^*$ reciprocal plane. The mosaic width of the sample was 0.04 degrees, and the instrumental resolution along the longitudinal direction was 0.002 r.l.u. (1 r.l.u. $= 2\pi/a^*$) as determined by the width of the (2 0 0) Bragg peak. With such a configuration we found that the momentum resolution around the CDW satellite (1.67 0 0) was 0.017 r.l.u. at $T = 5$ K. Measurements were taken on a CDW peak (1.67 0 0) and a nearby Bragg peak (2 0 0). Data obtained from the CDW reflection and Bragg peak are in good agreement with the Lorentzian function and the Gaussian function respectively as shown in figure 2. This figure demonstrates the very much larger width of the CDW as compared to Bragg peaks, indicative of a much reduced correlation length for the CDW.

Figure 3 shows the various conditions of temperature and magnetic field under which both the (2 0 0) Bragg reflection and the (1.67 0 0) CDW reflection were examined. In the first series of measurements (○), the sample was cooled from 35 K down to 4 K at zero field. No variation of the CDW wavevector was observed with temperature because the variation of the incommensurability, as measured by us and by Moncton [21], is comparable with the

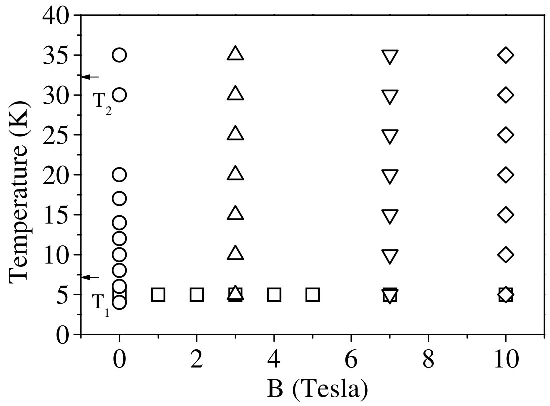


Figure 3. A schematic showing the differing conditions of temperature and applied magnetic field under which measurements were conducted. The individual runs are shown by the different symbols. The arrows on the temperature axis show the zero field superconducting transition temperature (T_1 = 7.2 K) and the CDW transition temperature (T_2 = 32.4 K).

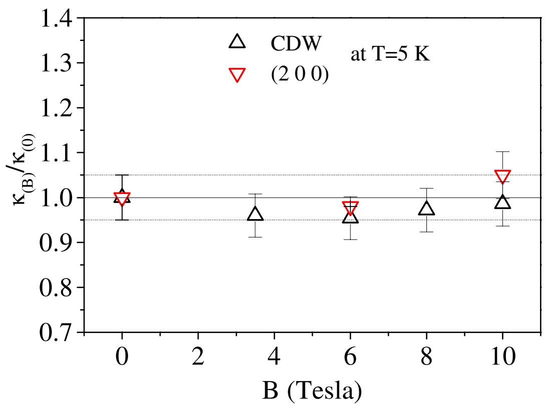


Figure 4. The variation of the inverse correlation length of both the (200) Bragg peak and the (0.67 0 9) CDW satellite with applied magnetic field at a fixed temperature of 5 K. The values have been normalized to their respective zero field values to minimize the effects of sample misalignment. No difference between the two data sets is observed, nor any significant variation with magnetic field up to 10 T. The horizontal line is a guide to the eye.

experimental geometrical resolution. At a fixed temperature of 5 K the field strength was then steadily increased from 0 T up to 10 T (□). We observed no variation in the position or width of the peak (corresponding to the wavevector and inverse correlation length, respectively). The variation of the inverse correlation length, which is related to the peak width, with applied magnetic field at a fixed temperature of 5 K is shown in figure 4. Because of possible misalignment of the crystal at high magnetic fields, the inverse correlation length for both the CDW (1.67 0 0) and the Bragg peak (2 0 0) are shown normalized to their zero-field values. No difference between the two sets of data is noted up to 10 T. Such measurements demonstrate that the upper bound on the magnetic field induced change of the correlation length is 10%. Theoretical studies by Bjeliš and Maki demonstrated that an applied magnetic field can reduce significantly the correlation length in the transverse direction being perpendicular to the magnetic field, but has only a small effect on the longitudinal direction [22]. Figure 4

