

# Commensurability effect and lock-in transition in Mo/Si superconducting superlattices

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We report the first observation of the lock-in transition in artificial superconducting superlattices, which takes place in tilted magnetic fields. The measurements were carried out on the Mo/Si layered system. The temperature dependence of the critical angle for the trapping of the vortices in the orientation parallel to the layer planes is determined by the previously known resistive method and by a new method based on the effect of commensurability between the intervortex distance and the superlattice wavelength. The temperature dependences of the critical angle obtained by the two methods practically coincide. The experimental results are consistent with the theoretical predictions of Feinberg and Villard.

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## Introduction

Many layered superconductors (high- $T_c$  oxides, artificial superlattices consisting of high-temperature compounds or conventional superconductors, intercalated dichalcogenides, etc.) have an inherently large anisotropy of the physical parameters. It is known that such an anisotropy plays a very important role, giving rise to many new phenomena, such as the dimensional crossover, fluctuation-induced decoupling and melting of the vortex lattice (VL), intrinsic pinning, lock-in transition, and so on. The static and dynamic properties of the vortex matter in the solid phase differ essentially from those in the homogeneous type II superconductors. According to the results of Ref. 1, the unit cell of the VL should be strongly distorted compared to the equilateral triangle that is characteristic for the Abrikosov VL. The VL parameters should depend intrinsically on the anisotropy coefficient  $\gamma = (M/m)^{1/2}$  and on the angle between the applied magnetic field  $\mathbf{H}$  and the anisotropy axis. Here  $M$  is an effective mass along the normal to the layer planes, and  $m$  is the in-plane mass. The influence of the anisotropy on the layered superconductor properties is revealed most dramatically in a parallel field and in a range of angles which are close to  $\theta = 0^\circ$  ( $\theta = 0^\circ$  for  $\mathbf{H}$  parallel to the layers). In particular, for parallel magnetic fields the effect of

the commensurability between the intervortex distance in the VL and the layered structure period  $s$  leads to oscillations of the critical current, resistivity, and magnetization [2–6]. The theory of the matching effects for this situation was developed in Refs. 7–11. Of the especial interest is the situation of strong layering considered in Ref. 10. It was shown that, when the intrinsic pinning energy  $E_p$  exceeds the elastic energy of VL shear deformation  $E_{el}$ , the vortices cannot cross the layers, and the period  $Z_0$  of the VL in the direction orthogonal to the layers is fixed, and it is determined by the initial conditions under which the vortex lattice was formed. This means that the VL should always be commensurate with the period  $s$  of the underlying pinning potential connected with the layered structure. In this case only the values  $Z_0 = Ns$  ( $N$  is an integer) are allowed;  $Z_0$  remains constant over a wide range of applied field intensities, while the unit cell area of the vortex lattice varies with the field only on account of flux line displacements along the layers. In the framework of the Lawrence–Doniach approach, for relatively high magnetic fields a sequence of first-order phase transitions between the vortex lattices with different orders of commensurability  $N$  is predicted [11].

Oscillatory dependence of the critical current  $I_c$  on the parallel magnetic field owing to the commensurability

surability effect has been observed on several kinds of multilayers: Nb/Ta [3], Nb/Pd [4] and Mo/Si [5]. It was shown that the  $I_c$  oscillations are accompanied by resistivity oscillations, and all features of the  $R$  vs.  $H$  and  $I_c$  vs.  $H$  curves correlate [5]. However, at low temperatures the zero-resistance regions that manifest the reentrance of superconductivity [5] appear instead of the resistance minima. The majority of the features of the nonmonotonic and reentrant behavior may be explained quantitatively in terms of the Ivlev–Kopnin–Pokrovskii theory [10]. The positions of the resistance minima and zero-resistance regions correspond to the stable states of the commensurate vortex lattices [5].

At temperatures close to the transition temperature  $T_c$  not all of the above-mentioned effects have been observed, because the intrinsic pinning [12], which creates large barriers for the transverse motion of the flux lines and gives rise to an effective locking of the vortices between the superconducting layers, becomes strong at temperatures sufficiently low that the condition  $\xi_{\perp}(T) \leq s$  holds [12,13]. Here  $\xi_{\perp}$  is the coherence length in the direction orthogonal to the layer planes.

