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Effects of charge-density-wave depinning on the low frequency shear compliance of $NbSe_3$

D. Staresinic[1,2], A. Borovac[1,2], K. Biljakovic[2], H. Berger[3], F. Levy[3], and J.W. Brill[1,a]

[1] Department of Physics and Astronomy, University of Kentucky, Lexington, KY 40506-0055, USA
[2] Institute of Physics, PO Box 304, 10001 Zagreb, Croatia
[3] Institute de Physique Appliquee, EPFL, 1015, Lausanne, Switzerland

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Abstract. We report on measurements of the voltage dependence of the complex shear compliance of two crystals of the charge-density-wave (CDW) conductor $NbSe_3$, for torsional frequencies between 1 Hz and 71 Hz. For both samples, there is a frequency independent, ∼1% increase of the magnitude of the compliance when the voltage exceeds the threshold for CDW depinning, but the internal friction has a striking sample dependence which we do not understand. For one sample, there is a frequency independent decrease in internal friction with CDW depinning, suggesting that the elastic changes are not relaxational and might reflect changes in the screening of the crystal strain by the CDW. For the second sample, the sign and magnitude of the change in internal friction is strongly frequency dependent, which we associate with a change in screening due to the finite electron diffusion time. The second sample also exhibits a frequency dependent peak in internal friction near threshold that may reflect relaxation of the CDW phase.

PACS. 71.45.Lr Charge-density-wave systems – 72.15.Nj Collective modes (*e.g.*, in one-dimensional conductors) – 62.30.+d Mechanical and elastic waves; vibrations – 62.40.+i Anelasticity, internal friction, stress relaxation, and mechanical resonances

1 Introduction

It is well known that the depinning and motion of charge-density-waves (CDWs) in quasi-one-dimensional conductors gives rise to a number of unusual electronic properties, including non-Ohmic conductivity, huge dielectric constants, and ac voltages produced by dc currents [1]. However, CDW interactions and depinning also affect the host lattice, changing the properties of both acoustic and optical phonons [2,3]. The most pronounced of these changes are reductions of low frequency elastic constants with CDW depinning [3–16].

These changes were originally observed in vibrating reed measurements of Young's modulus (Y) of TaS_3 [4,5], $NbSe_3$ [4,6], and $(TaSe_4)_2I$ [4,7], in which it was found that the flexural resonant frequency ($\propto Y^{1/2}$) decreases when the voltage applied to the sample exceeds the threshold (V_T) for CDW depinning, while the width of the resonance (proportional to the internal friction) increases for $V > V_T$. For TaS_3, the best studied material, the changes in Young's modulus were found to decrease with resonant frequency as $\omega^{-3/4}$ for frequencies above 1 kHz [8], suggesting that the elastic changes were anelastic, *i.e.* caused by the relaxation of internal properties with changing strain [5,17].

Mozurkewich proposed a phenomenological model in which the relaxing property is the local CDW phase, whose average relaxation time is "infinite" when the CDW is pinned but becomes finite for $V > V_T$ [9]; there is a dynamic glass-like transition at V_T, below which disorder in the CDW phase freezes in [18]. Mozurkewich found that if the strain dependent property is the CDW amplitude or stiffness, then the changes in elastic constants would be small and sample dependent (proportional to the threshold electric field), but if the strain dependent property is the CDW wavelength, then the changes in elastic constants would be large and sample independent, as was observed for TaS_3 [9,10]. Measurements of torsional resonances in TaS_3 revealed that the relative change in shear modulus (G) was an order of magnitude larger than that in Y, suggesting that the CDW wavelength was strongly dependent on transverse strain [11].

