Anisotropy of ordering kinetics in a single-phase adsorbed film: $c(2\times2)$O-Mo(011)

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Kinetics of ordering in the $c(2\times2)$O-Mo(011) system after temperature upquench is investigated by video low-energy electron diffraction. Variation of domain size is analyzed on the basis of the first moment of the structure function. We observed an anisotropy in the domain growth rate having anomalous orientation. The rate constant for the $\langle001\rangle$ direction is 1.5 times as large as that for the $\langle011\rangle$ one. The ratio is independent of temperature, i.e., the activation energies for ordering in the two directions are the same. The growth kinetics is characterized by a power law with the exponent $x=1/2$ for both directions. [S0163-1829(97)08040-5]

I. INTRODUCTION

In recent years the problem of ordering amorphous matter has attracted much attention.[1–3] Many experiments have been carried out with commensurate adsorbed layers, which are remarkable for a great variety of two-dimensional lattices showing different degrees of domain degeneracy p.[4] Ordering characteristics such as kinetics of domain growth, distribution of domain sizes, and thermal activation energies are usually derived from the low-energy electron diffraction (LEED) intensity profiles monitored during annealing.[3,5]

Though, in general, the fundamental theoretical ideas, viz. power-law kinetics and scaling relation have been confirmed experimentally, there is a considerable inconsistency in some important quantitative results. For instance, evolution of the average size $\langle L\rangle$ of the ordered-phase domain fits a general expression $\langle L\rangle\propto At^x$, where t is time of ordering, x is the growth exponent, and A is the temperature-dependent rate constant. However, the values measured of the growth exponent are scattered within a wide range of $x=0.2–0.5$ (Refs. 3 and 6) instead of showing one of the two universal values, theoretically predicted: 1/2 (for single-phase systems) or 1/3 (for double-phase systems). This leaves much room for speculations concerning the applicability of different theories as well as the validity of the concept of the influence of domain degeneracy on the exponent value.[3] The discrepancy may have different reasons. A simpler one is that an inappropriate measure for $\langle L\rangle$ was used in some studies. It should be noted that several different parameters of a LEED spot profile can be proportional to $\langle L\rangle$: the square root of the peak intensity, the inverse FWHM (full width at half maximum), and the first and second moments of the structure function.[2,3] The first of them is utilized often as the easiest and fastest for data acquisition. However, it is appropriate provided the number of coherent scatterers remains constant during the ordering evolution,[3,7,8] which is rarely realized. Even with the appropriate measure for $\langle L\rangle$ applied, sometimes a more general expression of the power-law fits real ordering kinetics:[9–11]

$$\langle L\rangle=At^x+B, \tag{1}$$

where the constant B describes other faster processes essential at the early stage of ordering, including formation of small initial domains. On the other hand, for rather large t, growth inevitably slows down, supposedly due to the presence of various defects. So choosing the appropriate range of data acquisition is crucial for the evaluation of x. Thus, the primary aim of this work was to study the ordering kinetics, keeping all the mentioned precautions, for the system $c(2\times2)$O-Mo(011), which is a single-phase one and has a rather high level of degeneracy ($p=4$).

The degeneracy of this system may be even larger. Relying on the hypothesis of triple-bond adsorption sites for O on (011) planes of bcc crystals, which was verified for the system O-W(011),[12–14] we assume that there can be two equivalent adsorption sites in each unit cell [inset in Fig. 1(a)], i.e., twice as many domain boundaries along the $\langle011\rangle$ direction relative to the $\langle001\rangle$ one, resulting in $p=8$. If ordering kinetics is really sensitive to the degeneracy, a different kinetics can be expected in this direction. In general, film ordering is held to proceed via surface diffusion, therefore the ordering characteristics should depend strongly on the relief of a substrate. Particularly, surfaces containing steps or atomic channels, existing due to the intrinsic crystal structure of the planes, favor an anisotropic diffusion. An ordering anisotropy is accordingly expected in the films adsorbed on such surfaces. A size anisotropy of ordered domains was actually found in the oxygen overlayer on a typical channeled surface W(112).[15] However, most of the LEED detectors used in the previous experiments[6,15,16] were not fast enough to follow the spot profiles continuously during ordering. Due to this the anisotropy of ordering has still not been studied extensively. Meanwhile, the Mo(011) plane is slightly anisotropic for surface diffusion (the projections of elemental jumps on the $\langle011\rangle$ and $\langle001\rangle$ directions differ by a factor of $\sqrt{2}$). This can favor an ordering anisotropy too. Thus, our second aim was to investigate kinetics in two dimensions by means of a fast enough detector.