It has been shown [14] that the intrinsic pinning causes another interesting phenomenon, namely a lock-in transition. Due to the anisotropy, at relatively small tilting angles the confinement of the vortices parallel to the layer planes becomes energetically more favorable than the creation of tilted vortices. The manner of the flux penetration in the oblique fields is modified at angles which are close to  $\theta = 0^\circ$ . Experimentally the lock-in transition is observed by several different methods. Among them there are the measurements of the microwave dissipation at different orientations of  $\mathbf{H}$  [15]; the lock-in transition is identified by the change in the dissipation mechanisms, which are controlled by the parallel and perpendicular components of the dc magnetic field, respectively. Evidence about the lock-in transition may be obtained from torque experiments [16–18] and from ac magnetic susceptibility measurements [19], from magnetization [20,21], and from resistive measurements [22]. A comparative analysis of all the methods used for determination of the lock-in transition is presented in Ref. 21. All these investigations have been performed on single crystals of high temperature oxides and organic layered superconductors.

It is known that artificial superconducting superlattices consisting of conventional superconductor films and some insulating interlayers may perfectly imitate the properties of high- $T_c$  compounds [23–

26]. The cited works concern the flux line creep and the  $H$ – $T$  phase diagram. Obviously, observation of the lock-in transition may be also expected in artificial superconducting multilayers. Here we report on finding the lock-in transition in Mo/Si superlattices (with a temperature of the superconducting phase transition of about 4 K [27]). For the study of this phenomenon we have used the resistive method described by Kwok et al. [22], as well as a new method based on the effect of commensurability between intervortex distance and the superlattice wavelength. We believe that the latter method provides the clearest evidence of vortex locking between the layers in oblique fields as compared with all other methods mentioned above. The explicit dependence of the critical angle for the lock-in transition on temperature is obtained for the first time.

### Sample preparation and experimental procedure

The measurements were carried out on a Mo/Si multilayered sample with Mo layer thickness of 22 Å and Si layer thickness of 34 Å. The sample consists of 50 bilayers. The Mo/Si multilayer was prepared by two-magnetron sputtering onto a glass substrate at  $T = 100^\circ\text{C}$  in argon. The working pressure of argon in the deposition chamber was  $3 \cdot 10^{-3}$  torr. The initial vacuum was no worse than  $10^{-6}$  torr.

Small-angle  $x$ -ray diffractometry was used for the determination of the superlattice period and for checking the degree of sample perfection. The number of satellite lines on the diffractograms for the samples investigated is 4, while for multilayers prepared in the same way with wavelengths equal to or exceeding 100 Å this number is about 10 or more. These data attest to the high regularity of the layering. The same conclusion follows from an electron microscopy investigation of the sample cross section. The latter also shows that the roughness of the interfaces does not exceed 7–8 Å. The multilayer period was determined with an accuracy of 0.1 Å.

The  $x$ -ray diffraction data showed that the silicon layers are amorphous and the molybdenum layers are microcrystalline, with a crystallite size of several nanometers. More details about the sample preparation and characterization may be found in Refs. 27–29.

The transport measurements were performed in a standard helium cryostat equipped with a 5 T superconducting coil. The geometry of the experiments is shown in Fig. 1. The orientation of the sample

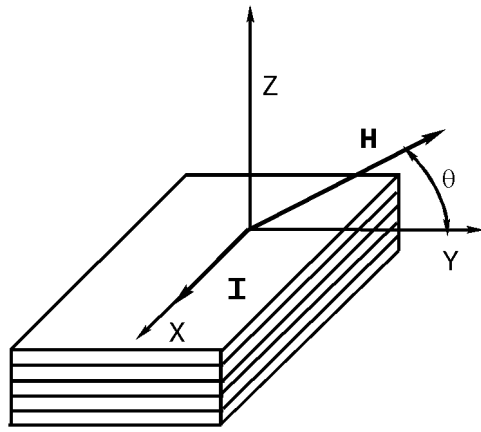


Fig. 1. Geometry of the experiment.

holder in a magnetic field was changed with the help of special rotation mechanism. The accuracy of the determination of the angle between the applied magnetic field  $\mathbf{H}$  and the layer planes was no worse than  $0.1^\circ$ . During the rotation of the sample the transport current was always perpendicular to the applied magnetic field. The parallel orientation was identified by finding the minimum in the resistance. The stabilization of the temperature at a given point was about  $10^{-3}$  K. The critical magnetic fields were defined in the resistive transitions with the use of the criterion  $R = 0.5R_n$ . The resistance measurements were carried out using the standard four-probe technique with a transport current of 1 mA.

