

Experimental report

25/01/2024

Proposal: INTER-593

Council: 10/2023

Title: Magnetic field dependence of the electronic excitations in NbSe₂

Research area:

This proposal is a new proposal

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Samples: NbSe₂ (P63/mmc a = b = 3.443Å, c = 12.547Å, alpha = beta = 90, gamma = 120)

Instrument	Requested days	Allocated days	From	To
THALES	3	3	08/12/2023	11/12/2023
ORIENTEXPRESS	1	1	06/12/2023	07/12/2023

Abstract:

Experimental Report

Instrument: ThALES

Experiment Number: INTER-593

Title: Electronic excitations in the superconducting state of $2H\text{-NbSe}_2$.

Experiment Date: 08/12/2023 – 11/12/2023

Experimental Team: Ahmed Alshemi, Elizabeth Blackburn (Lund University), Arno Hiess, Karin Schmalzl (ILL)

Local Contact: Arno Hiess

Introduction

$2H\text{-NbSe}_2$ is a highly two-dimensional superconductor ($T_c \sim 7$ K) which also displays charge density wave order ($T_{\text{CDW}} \sim 35$ K) with a propagation wavevector of $((1-\delta)\mathbf{a}^*/3 \ 0 \ 0)$, where $\delta \sim 0.02$ and is temperature dependent. The appearance of the charge density wave and the specific transition temperature can be sample dependent [1].

Experimental Configuration

Two experimental configurations were used during the experiment.

- Configuration A:** $k_f = 1.5 \text{ \AA}^{-1}$, W setup (1, -1, 1)
Elliptical guide – Selector – Virtual source – PG mono – open – Entrance slits – Sample – Analyzer slits – Be Filter – open – PG analyzer – (radial collimator) – Detector
Energy resolution = ?? meV as measured on ??
Slits used (T,B,L,R): D1: 12, 15, 11, 11; D2: 18, 18, 12, 12
The radial collimator was tested but then not used further as it did not improve the signal-to-noise ratio.
- Configuration B:** $k_f = 2.2 \text{ \AA}^{-1}$, long chair setup (1, 1, -1)
Elliptical guide – Selector – Virtual source – PG mono – open – Entrance slits – Sample – Analyzer slits – 60' collimation – PG analyzer – open – Detector
Energy resolution = 0.440 meV as measured on incoherent position $\mathbf{Q}_{\text{inc}} = (1.1 \ 0 \ 1.8)$.
Slits used (T,B,L,R): D1: 14, 14, 15, 9; D2: 18, 18, 14, 13

The sample was mounted in the 4 T horizontal field cryomagnet (134 OX HV 38). For part of the experiment, the sample was rotated inside the magnet ($\mathbf{a3p}$), and later, when the field was applied, the sample was fixed with respect to the magnetic field, which was placed along $\mathbf{c}^*$, and so the relevant rotation motor is $\mathbf{a3}$.

The entrance and analyzer slits were checked on the (004) and the (101) Bragg peaks, and were put to the larger option in both cases. This led to the slits being quite wide open in both cases. We did not redo them for Configuration B but had measured just after the sample was re-installed after it was removed.

Sample Details and Alignment

Two samples were provided by Kai Rossnagel (Kiel University), of mass 284 mg and 143 mg. The sample alignments were checked on Orient Express, to identify the $\mathbf{a}^*$ and $\mathbf{c}^*$ axes. The samples were wrapped in aluminium foil and then the foil was glued to some Al struts with hexagonal cut-outs using GE varnish. The samples are separated by ~ 8 mm horizontally. One of the samples contained some elements of powder. They were co-aligned to better than 1° .



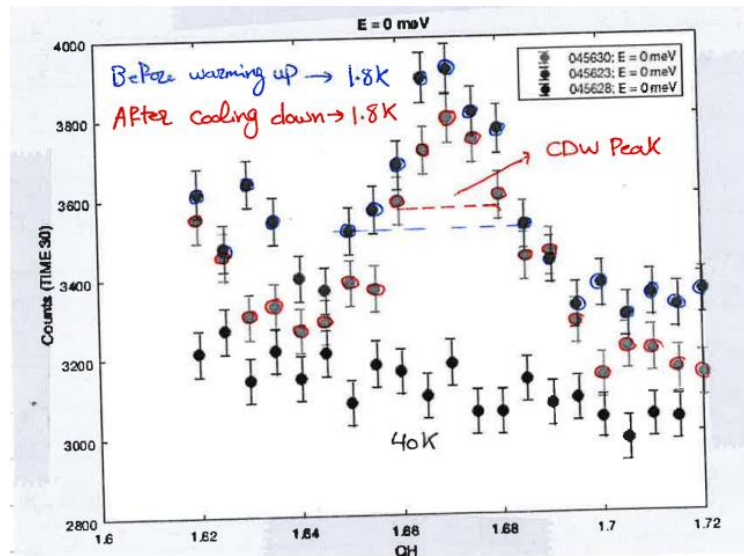
The lattice parameters were found to be $a = b = 3.4 \text{ \AA}$ and $c = 12.5 \text{ \AA}$ at low temperature (1.8 K). The space group is $P6_3/mmc$. The sample was mounted with a $[H0L]$ scattering plane. When aligning the sample, we used the (004) and then the (101)-type reflections. Initially, we tried to use the (100) reflection for alignment, but this is much weaker than the (101) reflections, which are very close in both a_3 and a_4 to the (100). The tails of the (101) affected the (100) reflection giving false lattice parameters. It is therefore better to check the (101) and (10-1) instead.

During the experiment, when in Configuration B, we could clearly see different crystal signatures; only one appeared to give a clear, undeniable charge density wave reflection.

A large background was observed throughout the experiment. This clearly came from the sample. The sample was lifted out of the beam and the background measured at $\mathbf{Q}_{inc}$ in 30 seconds was 134 counts as opposed to 7132 with the sample in. We found that when we shifted the sample stick height further to illuminate the cadmium protection wrapped above the sample, the counts leaped to 25000. We therefore removed the sample stick and removed the cadmium and an aluminium foil bag round the sample. On replacing the sample, the count level was reduced to 6640 counts at $\mathbf{Q}_{inc}$. We did not consider that this improvement was sufficient to improve the statistics as measured in Configuration A, and so switched to Configuration B at this point.

Charge Density Wave Order

A clear CDW Bragg reflection was observed in Configuration B at $(1.67 \ 0 \ 0)$. This is a strong reflection based on Moncton *et al.*'s evaluation [2]. The graph below has a counting time of 30 seconds per point.



Energy Scan Results

Energy scans were done at qCDW at 1.8 K and 10 K in Configuration B, and then several fields were measured, up to 4 T. However, we discovered that there was a reproducibility issue with the sample positioning that appeared at one point, and we had to offset a5.

References

- [1] D. J. Huntley and R. F. Frindt, Can. J. Phys. 52, 861 (1974).
- [2] D. E. Moncton *et al.*, Phys. Rev. B **16**, 801 (1977).