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IRRADIATION EFFECTS AND THE ROLE OF DISORDER IN LOW DIMENSIONAL CONDUCTORS

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The concentration of defects produced by irradiation can be measured by several independent methods such as longitudinal and transverse conductivity measurements at room temperature, spin susceptibility at low temperature, volume or lattice parameters change. The physical consequences of this kind of disorder are as follows. The superconductivity in $(TMTSF)_2PF_6$ is destroyed by a molecular concentration of 10^{-4}. The concentration of 2.10^{-3} corresponds to the full pinning of charge density waves in monoclinic TaS_3, 1T-TaS_2, TTF-TCNQ, TMTSF-DMTCNQ ... Microdiffraction experiments demonstrate some structural aspects of this pinning and conductivity, Hall effect and thermoelectric power measurements demonstrate the consequences of the pinning on the electronic properties of several low temperature charge density wave insulators. In most of the cases the metallic state is stabilized by a weak disorder. The problem of the charge density wave motion in presence of defects is discussed in connection with the electric field depinning experiments. Concentrations of the order of 10^{-2} or more correspond to a concentration range of localization by disorder.

INTRODUCTION

The litterature reports more than 20 recent irradiation

experiments on low dimensional conductors either organic or inorganic, either quasi-one- or quasi-two-dimensional[1-23]. A non negligeable part of these experiments has been performed in Fontenay-aux-Roses. The present review summarizes the most recent results in our group and is an attempt to compare a few aspects of irradiation effects in three kinds of low dimensional conductors : layered dichalcogenides such as TaS_2, quasi-one-dimensional inorganic metals such as TaS_3 and organic metals such as TTF-TCNQ.

In a preliminary and short part of the paper it is demonstrated that the mechanisms of damage production are known sufficiently well to provide good estimations of the defect concentrations.

The main part of the paper is devoted to a discussion of the charge density wave pinning. Microdiffraction experiments in TaS_2 and TaS_3 reveal some structural aspects of this pinning while transport properties measurements demonstrate its consequences on the electron gas behaviour. These transport measurements include Hall effect on TaS_2 and TMTSF-DMTCNQ, thermoelectric power on TaS_3 and TMTSF-DMTCNQ and conductivity on all the systems studied.

DETERMINATION OF THE DEFECTS CONCENTRATIONS

The greatest advantages of the irradiation are that it permits studies in situ and that at low irradiation doses the number of defects is always proportional to the irradiation dose. Thus, it is possible to follow physical properties as a function of defect concentrations up to a few 10^{-2} atomic or molecular fraction and to compare accurately different concentrations at the level of 10^{-5} atomic or molecular fraction. To know the absolute value of the defect concentration is more difficult and requires in each case an accurate study of the radiation damage process.

In inorganic compounds, fast electron irradiation is the best way of producing point defects, one by one, in a controlled way. They are produced by the direct collisions of the incident particles with the nuclei. The absolute concentration of displaced atoms can be estimated within a few 10^{-1} accuracy if the energies needed for displacing each kind of atoms have been determined at first (displacement threshold energies). We have observed that in 1T-TaS_2 and in TaS_3 the damage produced in the sulfur sublattice recovered completely around 10 K. This leads to

the conclusion that the defects stable around room temperature are associated with the displacement of tantalum atoms. In 1T-TaS_2 and in monoclinic TaS_3 the displacement threshold energies for tantalum displacements have been found to be 13 ± 3 eV and 15 ± 1 eV respectively[1,2].

In organic conductors the mechanisms of damage production are completely different. The best irradiation particles from the point of view of the damage definition are X or Γ photons or fast electrons. In these cases the number of defects produced by direct knock-on is completely negligeable with respect to the number of molecules destroyed by electronic excitations or ionizations. The mechanisms of damage production have been studied in a recent paper by G. Mihaly and L. Zuppiroli[3]. Irradiation damage is shown to be a local chemistry of excited molecules. The incident radiation provides the creation of strongly excited or ionized electronic states. If there is no decay of such a state during a sufficient time, either radiative or by electron interactions, a radical, a molecule or a group of molecules can rearrange in a new stable chemical structure. This is the defect responsible of the electronic barrier. It is difficult to imagine, at present, what sort of intramolecular and intermolecular modifications one can expect from this local interaction ; however, the defect structure being determined mainly by the structure of the host, the possible defect configurations are certainly very few and probably one or two. The fraction c of destroyed molecules with the total absorbed energy: $c = E/\bar{E}$ where E is the absorbed energy per molecular unit and $\bar{E}$ is 7.1 keV for TMTSF-DMTCNQ, 13.5 keV for TTF-TCNQ and 147 keV for TTT_2I_3 (room temperature values)[3]. The local structure of the molecules are probably responsible for these differences more than the crystal structures or the electronic metallic properties.