To study the voltage dependence of the average relaxation time, we studied the frequency and voltage dependence of the complex shear compliance J ($|J| \equiv 1/G \equiv J$) of TaS_3 at very low frequencies (*i.e.* below the frequencies of its mechanical resonances) [12]. For a general linear relaxation with a distribution of relaxation times $= A(\tau, V)$, the complex compliance can be expressed as an integral over Debye modes with relaxation times τ [17]:

$$J(\omega, V)/J_U = 1 + \int \mathrm{d}\ln\tau A(\tau, V)/(1 + \mathrm{i}\omega\tau), \qquad (1)$$

where J_U is the unrelaxed (*i.e.* $\omega = \infty$) compliance, and the internal friction $= \tan\delta \equiv \mathrm{Im}(J)/\mathrm{Re}(J)$, where δ is the

[a] e-mail: jwbrill@uky.edu

phase shift between the applied stress and the measured strain. We fit J to a "Havriliak-Negami" [20] distribution of relaxation times, assuming that the normalized shape of the distribution was independent of voltage, and found that the peak relaxation time diverged at threshold as $\tau_0 \propto (V/V_T - 1)^{-3}$ [12,13]. However, the total relative relaxation strength, $\int d \ln \tau A(\tau, V)$, was found to vanish near threshold in our fits, in apparent contradiction to the Mozurkewich model [9]. We suggested [12] that this was a consequence of our assumption of a constant shape of $A(\tau, V)$ and that near threshold there may actually be a large increase in relative weight for $\tau > \tau_0$. Similar changes in apparent relaxation strength have been observed for $(TaSe_4)_2I$ [14].

$NbSe_3$'s elastic anomalies, measured with flexural and torsional resonances, were found to have some interesting differences with those of TaS_3. In both materials, the Young's modulus anomalies decrease at high flexural frequencies as $\omega^{-3/4}$ [8], shear modulus anomalies are an order of magnitude larger than the Young's modulus anomalies [6], and the elastic anomalies depend in similar ways on the "depinning" frequency when the CDW is depinned with an ac rather than dc voltage [15]. Also, similar elastic "interference effects" are observed in both materials if ac and dc voltages are simultaneously applied to the sample [16]. However, the elastic anomalies are much smaller for $NbSe_3$ than for TaS_3, seeming (within considerable scatter) to be proportional to the threshold field [6], suggesting that the CDW amplitude or stiffness, and not wavelength, is the dominant strain dependent quantity [9]. Also, for some elastic constants, the internal friction was observed to decrease rather than increase with depinning, in some cases after going through a maximum near V_T [6], suggesting that the pinned relaxation time is finite. If so, the elastic anomalies would be expected to be very frequency dependent at low frequencies. This possibility motivated the present experiments, to measure the low-frequency shear compliance of $NbSe_3$.

2 Experimental techniques

The techniques used were similar to those we used for TaS_3 [12]. The sample, typically a thin crystal $\sim 10 \times 0.05 \times 0.01$ mm^3 was suspended between two current contacts, to which it was glued with silver paint, with a free, suspended length of $\sim$4 mm. A thin ($\sim$60 μm diameter, 1 mm long) magnetized steel wire was glued to the center of the crystal with conducting epoxy. Oscillating torque was applied to the sample by an alternating magnetic field ($\sim$10 G) provided by Helmholtz coils; twist angles were kept small enough (typically $\sim 1^\circ$) so that the strains (*i.e.* twist angles) were always proportional to the torques. The oscillation frequency was kept a few times less than that of the torsional resonance of the sample (typically 100$-$500 Hz).

The strains were measured by locating the sample inside a helical resonator cavity [21] (resonant frequency $\approx$450 MHz, quality factor $\approx$300), with the end of the steel wire near ($\sim$0.1 mm) the tip of the helix. The cavity was driven with a resonant RF signal; as the sample twisted, it phase modulated the output of the cavity, which was measured with a two phase lock-in operating in a magnitude ($\propto J$) / phase ($\propto \delta$) mode [12,21].

