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The anomalous growth of resistance fluctuations in orthorhombic TaS_3 below the liquid-nitrogen temperature

V Ya Pokrovskii†§, S V Zaitsev-Zotov‡, P Monceau† and F Ya Nad'†§
† Centre de Recherches sur les Très Basses Températures, CNRS, BP 166 X, 38042 Grenoble Cédex, France
‡ Institute of Radioengineering and Electronics, Russian Academy of Sciences, Mokhovaya 11, 103907 Moscow, Russia

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Abstract. It is found that, below 80 K, orthorhombic TaS_3 demonstrates spontaneous resistance fluctuations. The spectral density of the fluctuations grows as the temperature decreases. In the temperature range between 50 and 70 K the spectra reveal a mean relaxation time which decreases as the temperature decreases with an activation energy about 1300 K. We conclude that the main source of the resistance fluctuations are thermally assisted jumps of dislocations of the charge-density waves.

1. Introduction

The physical properties of quasi-one-dimensional conductors with sliding charge-density waves (CDWs), such as TaS_3 and $K_{0.3}MoO_3$, below approximately half the temperature of the Peierls transition reveal a number of new features, such as a reduction in the activation energy for the low-field conductivity, a dramatic increase in the threshold field E_T for the CDW depinning, and development of non-linear conductivity at $E < E_T$ [1]. The low-temperature properties of the typical quasi-one-dimensional conductor orthorhombic TaS_3 (o-TaS_3) have been studied in detail. A tremendous increase in anisotropy of the conductivity [2, 3], the disappearance of thermal hysteresis [4], and hopping conductivity [3, 5] were found for this compound in the low-temperature region. In general, these features are likely to indicate a new state of the CDW, probably highly disordered or glassy [6, 7]. However, the nature of the transition and the structure of the resulting state remain unclear.

It is well known that sliding CDWs generate both low-frequency noise of $1/f$ type, and narrow-band noise. At small electric fields $E \ll E_T$, CDW systems can also exhibit fluctuations in conductivity. In o-TaS_3, such fluctuations are found just below the Peierls-transition temperature $T_P \simeq 220$ K [8, 9]. In the vicinity of T_P, signs of thermally assisted depinning of the CDW are also observed in thin crystals of $NbSe_3$ [10, 11]. Lowering the temperature from the region of T_P freezes out these fluctuations.

At the same time, the behaviour of TaS_3 below liquid-nitrogen temperature is reminiscent of that in the vicinity of T_P. In fact, below 70–80 K a gradual collapse of the hysteresis loop of $R(T)$ is observed [4]; we also point out a weak irregularity in the logarithmic derivative of resistance around $T = 70$ K, which is sample dependent [12] but was demonstrated by all the samples studied in the present work. It could denote nucleation of a new state of the

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§ Permanent address: Institute of Radioengineering and Electronics, Russian Academy of Sciences, Mokhovaya 11, 103907 Moscow, Russia.

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CDW in this temperature region. One can expect these features to influence the resistance fluctuations.

The manifestations of the glassy properties of the CDW at low temperatures also gives us hope that we may be able to see resistance fluctuations at low temperatures, by analogy with spin glasses, where fluctuations of the magnetic moment are found to correlate with the frequency-dependent susceptibility. A study of such systems revealed peculiar relaxation times, and features of the transition into a glassy state [13]. The AC response of the CDW ($E \ll E_T$) has been studied in detail. In [14] the frequency-dependent conductivity revealed a characteristic time, whose temperature dependence above 80 K followed an $\exp(W/T)$ law, with $W = 850$ K corresponding to the Peierls gap Δ. A surprising correlation between this response of the pinned CDW and the low-frequency noise of the sliding CDW was observed [15]; the fluctuation–dissipation theorem was applied to explain this correlation. Below 70 K the average relaxation time was not obtained, as the dielectric strength of the relaxation was found to decrease strongly [14]. However, below 50 K, measurements of the dielectric constant yielded an average relaxation time which demonstrated a critical slowing down with decreasing T [16]. In principle, the same relaxation processes could be revealed as noise; activated by temperature, they could cause resistance fluctuations.