Recent experimental investigations on organic metals[4,5] have demonstrated that the quasi-one-dimensional character of these compounds provides a possibility to determine the concentration of damaged molecules. This result was based on the following arguments. Each defect interrupts the conducting chains and the electrons are forced to jump to a neighbouring chain. The resistivity along the chains increases by the fraction of transverse resistivity mixed into the longitudinal conduction path[3,4,6]. This leads to a decrease of the anisotropy with increasing defect concentration. On the other hand, the chain segmentation effect

modifies the transverse conductivity too. It varies exponentially[4,5] with the defect concentration. This leads for the molecular fraction of electronic barriers to the expression $c \simeq \Delta\rho_{/\!/} / \rho_{\perp}$ where $\Delta\rho_{/\!/}$ is the resistivity increase along the conducting axis and $\rho_{\perp}$ the transverse resistivity in the direction of lower anisotropy.

In a recent study of the magnetic properties of irradiated TMTSF-DMTCNQ, Amiell, Delhaès and Zuppiroli[7] have shown that the number of localized spins responsible for the low temperature tail in the susceptibility is roughly equal to the number of barriers determined by the relation $c \simeq \Delta\rho_{/\!/} / \rho_{\perp}$. This is not in contradiction with the previous irradiation results on TTF-TCNQ by Miljak, Korin, Cooper, Holczer and Janossy[8] and on $Qn(TCNQ)_2$ by Sanny, Gruner and Clark[19], and justifies, a posteriori, the defect concentration scale in the early irradiation work of Chiang, Cohen, Newmann and Heeger[9].

Finally, a very recent experiment by Trouilloud, Ardonceau, Bouffard and Zuppiroli[10] provides a third verification of the dose scale. They have measured the changes in size of crystals of TTF-TCNQ submitted to an electron irradiation. The damage scale, in this third experiment fits very well the previous "transport" and "magnetic" scales provided that one takes the total volume change per molecular defect to be 1 ± 0.2 molecular volume, a very reasonable value for a strong local chemical reorganisation of the molecules.