### Experimental results and discussion

In Fig. 2,*a* the typical dependences of the resistance on the parallel magnetic field at different temperatures are shown for a case of strong intrinsic pinning. At temperatures close to the transition temperature  $T_c$  all the resistive curves are smooth (they are outside the scope of this figure). Beginning from the temperature 3.5 K (the  $T_c$  for this sample is 3.67 K), minima appear on the  $R$  vs.  $H_{\parallel}$  curves and at still lower temperatures these minima are transformed to zero resistance regions (Fig. 2,*b*). These dependences closely correlate with the dependence of the critical current  $I_c$  on  $H_{\parallel}$ , as Fig. 2,*c* shows. As was proved recently [5], such nonmonotonic behavior of the critical current and resistivity and also the reentrance of superconductivity may be explained in terms of the commensurability effect that should be observed under the condition of strong intrinsic pinning. The locations of the  $R$  vs.  $H_{\parallel}$  minima (and the  $I_c$  maxima, respectively) correspond to the stable states of the commensurate vortex lattices. For the parallel field the

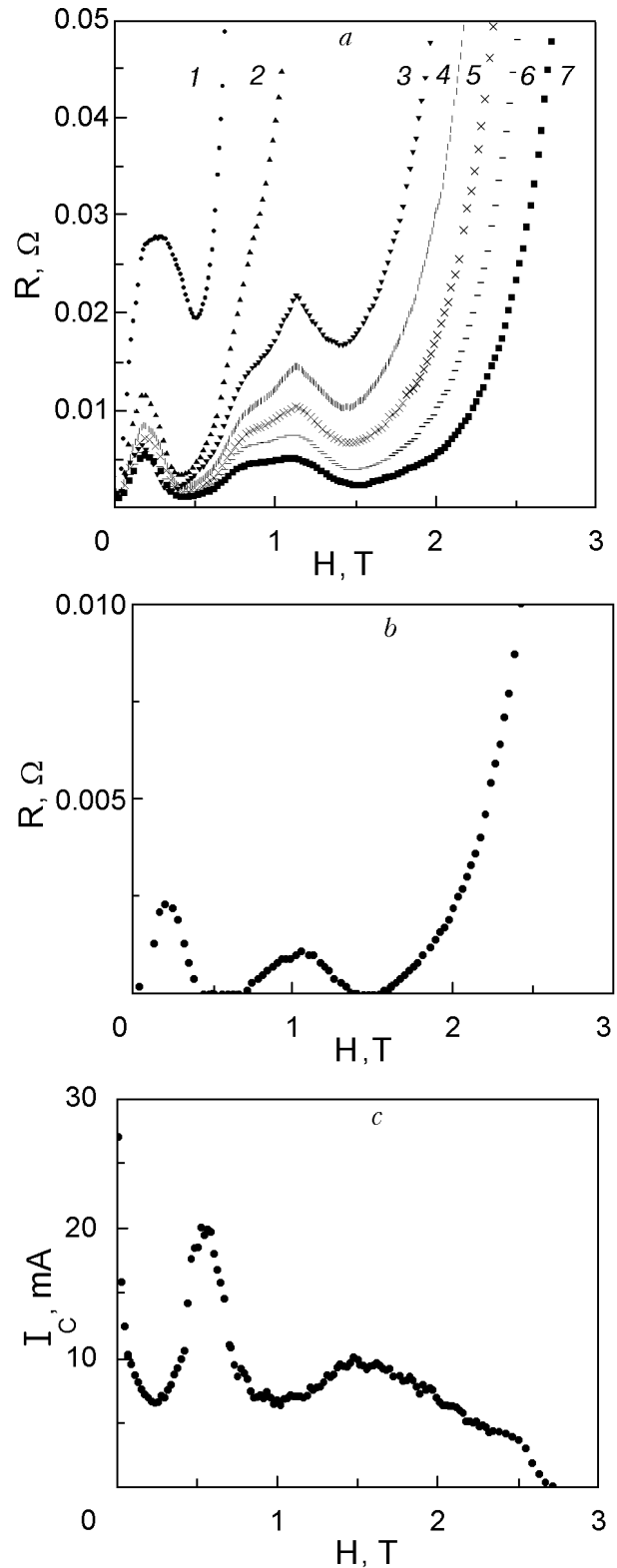


Fig. 2. Resistance as a function of the parallel magnetic field at different temperatures  $T$ , K: 3.504 (1); 3.478 (2); 3.467 (3); 3.462 (4); 3.459 (5); 3.453 (6); 3.447 (7) (*a*); at  $T = 3.44$  K (*b*). Critical current as a function of the parallel magnetic field (*c*).

positions of the minima do not shift with temperature, as would be expected for the matching effect.