II. EXPERIMENT

We utilized a video LEED technique to study the ordering of amorphous films adsorbed at low temperatures. The degree of coverage Θ was close to 0.25 corresponding to the $c(2\times2)$ phase of annealed oxygen films on Mo(011).[17–19] The experiment was performed at a base pressure of $\sim10^{-11}$ Torr. The setup details and the technology of
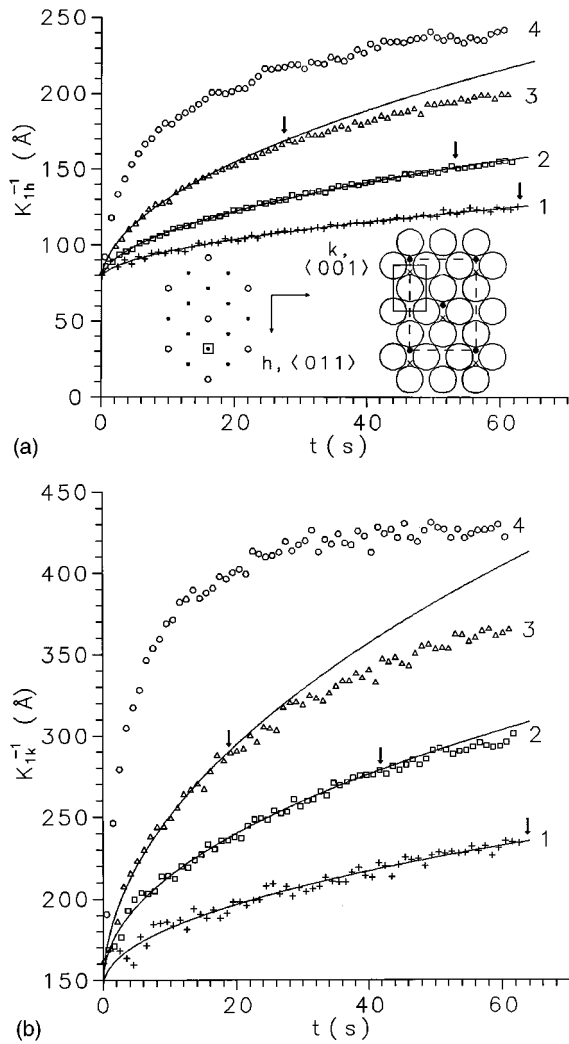
FIG. 1. Evolution of the inverse first moment of the superstructure LEED reflex from the $c(2\times2)$O overlayer on Mo(011) resulting from upquench to different temperatures T_o (K). 1—302; 2: 309; 3: 314; 4: 336; (a) and (b) are for the $\langle011\rangle$ and $\langle001\rangle$ directions, respectively. Lines show the power-law approximation curves, vertical arrows show the end points of approximation. Inset shows a scheme of the LEED pattern (to the left), a possible surface structure model (to the right), and principal axes. Open circles and dots in the pattern denote spots from the substrate and the overlayer, respectively. Solid-line and dashed-line rectangles in the model depict $c(1\times1)$ substrate and $c(2\times2)$ overlayer unit cells, respectively; dots and crosses show hypothetical equivalent adsorption sites.

sample preparation, cooling, and cleaning have been described elsewhere.[20] Oxygen was produced by the decomposition of CuO contained in a resistively heated platinum tube. For a measure of the oxygen coverage we used the exposure $\int\Delta p\,dt_a$, where Δp is the variation of the oxygen pressure in the chamber and t_a is the time of adsorption. The oxygen coverage was also determined more accurately by Auger amplitude and LEED intensity measurements. The chamber was equipped with a commercial four-grid LEED optics and a phosphor screen. The width of the LEED instrumental function corresponded to a coherence zone of 150 Å for the electron energy used $E=22$ eV.