In the present paper we demonstrate that decreasing the temperature to below 75 K gives rise to spontaneous resistance fluctuations. Moreover, the spectral density of fluctuations increases by orders of magnitude on further cooling; the increase proceeds at least down to the lowest temperatures studied, $T = 30$–40 K. We show that the fluctuations can be explained by the thermally assisted climb of dislocations, which develops in the equilibrium state of the CDW in this temperature range.

2. Experimental technique

We have measured broad-band voltage fluctuations in samples of o-TaS_3 connected in series with a source of DC voltage and a loading resistor. Six samples with dimensions (0.4–1) mm × (1–20) μm^2 have been studied. The indium contacts were either cold soldered or vacuum deposited. Both two- and four-contact configurations were used. The amplified AC component of the voltage at the sample entered a spectrum analyser. The measurements have been performed in the frequency range 0.2–10^3 Hz. The upper boundary of the range was restricted by the cut-off frequency of the circuit, which was determined by the parasite capacitance of the wires (about 50 pF) and the lowest of either the sample resistance or the loading resistance. Usually, the value of the loading resistance was well above the resistance of the sample (current-controlled regime). At the lowest temperatures (two-contact measurements), the loading resistance was sometimes lower than the sample resistance; this permitted the cut-off frequency of the circuit to be increased. The corresponding correction of the noise amplitude was taken into account. Further extension of the frequency range was not necessary, as the value of the noise above 1 kHz due to the resistance fluctuations usually did not exceed the thermal (Johnson) noise. As for the lowest frequency, it was restricted by the measuring time necessary to average the noise power.

In general, two- and four-contact measurement gave qualitatively similar results; the difference between them will be discussed. To reduce possible non-equilibrium effects we performed measurements only at fixed temperatures and currents, starting at least 15 min after the temperature setting.

3. Results

Figure 1(*a*) presents typical dependences of the spectral density of noise $S_f(V)$ on the voltage V applied to the sample (later referred to as sample 1) at several given frequencies. The curves were obtained in the four-probe configuration. The abrupt growth of the noise at around $V = 200$ mV is associated with the onset of non-linear conductivity (figure 1(*b*)). The study of noise induced by the sliding CDW is beyond the scope of this paper. The initial part of the curve ($V \lesssim 200$ mV) indicates the voltage range at which $S_f(V)$ is proportional to V^2 (the appropriate slope is indicated by the broken line), i.e. the relative resistance fluctuations $S_f(\delta R/R) \equiv [S_f(V) - S_f(0)]/V^2$ are nearly independent of the electric field applied. This result demonstrates the existence of spontaneous resistance fluctuations of the pinned CDW, which could be measured at an electric field not exceeding a certain value (200 mV for this particular sample). The temperature evolution of these fluctuations is the central subject of the present paper. Below we shall present the noise in terms of relative fluctuations of resistance.