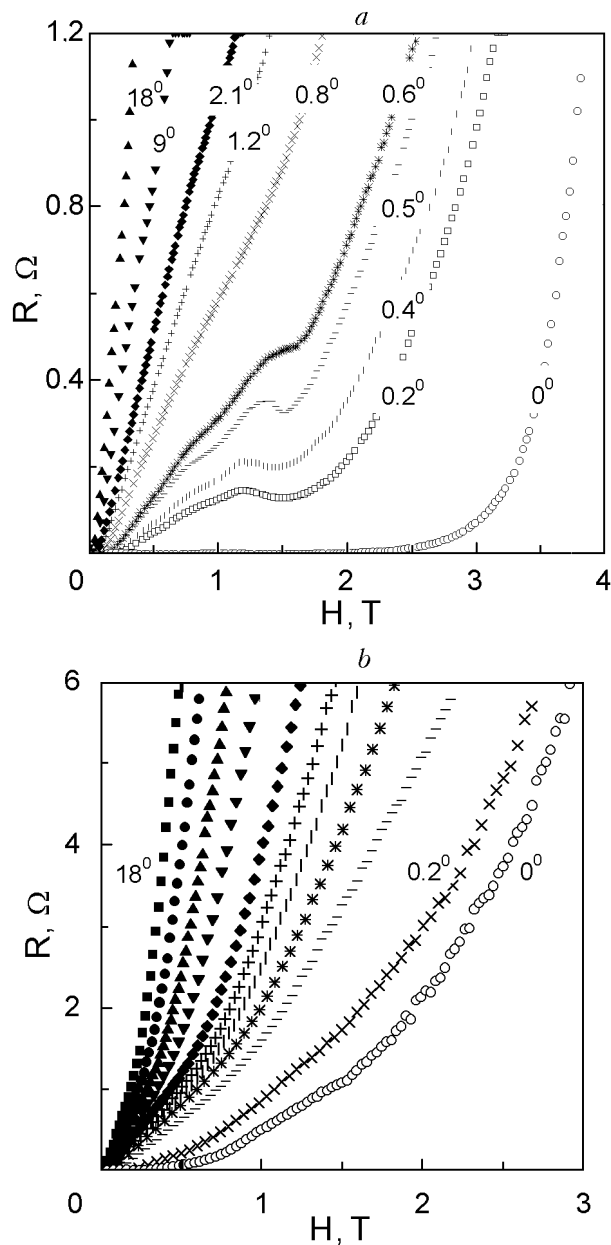


Fig. 3. Magnetic field dependences of the resistance at different orientations of the applied magnetic field at  $T = 3.44$  K (a) and  $3.503$  K (b).

The manifestations of the commensurability effects appear below some temperature  $T_0$ , where the condition  $\xi_{\perp} < s/\sqrt{2}$  is met [5]. This temperature, as was mentioned above, is equal to  $3.5$  K. The data presented in Fig. 1 correspond to the stable states of commensurate VL configurations with  $N = 1$  and  $N = 3$ . Other vortex arrangements with essentially different  $R$  vs.  $H_{\parallel}$  curve patterns can be observed on the same sample, as was shown in Ref. 5. Obtaining one or another vortex arrangement depends on the magnetic history. In this paper we shall deal with only one of the possible kinds of  $R$  vs.  $H_{\parallel}$  curve presented in Fig. 2.

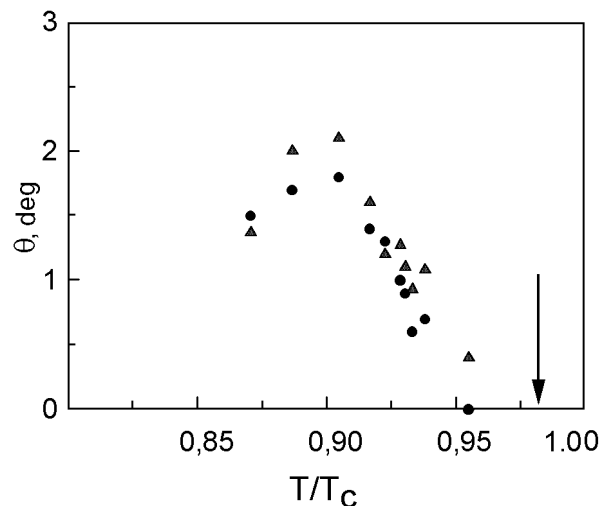


Fig. 4. The critical angle as a function of reduced temperature: obtained from the plots similar to Fig. 3,a (●); determined in the way shown in Figs. 5 and 6 (▲). Arrow shows the crossover temperature.