Only one diffraction spot specific for the $c(2\times2)$ struc-

ture has been investigated [inset in Fig. 1(a)]. This spot is chosen in order to exclude a contribution from another phase coexisting with the $c(2\times2)$ at $\Theta>0.25$.[17,19] The intensity distribution across the spot was monitored by the digital video system via program-controlled scanning. The major spot profile data have been collected in the frames of a 53×24-element window with the registration frequency of up to 4 pictures/s. A 150-Hz pulsed resistive heating within the intervals between the records was used to control the sample temperature.

The experimental procedure was as follows. Oxygen was adsorbed at $T_{ad}=78$ K on the surface preliminarily cleaned. This temperature was distinctly lower than that providing an overlayer mobility. Then the sample was upquenched (within the transient time of 1.8 s) to a steady value in the interval $T_o=302$–336 K necessary for ordering. The monitoring of the pulsed spot profile with a period $\Delta t=1$ s was started from the low-temperature state before upquenching. Finally the film was annealed at $T_{an}=1500$ K, the resulting LEED pattern was recorded, and Auger measurements were performed.

The spot profiles originally taken are two-dimensional time-dependent structure functions $S(q_h,q_k,t)$, with the q_h and q_k components of the scattering vectors being measured along the $\langle011\rangle$ and $\langle001\rangle$ substrate directions, starting from the spot center. Data processing consisted of background subtraction, deconvolution of the instrument response function, and calculation of the values considered to be the domain size measures, viz, K_{1h}^{-1} and K_{1k}^{-1}, which are the inverse first moments of the structure function along the h and k directions:

$$K_{1h}=\sum_h |q_h|S(q_h,0,t) \Big/ \sum_h S(q_h,0,t), \qquad (2)$$

$$K_{1k}=\sum_k |q_k|S(0,q_k,t) \Big/ \sum_k S(0,q_k,t). \qquad (3)$$

The choice of such a measure for $\langle L\rangle$ provided best statistics. The S value in the non-annealed state was taken as the background level. That for the fully annealed film was taken as the instrument function since its FWHM was proved to be equal to the FWHM of the clean-substrate reflex. Deconvolution was performed by the Fourier transform technique.[21]

III. RESULTS

Figure 1 shows the kinetics of domain size growth in terms of K_1^{-1} measured at different annealing temperatures for two crystallographic orientations. The scale for K_1^{-1} is calibrated from measurements of the positions of superstructure spots. The t axis is presented starting from the actual change of K_1^{-1}, which occurs upon an emergence of the fractional-order spots due to a temperature upquench. Before the upquench, a uniform background only was registered. Three known stages of growth,[3] initial-domain formation, domain growth, and slowing down of the growth, can be distinguished. They are most pronounced at high temperatures [see curve 4 in Fig. 1(b)]. The first stage (initial jump at

TABLE I. Domain growth parameters fitting Eq. (1) to the experimental data on kinetics of ordering for the $c(2 \times 2)$O-Mo(011) system at different ordering temperatures T_0: A,B,x. Subscripts h and k mark directions $\langle 011 \rangle$ and $\langle 001 \rangle$, respectively.