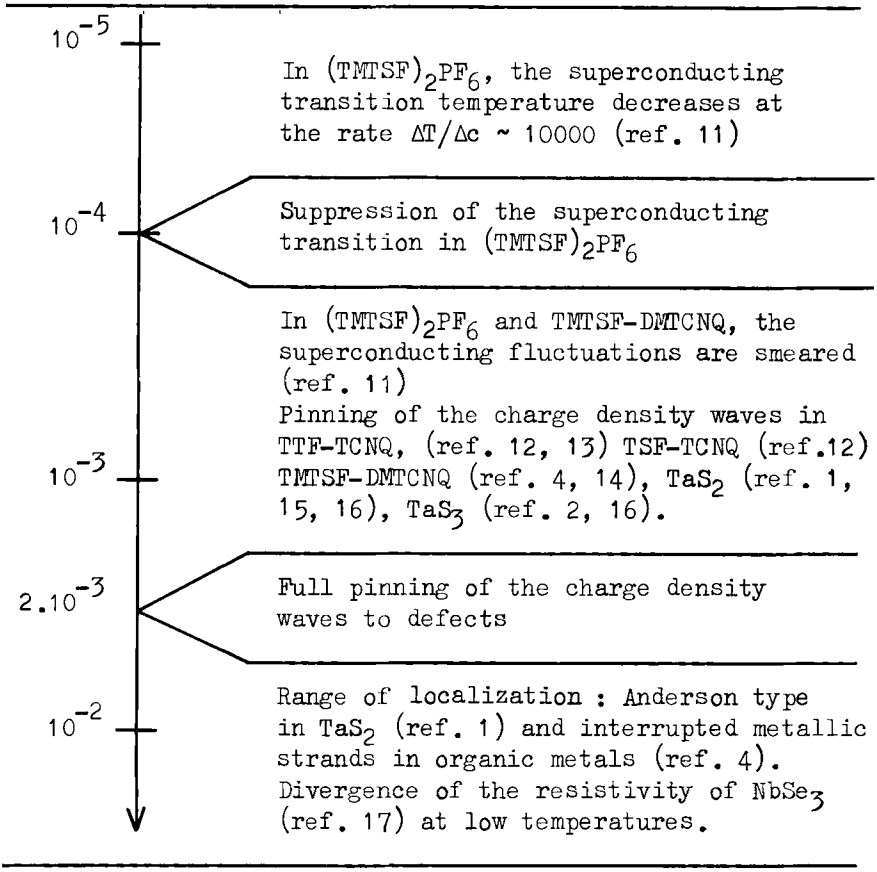
PHYSICAL PROPERTIES OF IRRADIATED SAMPLES

Introduction

The main consequences of irradiation on the properties of quasi-one-dimensional conductors are summarized on table 1. The concentration scale is common to organic metals and to 1D and 2D chalcogenides but we have to keep in mind that in the case of organic metals c corresponds to a fraction of damaged molecules when in chalcogenides it is a fraction of displaced atoms.

The superconductivity of organic conductors is a very fragile instability of the electron gas : a concentration of 10^{-4} is enough to destroy the superconducting transition[11]. Ten times more defects are needed to obtain the same result in $NbSe_3$, according to W.W. Fuller[17]. According

Table 1 Effects of Irradiation Disorder on Low Dimensional Conductors



to Mutka[18], several percent of defects are needed to destroy superconductivity in the layered compound 2H-$NbSe_2$. In the A 15 superconductors such as Nb_3Ge, the superconducting transition temperature decreases with increasing disorder but there is a saturation : it is impossible to remove superconductivity, even in the amorphous material. Nothing very general regarding defects and superconductivity in low dimensional conductors can be deduced from our present knowledge. We will not speak much more of superconductivity and superconducting fluctuations in the present review but it is worth mentioning that disorder and superconductivity in $(TMTSF)_2PF_6$ has been carefully explored in a very recent

paper by Bouffard et al.[11].

For similar reasons we will not speak very much here of the lower part of Table 1 : the localization range corresponding to concentrations of about 1% or more. The properties of the electron gas in this region depend on dimensionality and have been studied in ref. 1 for TaS_2 and 4 for organic conductors.

We have choosen to focus on the charge density wave pinning that is to say on the concentration range between 10^{-4} to a few 10^{-3}, because it is a 3-D effect ant it seems to depend much less on the precise chemistry of a large class of low dimensional conductors.

Structural Evidences of the Pinning

The pinning effect of defects on the charge density waves originates from the enhancement of the Friedel oscillation around a perturbing potential. In a low dimensional material that is apt to form charge density waves, the Friedel oscillation and its associated lattice distorsion have long range ; this induces pieces of charge density waves, the phases of which are determined by the perturbing potentials and the positions of the defects. Charge density wave pinning has been extensively studied theoretically[25] (see, for example the contribution of Bardeen to this conference) ; experimentally, the evidences of the pinning are hardly ever direct : most of the experiments deal with non linear transport properties due to the depinning by an electric field (see, for example, the contributions of Grüner or Monceau, this conference). More direct evidences of the pinning are reported in the present paper. These are electron microdiffraction studies in the disordered low temperature phases of 1T-TaS_2 and TaS_3 (monoclinic phase). The periodic lattice distorsions associated with the charge density waves produce diffuse lines and superstructures which can be observed on the microdiffraction patterns of pure and irradiated samples or followed "in situ" during irradiation at 7 K in a high voltage electron microscope[2,16].

In quasi-one-dimensional monoclinic TaS_3, uncorrelated charge density waves are present at room temperature ; they produce diffuse lines on the microdiffraction patterns. At low temperatures, below the metal to insulator phase transitions the charge density waves order into a tridimensionnal superstructure : the diffuse lines condensate into spots[24]. This is true only in absence of irradiation defects.

If the sample is irradiated at 7 K in a high voltage electron microscope, the charge density waves spots begin to disappear around a concentration of 2.10^{-4} displaced tantalum atoms. When the defect content is 10^{-3} there is no longer a tridimensional superstructure but uncorrelated charge density waves giving rise to diffuse lines very similar to those seen at room temperature in the pure sample. At 10^{-2} diffuse lines are still present. (The microdiffraction patterns can be seen in references 2 and 16, where the details of the experiments are described).