If the lock-in transition exists in the system investigated, one should expect the appearance of resistivity minima at small tilting angles  $\theta$  as well (the angle  $\theta = 0^\circ$  for the magnetic field parallel to the layer planes). In the range of angles where the vortex lines are trapped between the superconducting layers, the position of the resistance minima is bound to remain constant because the VL structure stays unchanged. As Fig. 3,a shows, this is indeed the case at sufficiently low temperatures. At larger angles the minimum disappears. The critical angle  $\theta_c$  dividing the  $R$  vs.  $H$  curves with and without a minimum depends on temperature, as follows from Fig. 4. At high temperatures there are no features on the  $R$  vs.  $H_{\parallel}$  curves for  $\theta = 0^\circ$  or for the tilted fields either. The first slight kinklike hints appear at  $T = 3.5$  K (Fig. 3,b), and for this temperature  $\theta_c$  is equal to zero with an accuracy of  $0.1^\circ$ .

In the paper of Kwok et al. [22] the lock-in transition was also observed on  $\text{YBa}_2\text{Cu}_3\text{O}_x$  single crystals by the resistive method. A sharp drop of the resistance was found on the  $R$  vs.  $\theta$  curves in the range of small angles and was convincingly interpreted by the authors as evidence of the lock-in transition. The critical angle  $\theta_c$  in these experiments was about  $0.3^\circ$ , and it was practically independent of temperature. However, the latter statement cannot be considered as very reliable because the investigations were carried out in a very limited temperature range ( $T/T_c = 0.993-1$ ). Probably the too-high slope  $dH_{c\parallel}/dT|_T$  of the parallel critical fields has prevented those authors from going to lower temperatures.

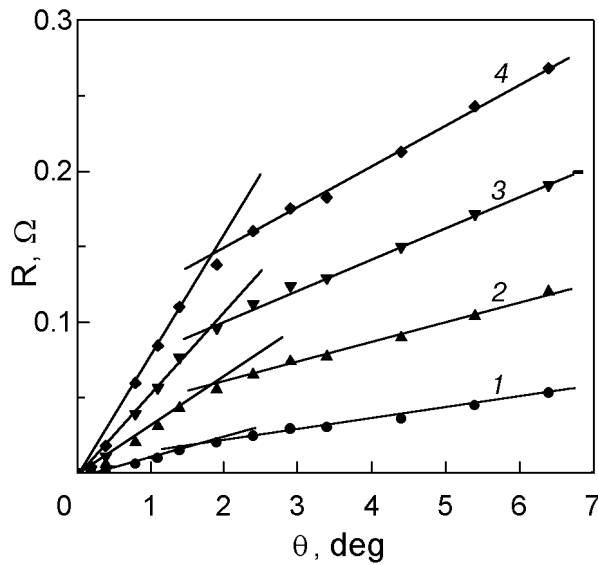


Fig. 5. Angular dependences of the resistance at different magnetic fields  $H$ , T: 0.2 (1); 0.3 (2); 0.4 (3); 0.5 (4).

A similar sharp drop in the resistance is observed in our experiments in the vicinity of the parallel orientation (Fig.5; compare it with Fig. 2 in Ref. 22). It corresponds to the fast increase of the critical magnetic field in the same range of angles (see Fig. 6, curve 4). As Figs. 5 and 6 show, the  $H_{c2}$  vs.  $\theta$  curves allow one to determine  $\theta_c$  as well as the  $R$  vs.  $\theta$  curves do. Curves of  $H_{c2}(\theta)$  for different temperatures are presented in Fig. 6. The values of  $\theta_c$  obtained from these plots are displayed in Fig. 4 along with those obtained from the plots similar to Fig. 3,a. It is seen that the  $\theta_c$  values determined by the two methods differ insignificantly.