T_0 (K)	A_h (Å s^{-x})	B_h (Å)	x_h	A_k (Å s^{-x})	B_k (Å)	x_k
302	1.56	23.4	0.51	3.54	43.8	0.49
309	3.24	22.5	0.48	6.66	44.4	0.48
314	5.10	22.5	0.52	9.87	43.8	0.50

$t \geqslant 0$) is too fast to be followed by our technique and manifests itself only by the non-negligible initial-domain size. Its value appears independent of T_o in the range used in this experiment. In what follows, the overlayer order emerged at this stage is called an initially formed one. At the second stage, the subsequent appreciable increase proceeds over quite some time. It is clearly seen in each curve. Evidently, this domain growth is thermally activated, taking a shorter time the higher the temperature. A transition to the last stage (leveling off) is rather protracted. It begins from a slight rate slowdown and ends with an eventual growth saturation. It should be noted that full annealing at 1500 K results in raising the leveling-off value up to that limited by the instrumental function.

The most remarkable feature of ordering kinetics for the system studied is an anisotropy of the growth rate. Indeed, at the same temperature, the slope of the K_{1k}^{-1} curve is greater than that of K_{1h}^{-1} [Figs. 1(a) and 1(b)]. It should be noted that even the initially formed domains exhibit a size anisotropy, which then persists until the leveling off, though it is reduced somewhat due to the earlier slowdown for the $\langle 001 \rangle$ direction.

To derive quantitative information on the growth law, the data of Fig. 1 have been fitted by Eq. (1), using A, B, and x as fitting parameters. As mentioned above, the main problem in the approximation is to avoid exceeding the domain growth regime. Since the quenching transient time in our experiments took only two recording periods, and the stage of initial-domain formation was even shorter, we chose the second nonzero experimental point as the beginning of isothermal domain growth in each curve of Fig. 1. The end points to be used in the approximation (marked by vertical arrows in Fig. 1) were determined by the following. A decrease in the growth exponent can be considered as a sign of the slowing down.[3] In practice, the range of approximation was gradually extended until the value of x changed by more than the least-square error. The ranges of satisfactory approximation are rather wide for $T_o = 302$–314 K. At $T_o = 336$ K the number of points was too small, so the corresponding fit is not presented here. The resulting fitting parameters are listed in Table I. As a matter of fact, K_1^{-1} in Fig. 1 is not an absolute average domain size $\langle L \rangle$, but a value proportional to it. The relationship between $\langle L \rangle$ and K_1^{-1} can be determined from the structure function shape. In our experiments, a Gaussian function was found to fit most closely the spot profile, hence we have $\langle L \rangle = 0.3 K_1^{-1}$.[22] This factor was used to obtain the $\langle L \rangle$ values given in Table I.

As shown in Fig. 1, a power law holds at high temperatures as long as the domain size nearly doubles along both axes. It should be emphasized that the parameter x (growth exponent) has the same value of 0.50 ± 0.03 for the different axes and temperatures, so the parameters A and B only are responsible for the growth anisotropy (see Table I).

IV. DISCUSSION AND SUMMARY

The mere existence of the anisotropy of the growth rate constant A is not very surprising because A is proportional to D^x,[3] where D is the diffusion coefficient, which should be anisotropic for the (011)Mo plane due to its atomic structure. Indeed, the ratio $D_{011}/D_{001} = 2$ is to be expected for a (011) bcc plane. Such a ratio has been actually found in simulations for very distinct hypothetical sets of adatom interactions on this plane[23] and has also been verified in surface diffusion experiments for the O-W(011) system.[24] Meanwhile, our data show just the opposite ratio of the growth rates along the $\langle 011 \rangle$ and $\langle 001 \rangle$ directions. The quantities of A_h and A_k presented for any T_o in Table I are in the constant (within $\pm 4\%$) ratio

$$r_{hk} = A_h/A_k = 0.5, \quad (4)$$

which, if it is caused by the diffusion anisotropy, gives $D_{011}/D_{001} = 0.25$.