Measurements of the elastic changes were made at 102 K, in the $NbSe_3$'s upper CDW state (with transition temperature $T_1 = 142$ K [1]); the complex compliance at a given frequency was measured as the voltage across the sample was decreased. Because these were 2-probe measurements, the measured resistances include the contributions of leads and contacts (a few ohms). Unlike semiconducting TaS_3, $NbSe_3$ remains metallic in its CDW states [1] and Joule heating can be significant for voltages near threshold [6]. To minimize Joule heating, samples were kept in $\sim$1/2 atm. helium gas. Even so, Joule heating changed the temperature of samples by $\leq$3 K during measurements. This was noticed in measurements of the sample resistance (*e.g.* see Fig. 2a), but for most samples had negligible effect on the elastic properties (*e.g.* no changes were observed below threshold).

Nonetheless, the presence of the helium atmosphere had a serious effect on the measurements: it caused a drift in the quality factor of the cavity as the level of the surrounding liquid nitrogen bath changed, even though a vacuum space separated the helical resonator from the bath. While the changing quality factor did not noticeably affect the phase shift of the modulation signal, it caused its magnitude to drift by up to 0.5%. Because of these cavity drifts, measurements were not attempted for oscillation frequencies $\omega/2\pi < 1$ Hz. For the best cases, the short term resolution of the experiment was $\partial J/J \sim 0.1\%$ and $\partial\delta \sim 0.03^\circ$; these values are, of course, much worse than for the torsional resonance experiment [6].

3 Results and discussion

Measurements were attempted on several nominally pure samples from the same growth batch prepared at EPFL, Lausanne. Results are shown in Figures 1 and 2 for the two samples with the least noise and cleanest anomalies. For other samples, the noise levels ($\partial J/J$ and $\partial\delta$) were comparable to the anomalies ($\Delta J/J$ and $\Delta\delta$) and/or there was excessive Joule heating, but the results for the cleanest of these samples were similar to those of sample A (Fig. 1).

The upper panel of Figure 1 shows the voltage dependence of the resistance (V/I) of sample A, and the lower two panels show the changes in magnitude and phase of its compliance at several frequencies. The changes in compliance are defined with respect to the values measured at $V = 0$. For the set of runs shown, the voltage points are widely separated to minimize the drift in the compliance data.

Within the scatter in the data ($\partial J/J \sim \pm 0.1\%$ and $\partial \tan\delta \sim \pm 3 \times 10^{-4}$), the anomalies in compliance ($\Delta J/J \approx 0.6\%$ and $\Delta \tan\delta \approx -1 \times 10^{-3}$) are independent of frequency for this sample, and appear (*within the voltage resolution of these runs*) as "steps" at the elastic threshold voltage $V_T \approx 0.2$ V, corresponding to a threshold electric field of $\approx$0.5 V/cm. Actually, in denser runs we also observed a sharp (apparently frequency independent)