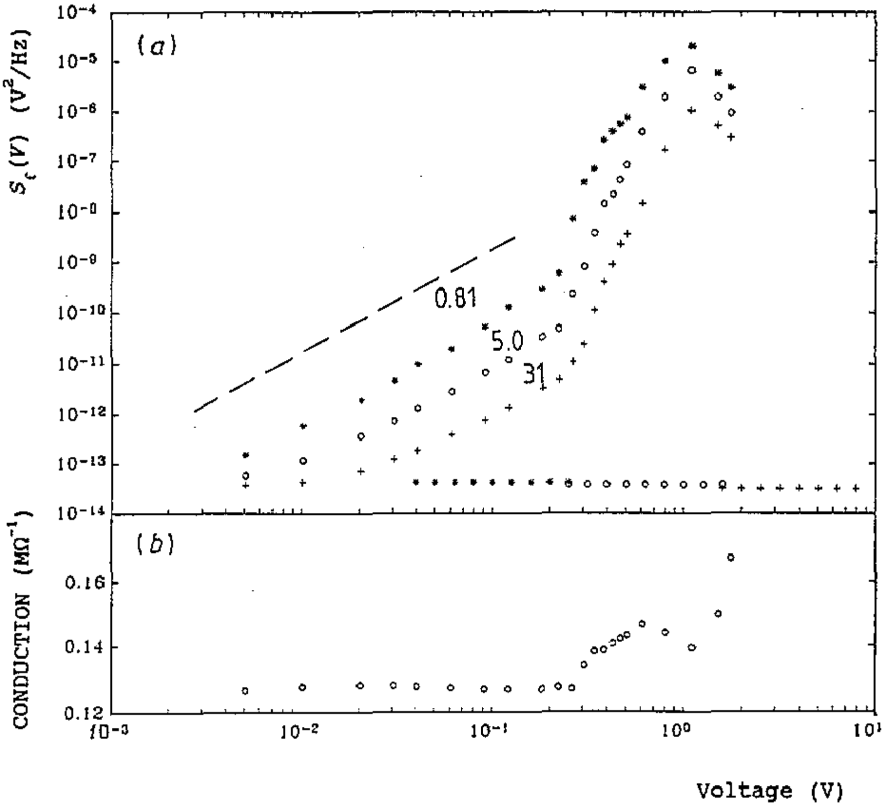

Figure 1. (*a*) Voltage dependence of the spectral density of noise at different average frequencies where the noise at zero voltage is shown in the right-hand part for each frequency with the corresponding symbol (sample 1: four-probe method; separation between the central contacts (deposited), 430 μm; cross sectional area, 4 μm^2; $T = 53$ K): ∗, 0.81 Hz; ○, 5.0 Hz; +, 31 Hz; – – –, slope $S_f(V) \propto V^2$. (*b*) Voltage dependence of conductivity, measured simultaneously.

Measurements in the two-probe configuration give analogous curves for this sample, although the influence of the electric field on fluctuations is observed at voltages above 35 mV, where the fluctuations are partly suppressed.

Now we consider the temperature dependence of the noise. Figure 2 shows typical temperature dependences of $S_f(\delta R/R)$ at a voltage $V \ll V_T$ for two samples: sample 1 (figure 2(*a*)) and sample 2 (figure 2(*b*)). We could perform measurements only at $T < 100$ K, because at higher T the noise associated with the resistance fluctuations did not exceed the background noise, unless V exceeded V_T. Each plot presents a pair of curves: four- and two-contact measurements performed at the same frequency in the same (central) sector of the sample.

Four- and two-contact curves demonstrate qualitatively analogous temperature dependences of the noise. This means that in both cases we deal with the noise of volume origin. Both curves indicate the growth of noise with decreasing temperature. Below $T = 75$ K the growth is more pronounced. The logarithmic derivatives of resistance displayed in the insets demonstrate minima at around $T \simeq 70$ K, in the region of the growth of the noise. However, the two-probe curves demonstrate more abrupt growth localized between 70 and 50 K. The form of these curves is dependent on the frequency at which $S_f(\delta R/R)$ is measured; at lower frequencies the region of abrupt growth is shifted to lower temperatures.

In the whole temperature range, the spectral density $S_f(\delta R/R)$ when measured by the four-contact method could be described by the $1/f^\alpha$ law with α around 1. The two-probe method gives, however, pronounced maxima in the frequency dependences of $S_f(\delta R/R)f$ between 70 and 50 K (figure 3).