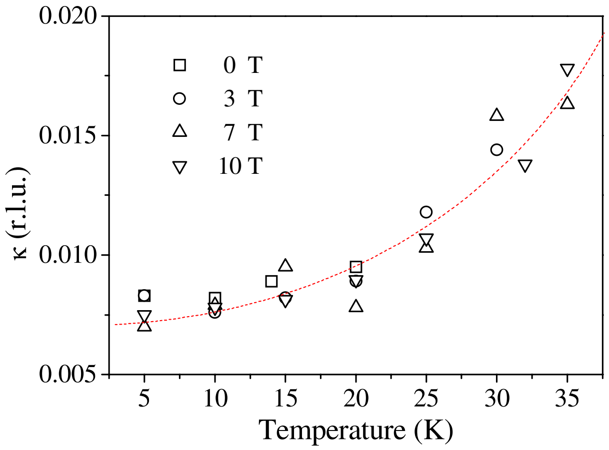


Figure 5. The variation of the inverse correlation length of the (0.67 0 9) CDW satellite along the longitudinal direction at fixed magnetic field (0, 3, 7 and 10 T) with temperature. The dotted line is a guide to the eye.

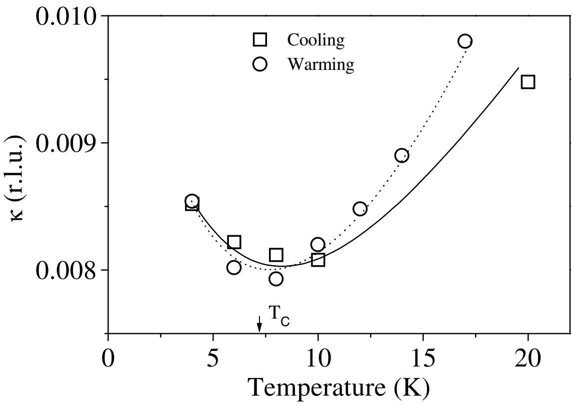
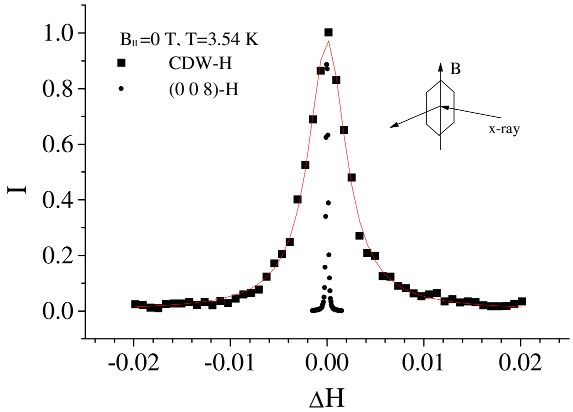


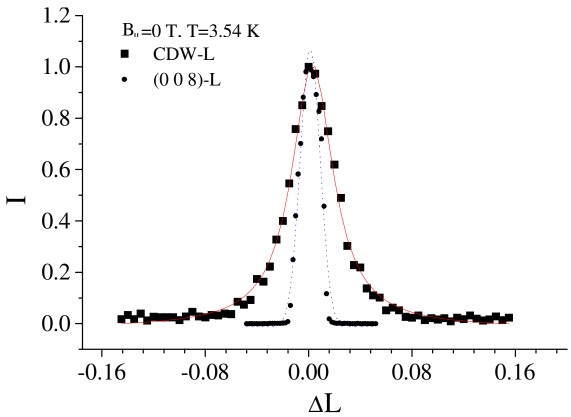
Figure 6. The variation of the inverse correlation length of the (0.67 0 9) CDW satellite at zero applied magnetic field (0 T) with temperature in the low temperature range 0–20 K. Two different data sets are shown, one obtained upon cooling the sample and the other upon warming the sample. The lines are guides to the eye.

shows only minor changes in the peak widths of CDW peak and Bragg peak when the magnetic field was increased, but the changes were only within 5% as compared with zero-field widths, which is just about the resolution limit. Again, the changes of the peak widths may be caused by movement of the sample holder at high magnetic fields, and thus we cannot be certain that an applied magnetic field has any discernible influence on the coherence length of the CDW.