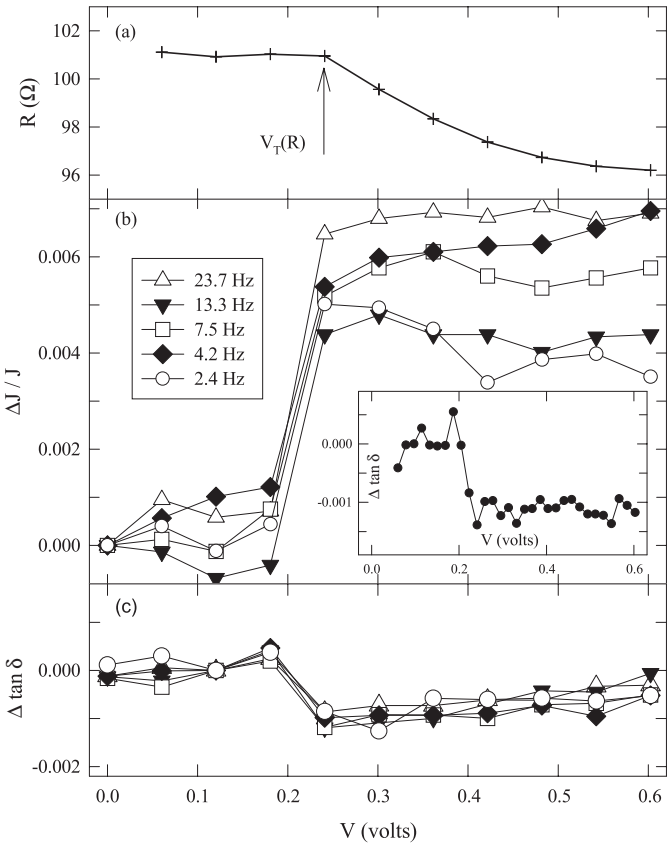

Fig. 1. Voltage dependence of the (a) resistance, (b) change in the magnitude of the shear compliance, and (c) change in internal friction for *sample A* at 102 K. J and $\tan\delta$ are measured at the torsional frequencies indicated in the legend. The vertical arrow in (a) shows the resistive threshold voltage. The lines are guides to the eye, with the scatter in the data indicative of the noise level. The inset shows the internal friction at 13 Hz measured for a denser set of voltages.

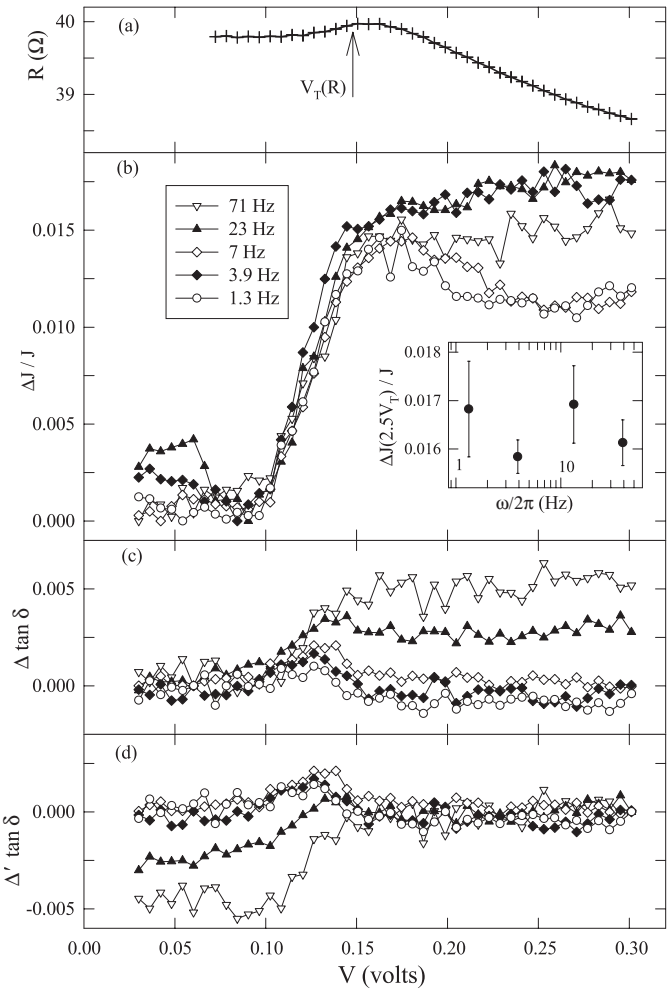

Fig. 2. Voltage dependence of the (a) resistance, (b) change in the magnitude of the shear compliance, and (c, d) change in internal friction for *sample B* at 102 K. J and $\tan\delta$ are measured at the torsional frequencies indicated in the legend. For (c), the changes in internal friction are defined with respect to their $V = 0$ values while in (d) they are defined with respect to their values at 300 mV. The vertical arrow in (a) shows the resistive threshold voltage. The lines are guides to the eye, with the scatter in the data indicative of the *short term* noise level. However, a slow drift also affected the magnitude of the compliance, as discussed in the text, and the inset to (b) shows the frequency dependence of the change in compliance between $V = 240$ mV($= 2.5V_{\rm T}$) and $V = 0$ when measured quickly.