Simultaneously with the spectral measurements which revealed the maxima, we have observed random switching between two levels on the time domains of voltage (random telegraph signal). The amplitude of the switching reached the value corresponding to $\delta R/R \simeq 10^{-3}$ at $T \simeq 50$ K. The average frequency approximately corresponded to the position of the appropriate maximum. This signal is peculiar for the thermally activated transitions between a pair of states. This type of noise was found to accompany the onset of the CDW motion [17]. In small samples of TaS_3 it was also observed at $E \ll E_T$ in the vicinity of T_P [9]. We emphasize that here we also deal with equilibrium noise rather than with a critical phenomenon; the maximum of $S_f(\delta R/R)f$ versus f revealed at $V = 40$ mV (figure 3), was also observed for V down to 10 mV [18]. The random switching of a two-level system is expected to have the Lorentzian spectrum

$$S_f(\delta R/R) \propto \tau_0/[1 + (f/f_0)^2] \tag{1}$$

where $\tau_0 \equiv 1/2\pi f_0$ is the average lifetime of the system in each state. In fact, the spectra around the maxima can be fitted by the Lorentzian form (figure 3, broken curve), although the maxima are somewhat wider. A better fit is achieved if one considers the noise as a superposition of Lorentzian spectra with distributed τ_0:

$$S_f \propto \int \mathrm{d}\tau_0 \frac{\rho(\tau_0)\tau_0}{[1 + (f/f_0)^2]} \tag{2}$$

where $\rho(\tau_0)$ is the distribution function of relaxation times. In figure 3 these fits are shown by the full curves. In this case the frequencies f_M corresponding to the maxima of $S_f(\delta R/R)f$ give the average value of f_0 [19].

It can be seen (figure 3) that f_M is temperature dependent. The inset in figure 3 shows the temperature dependences of f_M observed for the two samples (samples 1 and 2, both by the two-probe method). The dependences follow the activation law