In quasi-two-dimensional 1T-TaS_2, charge density waves with three different wave vectors are present at room temperature. They are ordered in a three-dimensional superstructure which is incommensurate with the lattice (fig. 1-a). At low temperatures, below the phase transitions the charge density waves lock on the lattice periodicity (fig. 1b).

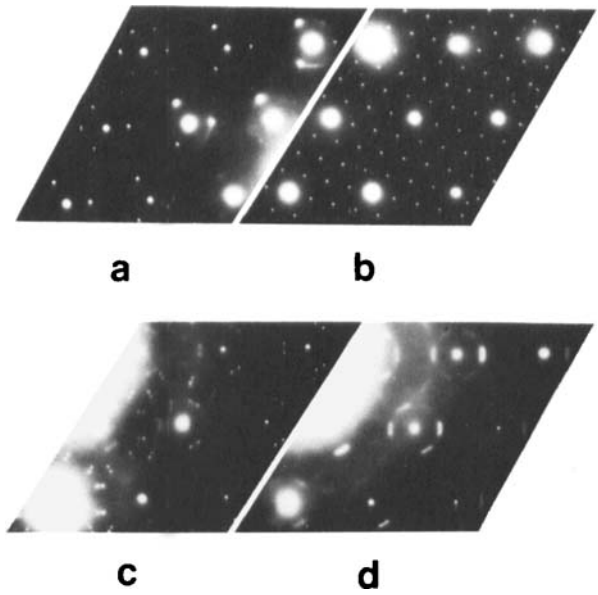


FIGURE 1 Charge density waves superstructures in 1T-TaS_2 : a/ pure crystal, 300 K, above the phase transitions ; b/ pure crystal, 100 K, below the phase transitions ; d/ irradiated crystal (3.10^{-2}), 100 K ; c/ irradiation and annealing at 400 K, observation at 100 K.

This is only true in absence of irradiation defects. When the sample is irradiated at 7 K in the high voltage electron microscope, the commensurability is lost for concentrations of defects of a few 10^{-3}. At 3.10^{-2} the pattern is shown on figure 1-d ; the distorsion is incommensurate at all temperatures between 7 K and 300 K but the spots grow progressively to circular segments when cooling down the sample below 150 K. This loss of coherence strongly suggests the charge density wave to be composed of small domains in which the defects impose slightly different distorsions. A more precise explanation was achieved by slightly reorganising the configuration of disordered charge density wave by a short anneal at 400 K within the microscope. Figure 1-c was obtained with the sample of fig. 1-d after such a treatment. The circular segments break in two or three more or less defined spots. The main features of this pattern are explained in ref. 16 but it is clear, at the first glance, that two particular kind of domains have been favoured (they are twin variants of the same charge density wave configuration). Further irradiation makes the coherence length of the distorsion very small and there is no more temperature dependance of the pattern between 100 K and 400 K.

The present study is one of the most direct evidences of the existence of domains of charge density waves pinned to defects.

Physical Consequences of the Pinning : the Stabilization of the Metallic State in Charge Density Wave Insulators.

The structural results above mentionned clearly demonstrate that irradiation defects fix the phases of the charge density waves, prevent their low temperature ordering and disturb their commensurability when they would like to lock on the lattice.

A well known consequence of that on the transport properties of the charge density waves insulators is firstly to decrease the temperature of the metal to insulator phase transitions[1,9,20,26] and, with further irradiation to smear the transitions completely out of the resistance versus temperature curves[1,12]. Thus an irradiation at a constant temperature below the phase transition, in the insulating state produces an increase of the conductivity which reaches values of the order and even larger than the room temperature values in the metallic state. This maximum was observed

at 20 K in TMTSF-DMTCNQ, TTF-TCNQ, TSF-TCNQ (ref. 4 and 12), and TaS_2 (ref. 1). In TMTSF-DMTCNQ and in TTF-TCNQ, the transverse conductivity was shown to increase with irradiation too and to reach a maximum at approximately the same dose of 2.10^{-3} (ref. 5 and 12).