peak in the internal friction at threshold, as shown in the inset. As previously noted [6], the elastic threshold voltage is less than the resistive threshold voltage, $V_{\rm T}(R)$, shown by a vertical arrow in the figure, suggesting that depinning occurs in the bulk of the sample at a lower electric field than it does at the current contacts. In fact, the anomalies for sample A are similar in shape and size to those of sample no. 2 of reference [6] measured at its ∼100 Hz resonance. For the present case, the frequency independence of the results indicates that the anomalies are *not relaxational* in origin, but are intrinsic to the depinned CDW. A possible mechanism is discussed below.

Results for sample B are shown in Figure 2; for these runs, the voltage points are more closely spaced. The increase in resistance (Fig. 2a) below the resistive threshold ($V_{\rm T}(R) \approx 150$ mV) indicates that there is Joule heating, as discussed above. As for sample A, the elastic threshold is below the resistive threshold. The apparent frequency dependence of the compliance for $V > 150$ mV shown in Figure 2b is a consequence of the drift in electronics discussed above; the results of an experiment in which the change in compliance was measured when the voltage was switched from 240 mV to zero are shown in the inset to the figure: at all frequencies, $\Delta J/J \approx (1.7 \pm 0.1)\%$. This value is larger than that of any of the samples of reference [6].

Unlike the change in the magnitude of the compliance, the change in phase shift is frequency dependent. Figure 2c shows the change in internal friction at each frequency, again referenced to its value at $V = 0$; *i.e.* $\Delta \tan\delta \equiv \tan\delta(V) - \tan\delta(0)$. The low frequency internal friction behaves similarly to that for sample A; *i.e.* it decreases by $\sim 1 \times 10^{-3}$ when the CDW becomes depinned, although for sample B the peak near threshold is more pronounced, *e.g.*

similar to that of sample no. 1 of reference [6]. However, for high frequencies the internal friction increases with depinning, similar to the internal friction associated with Young's modulus [6].

As represented in Figure 2c, $\tan\delta$ exhibits considerable dispersion for $V > 150$ mV; the internal friction increases with increasing frequency (by 6×10^{-3}), suggesting that the average relaxation time $\tau_{\text{AVERAGE}} \ll 1/\omega_{\text{MAXIMUM}} \approx 2$ ms at these voltages. However, since our measurements cannot determine the absolute phase shift at any frequency more precisely than $\sim 1^\circ$ [13], our assumption that the internal friction is independent of frequency below threshold may not be correct. In Figure 2d, we plot the voltage/frequency dependence of the change in internal friction referenced to its high voltage value, $\Delta' \tan\delta \equiv \tan\delta(V) - \tan\delta(300\,\text{mV})$; in this representation the dispersion occurs for voltages below threshold, with $\tan\delta$ increasing and saturating with decreasing frequency, suggesting that below threshold $\tau_{\text{AVERAGE}} \sim 40$ ms.

However, for either case, it is difficult to reconcile the apparent frequency independence of J with this large dispersion in damping. For relaxational behavior (*i.e.* Eq. (1)), the two quantities are related by the Kramers-Kronig relationship [17]:

$$(J_{\text{R}} - J_{\text{U}})/J_{\text{U}} = (2/\pi) \int \tan\delta(\omega')\, \text{d} \ln \omega', \qquad (2)$$

where J_{R} is the relaxed (*i.e.* $\omega = 0$) compliance, the integral extends over all ω', and we assume that the total relative relaxation strength $\int \text{d} \ln \tau A(\tau, V) \ll 1$. Therefore, in some voltage range , the magnitude of the compliance would be expected to have dispersion: $[J(\omega_{\text{HIGH}}) - J(\omega_{\text{LOW}})]/J > 4 \times 10^{-3}$, in contradiction to our observation. Reflection shows that the voltage/frequency dependences of $\Delta J/J$ and $\Delta \tan\delta$ (as plotted in either Fig. 2c or d) are inconsistent with equation (1) even if one allows the shape, width, or integral of A to vary with voltage.