$$\tau_0 = \tau_\alpha \exp(W/T) \tag{3}$$

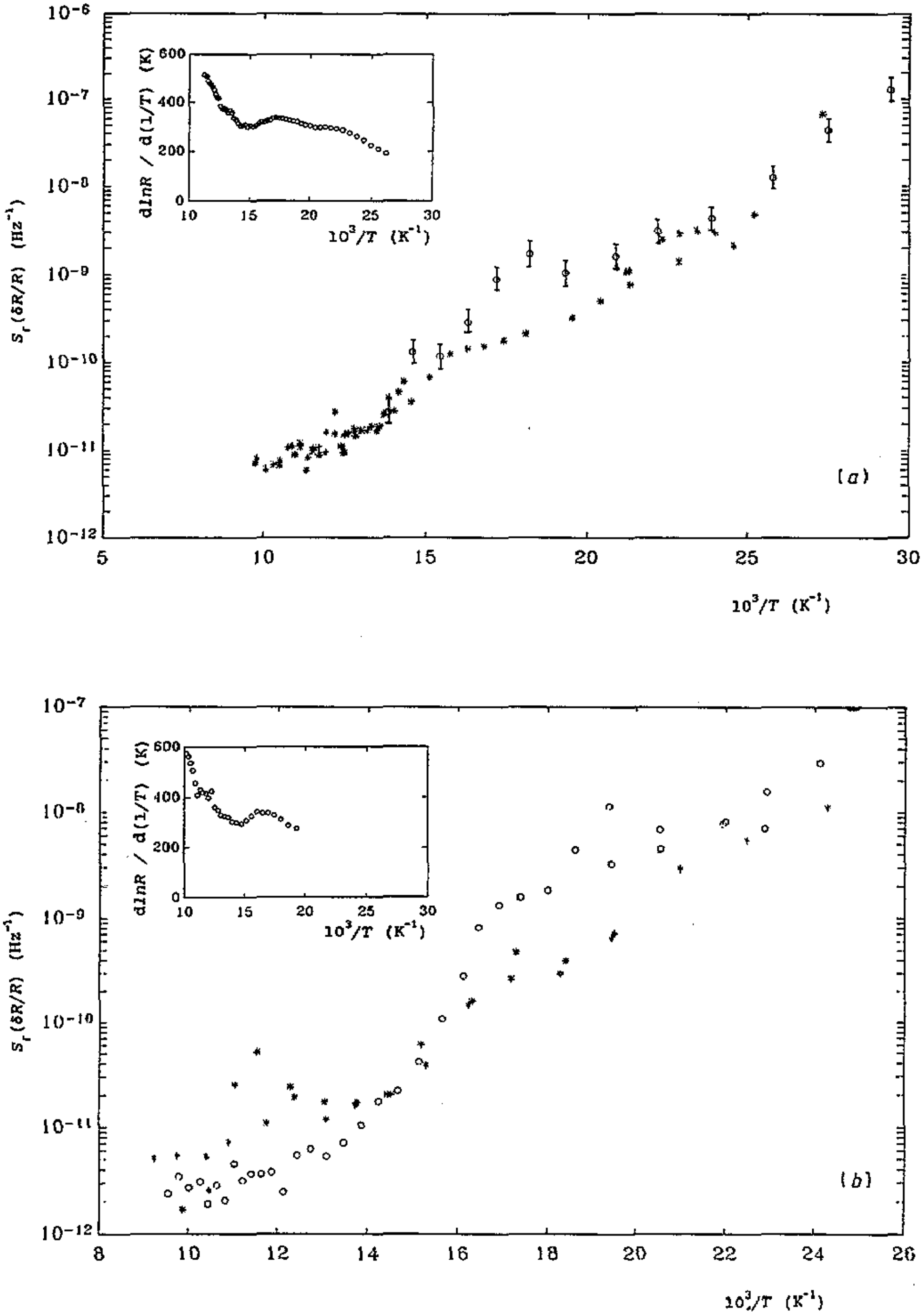

Figure 2. The dependence of the spectral density of resistance fluctuations versus the reverse temperature measured by the four-contact ($*$) and two-contact (○) methods (average frequency, 9 Hz): (*a*) sample 1; (*b*) sample 2 (separation between the central contacts (cold-soldered), 1.05 mm; cross section area, 14 μm^2). The insets show the logarithmic derivatives of the resistance: $\mathrm{d}(\ln R)/\mathrm{d}(1/T)$.

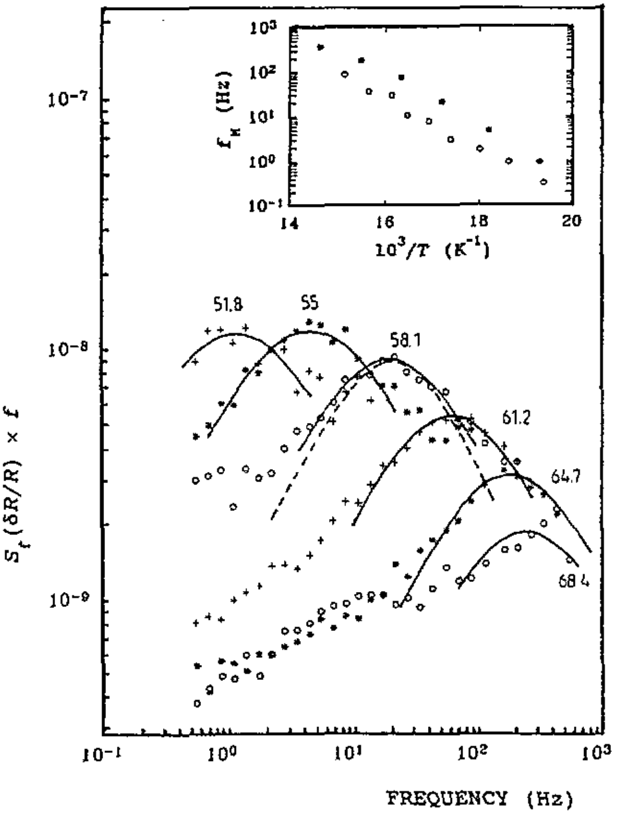

Figure 3. The noise spectra multiplied by f in the temperature range of the abrupt increase in the noise for various temperatures indicated on the plot (sample 1; two-contact method; $V = 40$ mV): – – –, Lorentzian spectrum; ——, superpositions of the Lorentzian spectra with $\rho(\ln \tau_0)$, distributed around the central relaxation time according to the Gaussian law, with the dispersion 0.65. The inset shows the frequencies f_M at which $S_f(\delta R/R)f$ reaches maxima versus the reverse temperature: $*$, sample 1; ○, sample 2.

with $W \simeq 1300$ K and $\tau_\alpha \simeq 10^{-13}$ s corresponding to a reasonable attempt frequency. It should be noted that we obtained approximately the same W for three samples with different kinds of contacts.

