

Physica B 284-288 (2000) 1657-1658



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Single-crystal rings of NbSe₃: a system for CDW interference?

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Abstract

We have produced new ring- and tube-shaped crystals of NbSe₃ and found reproducible synthesizing conditions. We have confirmed that the ring is a single-phase NbSe₃ crystal from X-ray diffraction, and that a charge-density wave (CDW) transition takes place near 140 K by observing satellites of electron diffraction peaks. The position of the satellites coincides with that of the conventional NbSe₃ at the CDW transition. Non-linear conduction due to the CDW sliding is also observed in the ring crystals. The threshold field is similar to the conventional ribbon-shaped NbSe₃ crystals. \bigcirc 2000 Published by Elsevier Science B.V. All rights reserved.

Keywords: Charge-density wave; Quantum interference; Ring; Topology

Macroscopic and mesoscopic topological matter such as a spherical and torus and Moebius strip have been the subject of much theoretical and experimental work for the sake of its wealthy physical phenomena. Persistent current in a metal ring threaded by a magnetic flux was proposed theoretically and observed experimentally [1]. Interference phenomena of electrons have also been investigated for a metal ring; the Aharonov-Bohm (AB) effect for thin gold rings [2] and the Altshuler-Aronov-Spivak (AAS) effect for tube-like Mg films in the weakly localized regime [3,4] and AAS-like effect for NbSe₃ with columnar defects [5]. Persistent current and interference phenomena can also be observed for the electronic condensate in superconductors of ring or tube shape. Can the persistent current exist also in ring- and tube-shaped charge-density wave (CDW) systems?

In order to observe those effects, it is necessary to obtain an ideal ring-shaped sample of a one-dimensional (1D) material, such as organic-conductors or CDW-NbSe₃ of MX_3 group systems. We have made ring- and

tube-shaped crystals of NbSe₃, and found reproducible synthesizing conditions.

The samples were prepared by the chemical vapor transportation method. Single crystals of ring NbSe₃ were grown by using a solidification technique. This employs a stationary closed quartz tube inside a box furnace. Starting materials were composed of a mixture of bulk Se and Nb. The purity of all materials used in this work was 99.999%. The mixture was reacted at 740°C for three weeks in situ quartz tube and then cooled to 240°C at which temperature the oven was shut off. Fig. 1 is



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Fig. 1. Ring-shaped crystal of $NbSe_3$. Diameter of the ring is 100 μm .



Fig. 2. Satellites of CDW of ring-NbSe₃ by electron diffraction at 138 K, which is below the CDW transition temperature T_1 . Q_1 (0 0.24 0) is the wave vector of the CDW, b^* the reciprocal lattice vector.

an optical microscope picture of a typical ring-shaped crystal of NbSe₃.

We ground up the samples of ring-shaped crystals in order to identify composition and crystal structure. The powder of the ring-shaped crystals shows a monoclinic NbSe₃ single phase by the measurements of the X-ray diffraction methods. We found the ring-shaped crystal is not a Nb₃Se₄, Nb₂Se₃, NbSe₂, Nb₂Se₉ or other binary compounds of Nb and Se. Lattice constants were a = 10.01 Å, b = 3.48Å, c = 15.63 Å, $\beta = 108.5^{\circ}$, respectively. The ring-shaped samples were confirmed to be single crystals of NbSe₃.

Next, we performed electron-diffraction measurement of the ring-shaped NbSe₃ at 138 K (less than T_1 , see



Fig. 3. Temperature dependence of resistivity of the ring-shaped crystal along the circumference of NbSe₃.

below). Fig. 2 shows satellites due to the CDW wave vector of the ring-shaped NbSe₃ crystals. The CDW wave vector Q_1 is (0 0.24 \pm 0.01 0), determined from the position of the satellites diffracted by superlattice reflection. The position of the satellites coincides with that of the conventional NbSe₃ at the CDW transition. We confirmed that a CDW transition takes place in this ring of NbSe₃ near 140 K. The CDW transition was also confirmed by the temperature dependence of the resistivity of a ring-shaped NbSe₃ (Fig. 3). For this crystal, the usual four-probe method was used. There are two resistivity anomalies both along the *a*- and *c*-axis, as in previous reports of temperature dependence of resistivity. They are caused by the CDW transitions at 145 K (T_1) and 54 K (T_2).

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