The conductivity measurements performed in situ during irradiations have demonstrated the existence of a highly conducting low temperature state corresponding to the full pinning of the charge density wave by defects. It was necessary to characterize a little better the electronic properties of this disordered phase. This was done by Fórro, Janossy and Zuppiroli in TMTSF-DMTCNQ (ref. 14). Fórro measured the Hall effect and thermoelectric power of pure and irradiated crystals. The main experimental results are plotted on figure 2 : In an irradiated sample of TMTSF-DMTCNQ containing 2.10^{-3} of damaged molecules, the Hall effect is small and temperature independent from 2 K to 300 K ; the thermoelectric power remains also small down to 4 K : the high temperature metallic state has been stabilized by a weak disorder. Further irradiation does not change very much the Hall coefficient and the thermopower.

Similar results were obtained by Mutka et al.[1] in irradiated TaS_2. Hall effect and conductivity measurements have demonstrated that a concentration of a few 10^{-3} defects produced by electron irradiation stabilizes the metallic state in this compound too.

Nevertheless, there is a significant difference in the appearance of the metallic state stabilized by disorder between 2D and 1D Peierls insulators : in 1T-TaS_2 there is a large range of defect concentrations where $d\rho/dT$ (the temperature coefficient of the resistivity) is positive at low temperatures[1], when in TMTSF-DMTCNQ, $d\rho/dT$ is never positive at low temperatures even in the concentrations range where Hall effect and thermoelectric power are very small. This is due to the blocking effect of defects in one dimension. The low temperature 1D metal stabilized by disorder is not a metal of infinite chains but a metal of segments bounded by defects. Electrons cannot escape these segments except by a hopping process[4] which dominates the conductivity even at concentrations of the order of 10^{-3}.

Of course, the blocking effect of defects is not effective in layered compounds. In two dimensions there are much more possibilities for the electron to escape the defect potential. A smooth cross-over between the metallic concentration range and an Anderson localization range has

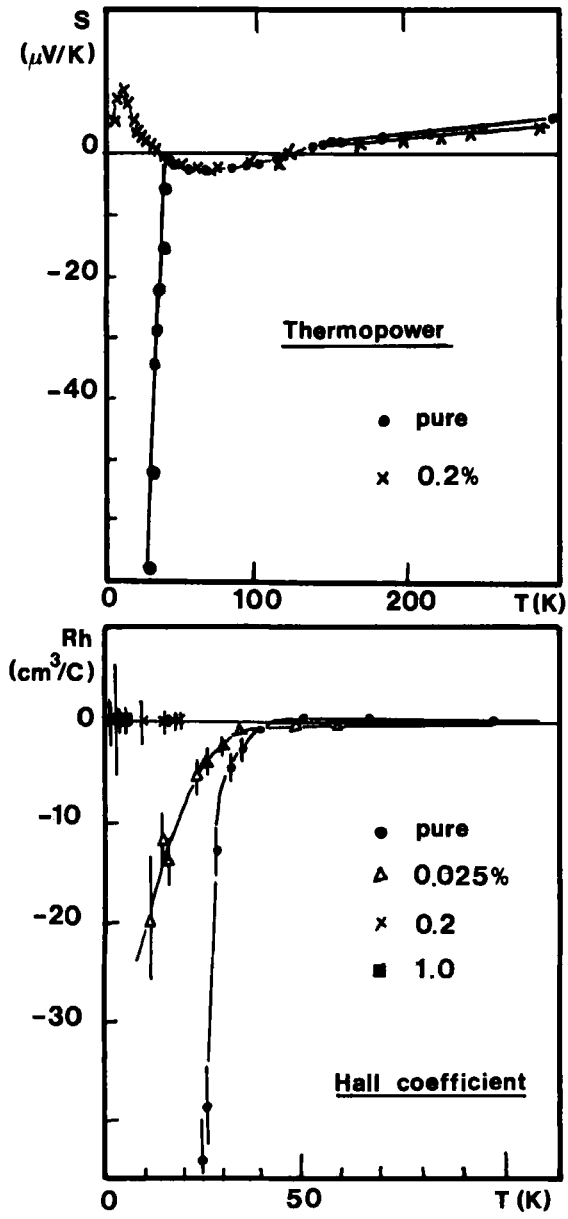


FIGURE 2 A weak disorder (2.10^{-3}) stabilizes the metallic state down to 2 K in TMTSF-DMTCNQ. Transport properties of pure and irradiated samples. From Forró et al.[14].

been observed in TaS_2 irradiated at low temperatures[1].