In the four-probe spectra, the maxima are very weak or are not observed at all. If the maximum is wide in comparison with the spectral 'window', i.e. τ_0 is distributed over decades, it is nearly impossible to observe it directly. Nevertheless, the temperature dependence of the average frequency of the fluctuations could be revealed. If a system has a temperature-dependent relaxation time, it is easy to see that the average slope α of $\log(S_f)$ versus $\log f$ measured in the spectral 'window' would change with temperature. This is really observed; as T is lowered from about 70 to about 50 K, α changes from less than unity to more than unity. Thus, from the four-contact measurements, we can also conclude that the average frequency of fluctuations decreases on cooling the sample.

In all samples we have observed an increase in the spectral power density of resistance fluctuations (at a given frequency) by 2.5 to four orders of magnitude as the temperature is reduced from 60–80 to 30–40 K, where $S_f(\delta R/R)$ attained about 10^{-7} Hz^{-1} at $f = 1$ Hz. The noise–temperature curves were sample dependent, as also were the $R(T)$ curves [3].

The higher the activation energy for $\ln R$ versus $1/T$, the more abrupt was the growth of noise, although we did not find a universal relation between R and $S_f(\delta R/R)$. In all cases, the relative increase in $S_f(\delta R/R)$ at a given frequency was higher than that of the resistance. We shall return to this in the discussion.

The temperature dependences discussed above were measured while the samples were subsequently cooled. Reversing the temperature variation did not affect the results qualitatively. Also, the spectra were reproduced after waiting for about 10 h. This shows that the fluctuations are stationary and are not associated with some slow relaxation process. However, the spectral density of the noise at frequencies below about 10 Hz for $T < 50$ K was somewhat lower, when the sample was subsequently heated. Similarly, a decay in $S_f(\delta R/R)$ at the lowest frequencies was observed at a fixed temperature when the measurements were repeated after about 10 h.

4. Discussion

To compare the value of the fluctuations with that in other materials demonstrating $1/f$ noise, one can use the semi-empirical Hooge relation [20, 21] which applies for semiconductors and metal films of different kinds:

$$S_f(\delta R/R) f = \gamma/N_c \tag{4}$$

where N_c is the total number of free current carriers in the specimen and γ is the Hooge constant which is around 2×10^{-3}. This relation could describe the increase in $1/f$-type noise with decrease in the concentration of current carriers. However, it gives the temperature dependence $S_f(\delta R/R) \propto R(T)$, which denotes a slower growth of noise than we have observed. Also, the predicted value of $S_f(\delta R/R)$ appears to be at least three orders of magnitude lower than observed. This discrepancy seems to indicate that the fluctuations are due to collective excitations rather than to single-electron excitations.

It is reasonable to suppose that the resistance fluctuations are caused by thermally assisted motion of domains of the CDW. One can expect that the character of this motion is different from that at higher temperatures. It is widely accepted now that at low temperatures the CDW turns into a disordered state [1]. The CDW is broken into parts by dislocations existing in the ground state in the volume. The underlying mechanism may be associated with a reduction in the screening of the pinning centres [16]; the effective growth of the CDW elastic modulus [22–24] can also result in the appearance of dislocations in the volume of the CDW [25, 26]. Thus, the dislocations develop because of the pinning, in contrast with the region around T_P [8, 9], where the nucleation of dislocations is thermally activated. The present experiment shows that at least above 50 K the dislocations of the CDW can move owing to the thermal fluctuations. The main source of the noise is thermally assisted jumps of these dislocations, proceeding through the phase slip (PS) process. We believe that at $T <$ 40–50 K the dislocations become more and more immobile, forming the glassy state [7, 16] and contributing to the inhomogeneous relief for hopping conductivity [5].

