Formation of a quasicrystalline Pb monolayer on the 10-fold surface of the decagonal Al–Ni–Co quasicrystal

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ABSTRACT

Lead has been deposited on the 10-fold surface of decagonal $Al_{72}Ni_{11}Co_{17}$ at room temperature to form an epitaxial quasicrystalline single-element monolayer. The overlayer grows through nucleation of nanometer-sized irregular-shaped islands and the coverage saturates at 1 MLE. The overlayer is well-ordered quasiperiodically as evidenced by LEED and Fourier transforms of STM images. LEED measurements also indicate that annealing the film to 600 K improves the structural quality, but STM shows that this causes the film to develop pores. Adsorption of C_{60} molecules on this surface showed that the pores are Pb-containing. Electronic structure measurements using X-ray photoemission spectroscopy indicate that the chemical interaction of the Pb atoms with the substrate is weak, and that Pb does not desorb from the surface upon annealing.

1. Introduction

Quasicrystals are metallic alloys that exhibit structure and symmetries that are not found in periodic crystals. The most common kinds of quasicrystal exhibit 5-fold (icosahedral) or 10-fold (decagonal) rotational symmetries as evidenced using diffraction techniques. A well-known aperiodic mathematical analogue to a quasicrystal is the Penrose tiling, which has previously been successfully mapped onto scanning tunnelling microscopy images from various quasicrystals [1,2].

These unusual materials have been the subject of intense study for the past two decades in a concerted attempt to unravel their structure and properties. The technologically desirable combination of high hardness coupled with low surface energy, low friction and other unusual properties, for example a negative coefficient of electrical resistivity as a function of temperature, has attracted much interest from research groups around the world [3]. The development in recent years of the ability to prepare quasicrystal surfaces with a terrace-step morphology suitable for surface science techniques has led to an upsurge in studies of clean quasicrystal surfaces and their interactions with atomic and molecular species.

The progress that has been achieved in understanding and classifying atomic adsorption on quasicrystal surfaces has been summarised in recent review papers [4–7]. Several atomic species have been found to chemisorb and subsequently form epitaxial complete layers and multilayer films. These can be arranged into a number of broad classifications. One class contains elements that form crystalline structures which are aligned along substrate high symmetry directions, with or without some intermixing, e.g. Fe adsorbed on i-Al–Pd–Mn [8] or Fe adsorbed on d-Al–Ni–Co [9]. Another class of adsorbates can be described as forming modulated multilayers. Such structures have been found for Cu adsorbed on the 5-fold surface of icosahedral Al–Pd–Mn (i-Al–Pd–Mn) [10] and for Co adsorbed on both i-Al–Pd–Mn and the 10-fold surface of decagonal d-Al–Ni–Co (d-Al–Ni–Co) [11]. They consist of small domains of crystalline material oriented in five directions corresponding to high symmetry orientations on the substrate. The domains themselves have a quasiperiodic structural modulation consistent with terms of the Fibonacci sequence [10,12,13].

A third class are pseudomorphic systems where the adsorbing species adopts the structure of the quasicrystal substrate. Such systems have only been discovered to date on quasicrystals with coverages from low sub-monolayer up to saturated monolayer. Adsorption of Sb and Bi on both i-Al–Pd–Mn and d-Al–Ni–Co [14] has been found to lead to the formation of monolayer pseudomorphic systems. Aluminium atoms form small pentagonal clusters ('starfish') on i-Al–Cu–Fe at low sub-monolayer coverages of ≈0.04 ML, although at higher coverages disordered adsorption occurs [15]. This has also been found for Si adsorption on both i-Al–

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Pd–Mn [16] and d-Al–Ni–Co [17]. In experimental and theoretical studies of rare gas adsorption on the 10-fold surface of d-Al–Ni–Co, Xe was found to physisorb, with the strength of the adsorbate–adsorbate and adsorbate–substrate interactions being comparable. The first layer had 5-fold symmetry, and a transition to 6-fold symmetry indicating bulk Xe formation was observed after a coverage of one monolayer [18–22].

Recently Pb has been added to the list of adsorbed species which form pseudomorphic overlayers [23]. When dosed on the 5-fold surface of i-Al–Pd–Mn at low sub-monolayer coverages, Pb atoms were found to nucleate into pentagonal structures. As the atomic density of the atoms increased, an interconnecting network was formed until a saturated quasicrystalline monolayer was observed. Using scanning tunnelling spectroscopy and ultraviolet photoemission spectroscopy, this Pb monolayer was found to display an electronic pseudogap similar to those found in bulk quasicrystalline materials [24].

In this paper, we describe the results of experiments probing the adsorption of Pb on the 10-fold surface of d-Al–Ni–Co. This surface has been shown to be a relaxed truncated bulk structure, having the same composition as the bulk. The outermost layer spacing is contracted by 10% relative to the bulk interlayer spacing, while the next layer spacing is expanded by 5%. A small degree of intra-layer rumpling is observed within each layer [25,26]. This surface is chemically and structurally distinct from that of i-Al–Pd–Mn; therefore it is of considerable interest to investigate whether quasiperiodic ordering similar to that found for i-Al–Pd–Mn/Pb is repeated in this system.