In monoclinic TaS_3 localization effects are so strong below the phase transitions, that there is no stabilization of the metallic state eventhough the charge density waves are fully pinned by irradiation defects and the phase transitions smeared out from the resistivity versus temperature curves[2]. In fact, TaS_3 is shown to be the first low dimensional conductor in which the pinning of the charge density waves produces no changes in the low temperature resistivity.

The Charge Density Wave Motion in Presence of Defects

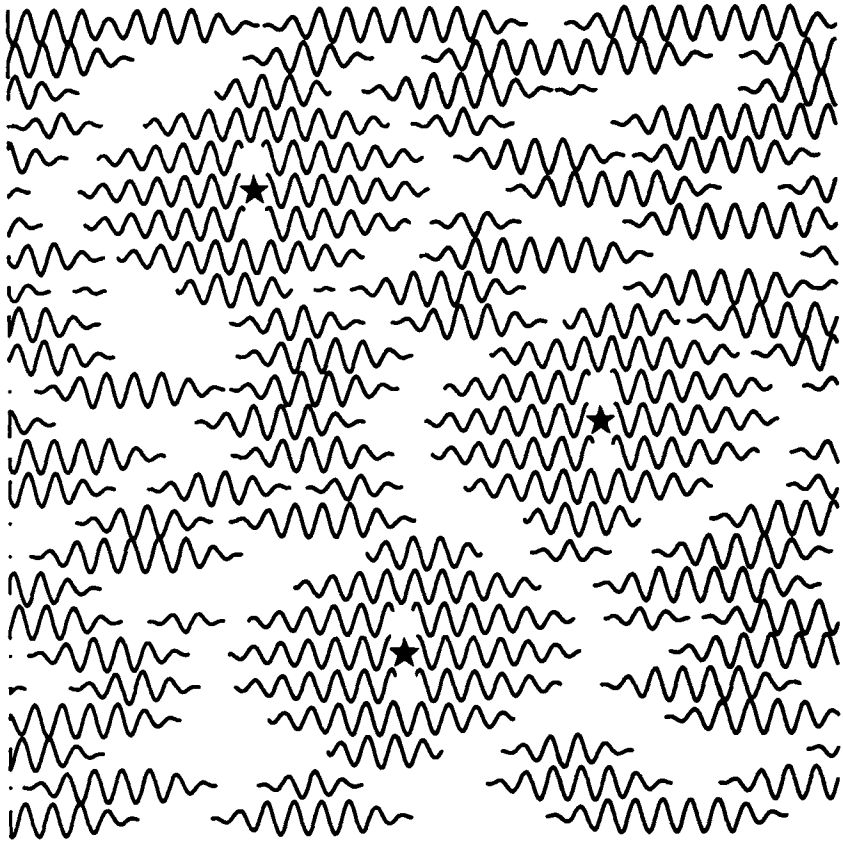


FIGURE 3 Schematic representation of the fluctuating, disordered charge density waves in presence of strong defects (TTF-TCNQ, 100 K).

When they are not pinned to the lattice, charge density waves can carry electronic current. More precisely, the electrons condensated below the Peierls gap can move coherently, together with the periodic lattice distorsion which opens the local gap. A collective contribution is added to the single electron conductivity. Because of the presence of this "gliding-gap" the scattering limiting the usual one-particule conductivity is strongly reduced as far as the condensated electrons are concerned (coherent phonon drag).