Measurements were also taken at variable temperature (35 K to 5 K) with a fixed applied magnetic field as shown in figure 3. Figure 5 displays these field-cooling measurements of the CDW reflection. There were some difficulties in fitting the peak profiles close to the transition temperature (32 K) at higher magnetic fields due mainly to their weak intensities. However, the decrease of the coherence lengths on the field-cooling measurements was dominated by temperature effects, as observed in the zero-field measurements in figure 5. No significant differences between runs in the range 0 to 10 T were observed.



(a)



(b)

Figure 7. The widths of a Bragg reflection (008) and a CDW satellite (0.33 0 9) at $T = 3.54$ K and $B = 0$ T demonstrating the very different correlation lengths in both the H and L directions. The fitted lines are Lorentzian and Gaussian functions for the Bragg and CDW satellite respectively.

All of the above results suggest there is no discernible effect on the CDW satellites by the application of high magnetic fields. However the inverse correlation length of the CDW satellite does show a significant variation with temperature (as shown in figure 5) and in the low temperature regime (4–15 K) at zero magnetic field displays a possible minimum at approximately 7 K (see figure 6). This is very close to the superconducting transition temperature ($T_C = 7.2$ K) and may be evidence of an interaction between the CDW and superconducting condensates such that as the temperature is lowered within the superconducting CDW state the inverse correlation length increases. This could be imagined to be because of possible frustration between the two competing states leading to a reduction in the CDW coherence length. Unfortunately this possible minimum in the inverse correlation length was very close to the minimum temperature obtained in the experiment. Furthermore any possible increase in the inverse correlation length, which would be a signature of such an interaction, would also be small.

In order to test this hypothesis, another experiment was undertaken with the sample remounted such that the layers were parallel to the field direction. In this geometry, with the sample a^* and c^* directions in the horizontal scattering plane the experimental resolution

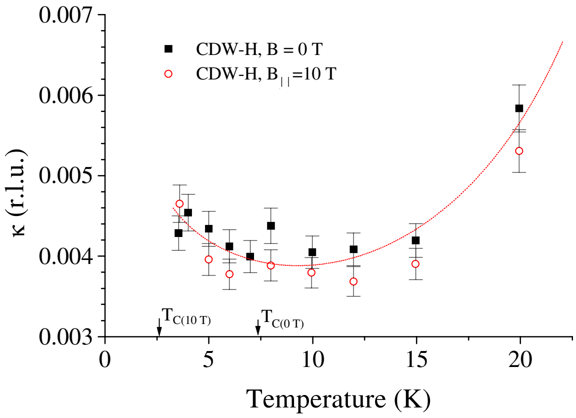


Figure 8. The variation of the inverse correlation length of the (0.67 0 9) CDW satellite at fixed magnetic field (0 and 10 T) with temperature. The arrows on the temperature axis show the zero-field superconducting transition temperature ($T_{C(0\ T)} = 7.2$ K) and at high magnetic field ($T_{C(10\ T)} = 3$ K).

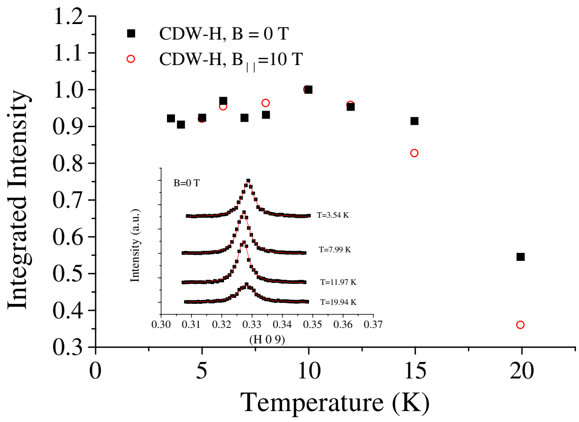


Figure 9. The variation of the integrated intensity of the (0.67 0 9) CDW satellite as a function of temperature at both zero and high applied magnetic fields. The inset displays the peak at various temperatures up to 20 K.