In addition to this unusual dispersion in damping, which occurs over a wide voltage range, the peak near threshold is also frequency dependent. The magnitude of this peak is sufficiently small ($\Delta \tan\delta \sim 1 \times 10^{-3}$) that resulting dispersion in J, in a relaxational model, would be comparable to its noise. In Figure 3, we plot the frequency dependence of the peak voltage, normalized to threshold (taking $V_{\text{T}} = 95 \pm 5$ mV). Associating this internal friction peak with a process with a relaxation time $\tau_0(V_{\text{PEAK}}) = 1/\omega$, we see that $\tau_0 \propto (V/V_{\text{T}} - 1)^{-4}$, similar to the divergent time constants found for TaS_3 and $(TaSe_4)_2I$ [12–14]. We therefore associate this loss peak with CDW phase relaxation [9]; it is presumably responsible for only part of the increase in J, as discussed below. (The internal friction peak for sample A may have a similar origin, but its apparent frequency independence would imply a much stronger power law.) The much smaller loss peaks in $NbSe_3$, as compared to TaS_3 [12] and $(TaSe_4)_2I$ [14], suggests that its CDW properties are much less strain dependent [9].

We now consider non-relaxational mechanisms for the elastic anomaly. Several mechanisms by which the lattice intrinsically softens with CDW depinning have been proposed [16,22–25], but the only one in which the depinning is accompanied by a decrease in damping is that discussed by Virosztek and Maki (VM) [25]. In the VM model, the lattice is stiffer when the CDW is pinned than depinned because the depinned CDW can contribute to the screening of phonons whereas the pinned CDW cannot. In addition to this frequency independent softening, proportional to the electron-phonon coupling constant, there are changes in damping: the internal friction increases with depinning if the phonon frequency $\omega \gg \omega_{\text{D}} \equiv Dq^2$ and decreases if $\omega \ll \omega_{\text{D}}$, where q is the phonon wavevector and D is the electron diffusion constant, so that ω_{D}^{-1} is the time for electrons to diffuse distance q^{-1} [25].

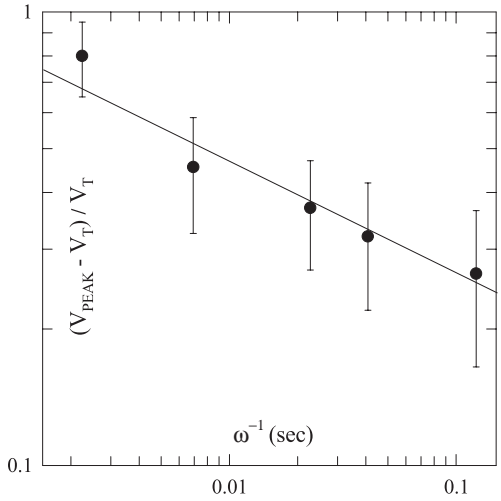

Fig. 3. Frequency dependence of the voltage (V_{PEAK}) at which internal friction peaks are observed (normalized to the elastic threshold voltage, $V_{\text{T}} = 95 \pm 5$ mV) in *sample B*. The reference line has slope $= -1/4$.