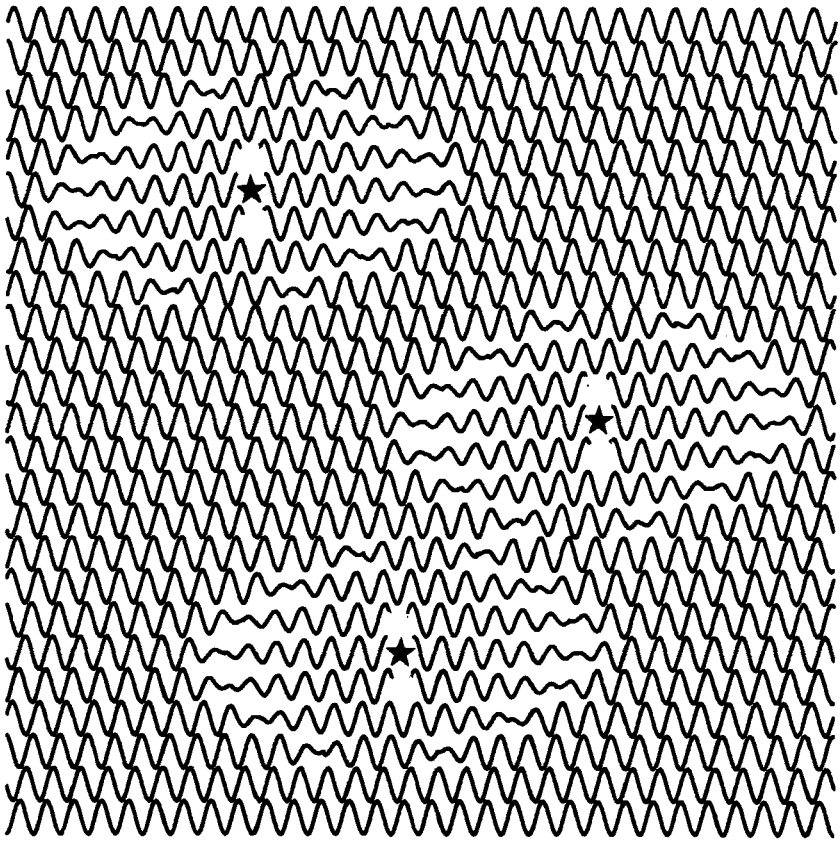


FIGURE 4 Schematic representation of the ordered charge density waves in presence of strong defects ($NbSe_3$, 50 K).

Evidences for an extraconductivity due to charge density waves have been reported[26,27] in two kind of systems represented schematically on figures 3 and 4. Figure 3 could represent TTF-TCNQ at 100 K where the collective conductivity is essentially related to 1D fluctuations[26]. The charge density waves do not feel the transverse interactions enough to be ordered ; in the "pure" sample the fluctuating conductivity is limited by the 1-D coherence length of the charge density waves. Figure 4 could represent $NbSe_3$ at 50 K where an extraconductivity due to charge density waves is observed in the "pure" sample when a low depinning field is applied[27]. It appears in a range of temperatures where the charge density waves are ordered but incommensurable with the lattice. In both cases strong pinning centres such as irradiation induced defects appear as rigid inclusions in the charge density waves system. They rigidly impose the phases at a non negligeable distance from their centre, along the chain as well as on several adjacent chains. They cannot be treated as point defects which pin the phase at a single point on a single chain.

The pinning of the charge density waves by defects in the metallic phase of TTF-TCNQ and the subsequent effect on the collective contribution to the conductivity has been recently studied by Bouffard, Chipaux, Jerome and Bechgaard[13]. They have demonstrated that a concentration of about 2.10^{-3} is enough to block all the fluctuating contribution due to charge density waves. This corresponds on the chain to one defect each 500 molecules. The coherence length of the charge density wave being significantly smaller than 500 molecular distances at any temperature in the metallic range (30 molecular distances at 60 K and 3 molecular distances at 150 K)[28], the results of Bouffard et al. confirm that pinning is not strictly one-dimensional. Their results suggest that the defect at 100 K is a rigid inclusion of 2 or 3 coherence lengths extension along the chains, interesting 10 chains approximately. In these conditions it is not surprising that the concentration of 2.10^{-3} that they found is very close to the concentration of defects of the same nature needed to destroy completely the charge density wave ordering in the insulating phase and to stabilize the metallic state.

In $NbSe_3$ and related compounds (fig. 4) the problem seems more complicated and the irradiation experimental results more controversial[17,29]. The picture of figure 4 strongly suggests the depinning problem by an electric field

in presence of strong defects to be similar to the problem of the plastic deformation of a metallic matrix in presence of inclusions harder than this matrix. This analogy replaces the electric field by a stress, the charge density wave current by a deformation and the threshold field by the yield stress for plastic deformation. A considerable litterature has been written on this subject[30] and this kind of tridimensionnal approach seems to us more appropriate than the one dimensional approach where independent charge density wave segments are considered to be depinned separately (see Portis, this conference). The present kind of intuition could also explain the curious metastable states of the charge density wave observed by Gill (this conference) which in the plasticity language could be replaced by relaxation or creep. Let us mention that Lee and Rice have already considered the possibility of motion of dislocations in the charge density wave lattice[25].