The constancy of ratio (4) manifests a trend to self-similar anisotropic growth in two dimensions. Besides, its temperature independence points to equal activation energies for the growth in both directions. Presenting A in the usual Arrhenius form $A(T) = A_0 \exp(-E_o/kT)$ and substituting it in Eq. (4), we find $(A_{0h}/A_{0k}) \exp[-(E_{oh} - E_{ok})/kT] = $ const. Assuming a weak dependence of A_0 on T, we should assume $E_{oh} = E_{ok}$ to satisfy the total temperature independence of r_{hk}. From the Arrhenius plots of the parameters A_h and A_k, the ordering activation energies are found to be $E_{oh} = E_{ok} = 0.75 \pm 0.05$ eV. The orientation independence of E_o means that the orientation dependence of the rate constant A is determined by that of its prefactor A_0.

Since one should have $D \propto A^{1/x}$, the activation energy for surface diffusion E_d is related to E_o as $E_d = E_o/x$, which gives $E_d = 1.5$ eV. This value is substantially larger than $E_d = 1.0$ eV found for oxygen surface diffusion on Mo(011) by the field emission fluctuation method.[25] The discrepancy of the E_d values obtained in different experiments can be sometimes attributed to a distinct state of overlayer order involved in these experiments and therefore to distinct numbers of actually interacting neighbor adatoms.[26] However, accounting for the actual repulsive interaction in our experiment would decrease the E_d value, thus having an effect qualitatively opposite to that which could explain the difference.

It should be considered that domain growth in the single-phase system is implemented through diffusional motion of walls separating antiphase domains rather than of single adatoms. To understand the strange prefactor ratio and high E_d, we suggest that the diffusion process that controls ordering kinetics occurs in the domain wall zone and is unlike the predominantly intradomain diffusion observed, e.g., in field emission fluctuation experiments.[23–26] It is reasonable to search for a cause for the mentioned diffusion anomalies by means of analyzing the characteristics of the domain walls and uniform phase, which can affect the diffusion coefficient, such as concentration and overlayer structure. Recently a Monte Carlo simulation of the domain growth kinetics for a nonstoichiometric two-dimensional structure[27] verified that the different local concentration in the domain wall, in comparison to the uniform phase, has a great influence on the growth rate. Interestingly, it causes the modification of the rate constant rather than the essential time dependence law. Clearly, the local concentration may be unequal for the domain walls of different kinds specific for particular orientations. Hence such an effect may result in an anisotropy of the growth rate, concurrent with the above discussed effect due to anisotropic elemental diffusion jumps. The greater the domain degeneracy, i.e., the greater the variety of the domain walls, the greater this effect should be. In order to evaluate the importance of the discussed effect for the system studied, the structure of the domain walls should be investigated in detail. However, it can be the subject for a special study.

Now we consider the role of the overlayer structure. We concluded above that the E_d value obtained by the ordering kinetics method used in the present study is in rather poor agreement with that obtained by the field emission fluctuation method[25] even if an overlayer disorder is taken into account in the framework of a model of adsorption on the rigid substrate. Meanwhile, in the domain walls, some adatoms are forced to occupy unnaturally close adsorption sites, unlike in any ordered phase of the O-Mo(011) system.[17–19] For example, with triple-bond sites shown in inset in Fig. 1(a), the neighbor sites marked by crosses and dots may be occupied in the domain wall region. Such constrained adatom arrangements in the domain wall region may induce some strains in the substrate. A reconstruction was found for the $c(2\times2)$O-Mo(011) phase upon cooling,[28] making it seem all the more likely for constrained configurations of the domain walls. It is worth noting that the reconstructed domain walls were experimentally observed in the oxygen overlayers, though on another substrate W(011).[13] According to Ala-Nissila and Ying,[29] local overlayer distortion can result in anomalous diffusion properties. In particular, a deep enough reconstruction can increase the diffusion barrier and, for a specific potential relief, the diffusion anisotropy ratio D_{011}/D_{001} can be reduced appreciably. Thus, assuming the validity of the local reconstruction model for the domain walls in the O-Mo(011) system, both high E_d and inverse D_{011}/D_{001} observed could be explained. However, so far there is no experimental evidence for distorted structure of domain walls in this system.