The VM model was derived for propagating phonons. For an acoustic phonon with velocity $v \approx \omega/q$, the hydrodynamic (*i.e.* $\omega \ll \omega_{\text{D}}$) regime corresponds to $\omega \gg v^2/D$. Taking $D \sim k_{\text{B}} T\sigma/(ne^2)$, where σ is the normal conductivity ($\sim 5000\ \Omega^{-1}\,\text{cm}^{-1}$ at 102 K [1]) n is the electron density ($\sim 4 \times 10^{21}$ cm^{-3} [26,27]), and $v \sim 10^5$ cm/s for "soft" shear modes [6,11], the hydrodynamic regime corresponds to $\omega/2\pi \gg 200$ MHz. However, for our forced oscillations, the situation is very different: for all frequencies, the effective wavevector $q \sim \pi/L \sim 8$ cm^{-1}, where L is the length of the crystal, so that the hydrodynamic limit corresponds to $\omega/2\pi \ll \omega_{\text{D}}/2\pi \sim 1$ Hz!

In view of the complexity of the band structure [26,27], it is certainly possible that the effective diffusion constant, and therefore ω_{D}, may be more than an order of magnitude larger than this estimate. We therefore suggest that VM screening anomalies dominate the low frequency shear modulus response for both samples. The frequency independent results for sample A suggest that we are in the low frequency limit for all measured frequencies, while the frequency dependent results for sample B suggest that for this sample $\omega_{\text{D}}/2\pi \sim 20$ Hz.

We note that in subsequent work in which the effects of long-range Coulomb interactions were included, VM [28] showed that these screening anomalies would only be observed for transverse waves propagating perpendicular to the quasi-one-dimensional conducting chains (**b** for $NbSe_3$), making their relevance to our experiments questionable. However, the relatively strong interchain interactions [1,27] in $NbSe_3$ might allow screening anomalies to be observed in our experiments. Furthermore, because of the difficulty in controlling precisely how the magnetic wires are glued to the sample, $G = 1/J$ may not only be an undetermined combination of the stiffness constants C_{44} and C_{66} [11], but include contributions from other shear moduli as well.

In fact, the latter effects may account for the sample dependence of the shear anomalies in $NbSe_3$; however, it is not clear why the diffusion time, ω_D^{-1}, should also be sample dependent. We note that not only were samples A and B of the same length (and therefore should have the same effective q), but they also had lengths close to those of reference [6], which all seem to be in the hydrodynamic limit at much higher frequencies. It is also not clear why the frequency dependence of the damping peak near threshold is so sample dependent. It is possible that the CDW depinning anomalies are affected by the transverse dimensions of the samples, reflected in their resistances, or "uncontrolled" properties, such as strains or crystal defects introduced by the glues at the contacts and/or magnetic wires. Also, as for TaS_3, it is not at all clear how to connect the present results with the high frequency Young's modulus results of reference [8].

As discussed above, our previous results on TaS_3 certainly suggest that its elastic anomalies are predominantly relaxational; however, they may also have small "screening contributions" which affected the fits of reference [12]. Note that TaS_3 and $NbSe_3$ have similar electron mobilities, and therefore diffusion constants [1].

In summary, our examination of changes in the low frequency shear compliance of $NbSe_3$ with CDW depinning have found different behaviors for two crystals from the same growth batch. For both samples, the magnitude of the compliance increases by $\sim$1% with CDW depinning, and this increase is independent of frequency. However, for one sample, the internal friction has a frequency independent decrease when the CDW becomes depinned, while for the other the sign and magnitude of the internal friction change are frequency dependent. We suggest that these anomalies are due to changes in the screening of the elastic deformation by the CDW, as proposed by Virosztek and Maki [25]. Within the context of this model, the sample dependence would be caused by a difference in the electron diffusion time, which we do not understand; however, "extrinsic" (*e.g.* contact or size) effects may play a role. The low frequency elastic properties of $NbSe_3$ differ from those of TaS_3, in which the shear compliance anomalies appear to be predominantly relaxational, with a relaxation time which diverges at threshold [12]. However, the second $NbSe_3$ sample does exhibit a frequency dependent peak in damping near threshold, which might be associated with relaxation of the CDW phase, similar to the behavior of TaS_3.

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