Two different irradiation experiments of $NbSe_3$ have been already published and they are in contradiction[17,29] : Fuller et al. have irradiated with 2.5 MeV protons and they found the threshold field to vary linearly with the defect concentration on more than one decade. Monceau et al. have irradiated with 3 MeV electrons and surprisingly they don't find any important pinning effect with similar concentration.

Charge Density Wave Insulators and Metals

TaS_3, 1T-TaS_2, TMTSF-DMTCNQ, TTF-TCNQ etc ... are low temperature charge-density wave insulators. But charge density wave onset, ordering or commensurability have not always the same drastic effects on the Fermi surface as in these compounds. $NbSe_3$, 2H-TaS_2, 2H-$NbSe_2$ etc... are well known to be low temperature metals and even superconductors. It is interesting to compare the pinning efficiency of irradiation defects in these two different types of systems. We present a preliminary experiment concerning a series of layered compounds very different from each other from the point of view of their transport properties (fig. 5). 2H-$NbSe_2$ and 2H-TaS_2 are low temperature metals and superconductors. The charge density wave anomalies are hardly ever visible on the resistance versus temperature curves. When irradiated with electrons or neutrons the resistance versus temperature curves change in agreement with the Mathiessen's rule

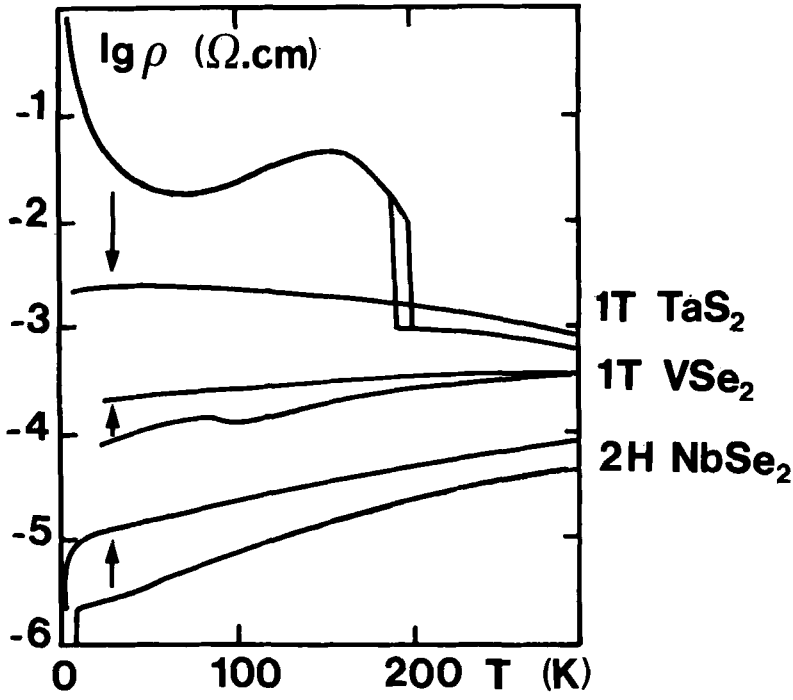


FIGURE 5 Resistivity versus temperature curves of 3 different layered compounds before and after an electron irradiation creating a concentration of defects of the order of 1%.

$$\rho(c,T) = \rho_{phonons}(0,T) + c\ \rho_{defects}$$

(please, notice that the scale on fig. 5 is logarithmic).

"Pure" 1T-TaS_2 has a resistivity which is at least one order of magnitude larger in the whole temperature range. It is a charge density wave insulator and defects stabilize the low temperature metallic state, as demonstrated previously.

1T-VSe_2 is an intermediate compound and it exibits an intermediate behaviour under irradiation. Defects are ten

times more efficient at low temperatures than at room temperature.

These coherent but preliminary results demonstrate that the consequences of pinning are different and depend on the strength of the charge density wave in the pure compound.

CONCLUSION

After a work of 4 years on irradiated low dimensional conductors the principal effects of strong defects on the metallic properties, electronic instabilities, fluctuations, and phase transitions are rather well understood. The concentration scales of defects in irradiated samples have been established carefully. Irradiation proved to be a good method for exploring disorder in low dimensional conductors and distinguishing between the genuine intrinsic properties and extrinsic ones due to the sample preparation.

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