The next interesting feature of domain growth for the system under investigation is the significant domain size reached at the first stage of ordering. The parameter B can be used as an estimate for it. The B values in Table I show that growth begins from the domains composed on the average of two and a half $c(2\times2)$ unit cells in the $\langle011\rangle$ direction and about seven such cells in the $\langle001\rangle$ direction. Recently a Monte Carlo simulation of the ordering kinetics was performed specifically for the system $p(2\times1)$O-W(011).[30] This has revealed a steep rise in the diffusion barrier at very early times of ordering due to fast variation of the number of interacting neighbor adatoms upon ordering. This result gives a clear basis for admitting $B\neq0$ also in the case of the $c(2\times2)$O-Mo(011) system since a similar rise in the diffusion barrier is expected for it during ordering. The nonzero initial-domain size found in this work agrees with that derived from the data reported for other systems.[15,31] However, in some works using the square root of the peak intensity as a measure for $\langle L\rangle$ (Refs. 6, 15, and 16) the initial-domain size was treated as being negligible.

Some late-time kinetics features observed in the present study for the $c(2\times2)$O-Mo(011) system (such as slowing down, leveling off) are in good agreement with the experimental data reported earlier for other systems.[3] Slowing down is temperature dependent and recorded long before the domain size reaches the instrumental limit. The leveling-off value does not correspond to a true saturation of growth and can be treated rather as some intermediate ordering characteristic, which is improved by further annealing at higher temperatures. Thus there is a higher potential barrier limiting the growth at this stage (or several such barriers). Some approaches to understanding the causes of a growth slowdown and eventual leveling off were suggested in Ref. 16. Omitting those corresponding to measurement techniques, we will consider two diffusion-limiting effects specific for the film ordering experiments: a pinning effect from uncontrolled impurities and an effect due to the finite size of substrate terraces. The latter does not seem to play an appreciable role because the typical terrace sizes on Mo(011), under the hardest condition of thermal treatment, exceed 500 Å (Ref. 32) (far above $\langle L\rangle$ considered). As for impurities, their presence is difficult to rule out completely. However, the estimated concentration of defects necessary for pinning at the leveling-off size such as in our case $(80\times130\ \text{Å}^2)$ corresponds to a fairly appreciable value of 1×10^{-3} monolayers. Such a contamination level can be accumulated in vacuum characterizing our experiment during an hour, which is much longer than the typical measurement time (10 min). As was noticed earlier,[3,16] there is no convincing explanation for slowing down. Its cause should be searched among processes preventing infinite growth of a domain size. One of the processes may be a mesoscopic domain structurization that relieves, with mediation of the substrate, the surface stress induced by the overlayer. Such a phenomenon has been found by STM for the O-W(011) system,[13] which is closely related to the O-Mo(011) system studied here.

Our data on the growth exponent at the stage of the power-law kinetics correspond within ±6% to the Lifshitz-Allen-Cahn theory[33,34] predicting a power-law evolution with the growth exponent $x=1/2$ for single-phase commensurate structures with a double-degenerate domain ground state $(p=2)$. This finding obtained for a multidegenerate system is, to our knowledge, the second case (after Bush and Henzler[31]) of close agreement with a theory developed in the framework of $p=2$. The possible influence of p on x is still

under discussion.[3] Theoretically, x might become smaller for $p > 2$.[33] However, the universality of $x = 1/2$ was experimentally verified up to $p = 3$,[31] and our observation extends the universality to $p = 4$ or perhaps to $p = 8$. Taking into account this result and the above discussion of preexponential factor in relation to the domain degeneracy, we suggest that the domain degeneracy influences the preexponential factor rather than the growth exponent.

In summary, we have found an anisotropy of ordering in a single-phase 2D system, with the axis of fast domain growth being orthogonal to that of fast single-adatom diffusion. The domain growth kinetics exhibits the classical power-law behavior with the growth exponent equal to 1/2 not depending on direction. The ordering activation energy is also independent of direction. Hence, the anisotropy of ordering is completely caused by that of its preexponential factor.

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