One can imagine two possible types of motion for the dislocations of the CDW: longitudinal glide, in which the total number of the CDW periods in the sample is conserved, and transverse climb of dislocations of the CDW provided that the PS acts at the chains crossed by the dislocation lines. The climb changes the total number of the wavelengths of the CDW. According to [27], the longitudinal creep is also accompanied by PS; so it is difficult to distinguish between the two kinds of motion.

Although the character of the motion of the dislocations remains unclear, we have some arguments in favour of the climb of dislocations as the source of noise. In the four-probe configuration, when the electric field is uniform between the potential contacts, the fluctuations are nearly independent of the electric field until it approaches E_T (see figure 1). At the same time, in the two-probe configuration the noise was affected by $V \gtrsim 30$ mV (only 15% of E_T). This corresponds to the climb of dislocations, which is governed by the strain of the CDW. The strain is not affected by the longitudinal electric field $E < E_T$. In the two-probe configuration the non-uniform electric field near the current terminals [28, 29] and, probably, the injection (or extinction) of free carriers by the terminals [30] affect the conditions of climb of the dislocations.

It should also be noted that the noise associated with the longitudinal jumps of the CDW could be related to the dielectric response $\sigma(\omega)$. Although such a relation is not universal, we can suppose that both σ and S_f would reveal the same relaxation times τ_0 characterizing the creep of the CDW excitations. The measurements of $\sigma(\omega)$ did not give a specific τ_0 for 50 K $< T <$ 70 K [14, 15]. The dielectric strength of relaxation [14] was found to disappear almost completely below 75 K. The relaxation times obtained (figure 3, inset) do not correspond to the extrapolation of the data either from the high [14, 15] or from the low [16] temperatures [31]. Although the reason for this discrepancy is not completely clear, we can suppose that, in the present experiment, another process is observed (probably climb of the dislocations), which is undetectable by measurements of $\sigma(\omega)$. The fluctuations have some common features with that observed in Mo_2S_3 [32]. It is not clear, however, whether this compound is a CDW system.

Let us consider a dislocation line crossing a CDW chain adding (or removing) two conduction electrons per chain. It is clear that such jumps would cause fluctuations in conductivity. These fluctuations have the same origin as the steps of R observed in [33], although the process of charge conversion may be more complicated below about 75 K [34]. In this case, one expects an increase in $S_f(\delta R/R)$ as the temperature decreases; the same PS acts, extracting (or consuming) two conducting electrons per chain, which causes a higher relative change in resistance at lower T.

Thus, the activation energy for $\tau_0(T)$ (equation (3)), 1300 K, characterizes the barrier for the climb of dislocations. This value is of the same order as the activation energy of the PS obtained at higher T [35], where it was estimated as the condensation energy per electron multiplied by the number of electrons in the amplitude-coherence volume. The difference from the measurements in [35] is that here we expect climb of dislocations which already exist in the random field induced by impurities, rather than their nucleation under electric field. However, this difference is not essential, if one considers the creep of a dislocation as the motion of agglomerated phase solitons [30].

Comparing the noise in the two- and four-contact configurations, one can see that the former has more 'coherent' features (random telegraph signal and well defined average relaxation times), which are evidently due to the synchronization of the PS acting around the current terminals [28–30].

Thus, two-contact measurements revealing the activation energy for the PS do not give the actual distribution of τ_0. We can estimate the distribution of τ_0 in the four-contact configuration from the temperature dependence of the slope of $S_f(\delta R/R)$ versus f. It is easy to show that the observed dependences $\alpha(T)$ would be observed (with $W = 1300$ K) if τ_0 is distributed over about four orders of magnitude around the average relaxation time.

In conclusion, we have found an increase in the low-field resistance fluctuations below 70–80 K and show that they are associated with thermally assisted jumps of equilibrium dislocations of the CDW. Their development in the sample volume is peculiar to the low-temperature region.

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