is much higher. The resolution was measured and found to be 1.8×10^{-4} Å^{-1} in the transverse direction and 4.5×10^{-3} Å^{-1} in the longitudinal direction as measured on the (008) Bragg peak at 3.54 K (see figures 7(a) and 7(b)). Measurements of the (0.33 0 9) CDW satellite show a much greater width demonstrating that the CDW has not developed a long-range order even at 3.54 K. The very high wavevector resolution at the (0.33 0 9) position allows us to consider the maximum quantitative limit of any wavevector variation with applied magnetic field. Since a wavevector shift equal to three times the instrumental resolution would have been observable, a conservative upper limit of $\Delta q_a/q_a \leqslant 1 \times 10^{-3}$ on the shift of the CDW wavevector in a magnetic field of 10 T can be given ($q_a = 0.33\,a^* = 0.601$ Å^{-1}). By applying a magnetic field perpendicular to the $NbSe_2$ layers within the crystal, we depress the superconducting transition temperature (T_C) such that at 10 T it has been reduced to about 3 K [23]. Scans taken through the (0.33 0 9) CDW peak were fitted to Gaussians and the peak intensities and widths were extracted. However, figure 8, which displays the variation of the CDW inverse correlation length (peak width) with decreasing temperature, at both zero applied field and at a magnetic field of 10 T, shows no differences between the two data sets. This is strong evidence against any interaction between the CDW state and the superconducting state. Similarly a plot (figure 9) of the variation of the CDW integrated intensity (proportional to the square of the amplitude of the structural distortion associated with the CDW) with temperature at both ambient magnetic field and 10 T shows very similar behaviour. The peak amplitude does not does change with magnetic field by more than 10%. Given that the peak width is also independent of magnetic field the peak intensity is proportional to the integrated intensity, which again does not vary by more than 10%. This 10% limit on the change of the integrated intensity therefore corresponds to an upper bound of 5% on the variation of the CDW order parameter in a magnetic field of 10 T.

3. Discussion

In a CDW state the density wave can be regarded as a modulated electron density state consisting of both electron density of spin-up and spin-down in phase. If they shift in a phase of $\pi/2$, the system is a pure spin-density wave (SDW). The application of a magnetic field can lift the degeneracy of both electron density states, and then shifts the spin-split phase ϕ from 0 toward $\pi/2$, and thus CDW and SDW states coexist during the process. Changes in the CDW wavevector and the amplitude would be therefore expected [22]. Studies of the instability of quasi-low-dimensional materials with a nested Fermi surface exposed to an applied magnetic field have also shown a tendency toward the formation of a SDW and pointed out that the CDW condensate could be suppressed by a strong magnetic field. The TDPAC experiment by Butz *et al* on $2H$-TaS$_2$ provided the evidence of the coexistence of both CDW and SDW [24], and a coupling between both superconducting and CDW condensates was observed in Raman scattering with applied magnetic fields perpendicular to layers of $2H$-NbSe$_2$ [13]. Our results are more in accord with those of Fleming *et al* who observed no change in the CDW wavevector or coherence length with applied electric field in NbSe$_3$ [25] and with Kiryukhin *et al* who reported no change of the CDW wavevector in NbSe$_3$ with applied magnetic field [11]. Indeed the coupling between the superconducting and the CDW condensates is also presumed weak as little change was observed below 7.2 K (T_C). Further any coupling between the superconducting condensate and magnetic field is not appreciable enough to change the CDW satellites.

In summary, we have undertaken a number of high-resolution x-ray scattering studies of the CDW satellites in $2H$-NbSe$_2$ under a variety of temperatures and applied magnetic fields. Our measurements show that there is no discernible change in the CDW wavevector with applied magnetic field up to 10 T. In addition the CDW correlation length, which is discernibly shorter than the structural correlation length, as evidenced by the width of Bragg reflections, also displays no change with applied magnetic field.

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