According to the theoretical results of Feinberg and Villard [14], for  $H_{c1} \ll H \ll H_{c2}$  the region of field orientations for which the flux lines are trapped parallel to the layers (i.e., the critical angle  $\theta_c$ ) should depend on the barrier height associated with the intrinsic pinning, on the anisotropy parameter  $\gamma$ , and on the magnetic field intensity. The value of the critical angle is determined by the expression [14]

$$\cos(90^\circ - \theta_c) = \frac{1}{\pi} \left[ 2a_1 \frac{H^*}{H} \left( 1 + \gamma^2 \frac{H^*}{H} \right) \right]^{1/2}. \quad (1)$$

Here the field  $H^*$  is of the order of the first critical field  $H_{c1}$ ; the value  $a_1$  characterizes the barrier height ( $a_1(T) = \exp(-c\xi(T)/s)$ ;  $c$  is some numerical constant),

$$\gamma = (M/m)^{1/2} = (dH_{c\parallel}/dT)|_{T_c} / (dH_{c\perp}/dT)|_{T_c}.$$

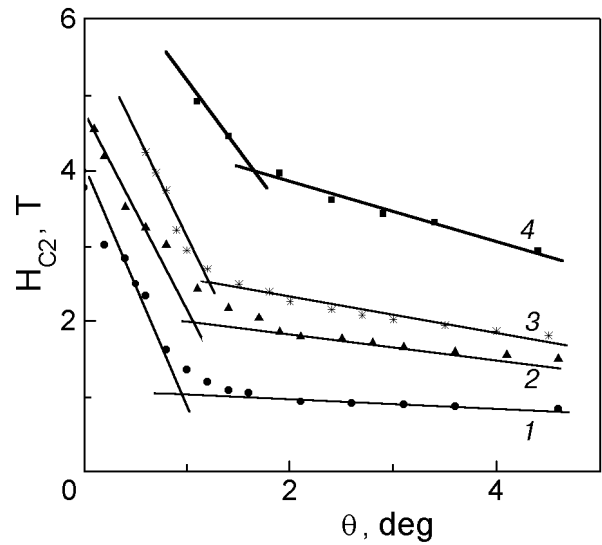


Fig. 6. Angular dependences of the parallel upper critical field at different temperatures  $T$ , K: 3.441 (1); 3.362 (2); 3.318 (3); 3.252 (4).

As the above formula shows, the value of the critical angle  $\theta_c$  separating the two ranges of angles, one in which the vortices are parallel to the layers and one in which the direction of the induction vector  $B$  is tilted with respect to the layers, is mainly determined by the barrier height. The value of  $a_1(T)$  grows with decreasing temperature. If the critical angle is determined from the  $H_{c2}$  vs.  $\theta$  curves, like those in Fig. 6, the variation of  $H$  with the change of the temperature, according to formula (1), may influence on the  $\theta_c$  value too. This may lead to a decrease of  $\theta_c$  at low temperatures. However, the characteristic magnetic field  $H^* \approx H_{c1}(T)$  also increases at low  $T$ . Thus at first glance, the influence of the  $H^*/H$  factor is not so essential as that of the  $a_1(T)$ .

The lock-in transition may be observed in the quasi-two-dimensional state, where substantial modulation of the vortex core energy in the direction orthogonal to the layers exists [12]. It means that one can observe this transition only at temperatures which are less than the crossover temperature

$$T_{cr} = T_c(1 - 2\xi_{\perp}^2(0)/s^2).$$

Indeed, the critical angle goes to zero at a temperature lower than  $T_c$  (Fig. 4). The crossover temperature for this sample is shown by an arrow. The onset of the lock-in transition is observed just below this temperature. As Fig. 4 shows, the critical angle increases with the lowering of  $T$ , and only at the lowest temperature of the measurements is the tendency for diminishing  $\theta_c$  observed. This latter tendency may be associated with the competing

effect of the barrier height  $a_1(T)$  and magnetic field. At sufficiently low temperatures the variation of the coherence length  $\xi(T)$  slows down, and magnetic field enhancement may begin to play the dominant role.

The authors are now investigating the lock-in effect on superlattices with different anisotropy parameters. The results of this study will be published elsewhere.

In summary, the lock-in transition has been observed for the first time on artificial periodic layered systems, Mo/Si including a conventional superconductor as one of the components. Previously it was shown that the new method of investigation of the VL structure in layered superconductors, which is based on the commensurability effect, works fairly well in the case when the magnetic field is parallel to the layers. Here we have shown that this method is also valid for tilted fields at relatively small misalignment between  $\mathbf{H}$  and the layers. We believe that the data about the locking of vortices obtained from the  $R$  vs.  $H$  curves are direct, in distinction to all the other, indirect methods mentioned in the Introduction. For the first time we have obtained the explicit dependence of the critical angle for the lock-in transition on the temperature. An additional important conclusion which follows from our experiments is that, due to the lock-in effect, some deviation from a precisely parallel orientation does not lead to errors in the determination of characteristic fields corresponding to the stable states of the commensurate vortex lattices.

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