2. Experimental details

The $Al_{72}Ni_{11}Co_{17}$ quasicrystal samples, produced at Ames laboratory using the melt decantation method [27], were polished successively with 6, 1 and 1/4 µm diamond paste before introduction to vacuum and thereafter were prepared in cycles consisting of 45 min sputtering with 3 keV Ar^+ ions followed by 4 h annealing to 1070 K, using electron-beam heating, up to a total annealing time of 20 h. Following this preparation, low energy electron diffraction (LEED) patterns had well defined peaks and impurities were undetectable by Auger electron spectroscopy.

The scanning tunnelling microscopy (STM) studies and the Auger electron spectroscopy (AES) measurements were carried out in an Omicron variable temperature STM (VT-STM) chamber at the University of Liverpool. The base pressure of the system was 1×10^{-10} mbar. The Pb evaporation was performed using an Omicron EFM-3 electron-beam evaporator, with an ion flux monitor reading of 120 nA giving approximately 0.2 monolayer equivalent (MLE) coverage after 15 min. One MLE occurs with the completion of the wetting layer as observed using STM. The chamber pressure did not exceed 2.5×10^{-10} mbar during evaporation. The C_{60} evaporation was performed using a thin W filament wrapped around a Pyrex crucible containing C_{60}, with temperature regulated using a K-type thermocouple tightly strapped to the crucible. During evaporation the W filament glowed yellow and was approximately 3 cm away from the sample. Evaporating temperatures in the range 500–520 K were used and the chamber pressure did not exceed 4×10^{-10} mbar during evaporation.

The X-ray photoemission spectroscopy (XPS) measurements and LEED results were obtained at the Ecole des Mines, Nancy, France. STM and AES measurements were also carried out for comparative purposes. In those experiments the Pb source was calibrated by means of XPS and STM using an Al(1 1 1) crystal. In both experiments, Pb adsorption was carried out with the sample at room temperature. The X-rays are from a Mg Kα source (1253.6 eV).
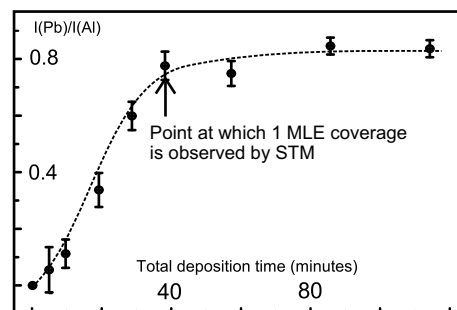

Fig. 1. Plot of Pb(NOO)/Al(LMM) Auger peak intensity ratio versus deposition time for 1 MLE of Pb adsorbed on the 10-fold surface of d-Al–Ni–Co.

3. Results and analysis

Fig. 1 shows AES measurements of the film during deposition. The ratio of intensities of Pb(NOO) and Al(LMM) peaks is plotted as a function of coverage. Following an initial rapid uptake, the Pb coverage saturates at monolayer coverage. This behaviour was also observed for i-Al–Pd–Mn/Pb, and is discussed further in Section 4.

The clean surface had a step-terrace morphology with terrace widths of order 20 nm as observed using STM. STM images yielded a dense 10-fold fast Fourier transform (FFT), again indicative of quasiperiodic ordering. An overview of the growth of the Pb film is given by the STM images in Fig. 2. Fig. 2a shows the system after deposition of 0.3 MLE of Pb. The Pb atoms can be seen to nucleate in small islands on the surface of average size ≈1 nm. During growth of the film, noise at the edges of Pb islands evidenced the diffusion of Pb adatoms on the surface. This is tip-induced, but nevertheless indicates a high mobility for Pb atoms on this surface. Resolution within the islands themselves was poor. When the coverage is at sub-monolayer levels, resolution of the substrate is poor also. This could be attributable to the high roughness of the partially dosed surface reducing the ability of the tip to track protrusions. After further evaporation to a coverage of 1 MLE, the surface was completely covered with Pb atoms as shown in Fig. 2b.

Fig. 3a shows a 15 nm × 15 nm image of the Pb monolayer film (before annealing). At this coverage, atomic-scale resolution was routinely obtained. A 5-fold pentagon (defining a 5-fold hollow) is circled. Such hollows are a very distinctive feature of clean quasicrystalline surfaces; on the clean surface of i-Al–Pd–Mn they have been dubbed 'dark stars' [28,29]. Also circled is another larger 5-fold feature which is reminiscent of structures found on the 5-fold surfaces of icosahedral quasicrystals [28] and on the clean surface of d-Al–Ni–Co [26]. The observation of these features indicates that the structure of the quasicrystalline overlayer has similarities to that of the clean surface. The distance between the bright protrusions forming the pentagonal hollow is 4.9 Å, similar to the size of the pentagons observed for i-Al–Pd–Mn/Pb. Fig. 3b shows an FFT from the surface shown in (a), obtained from an image 50 nm × 50 nm in dimensions. Three distinct rings of spots are observed; the radii of the rings are τ-scaled.[2]

Fig. 4a shows the 1 MLE coverage after annealing to a temperature of 600 K for 15 min. The film develops nanosized 'pores'. Analysis by thresholding of many plane-compensated STM images reveals that 12 ± 2% of the surface is made up of pores following annealing. The size of the pores ranges from 3 to 150 nm², with a wide distribution in-between. Fig. 4b shows an FFT from the sur-

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[2] The number $\tau = \frac{1+\sqrt{5}}{2} = 1.618\ldots$, known as the golden ratio, is intrinsic to the geometry of pentagons, to Penrose tilings and the Fibonacci sequence.
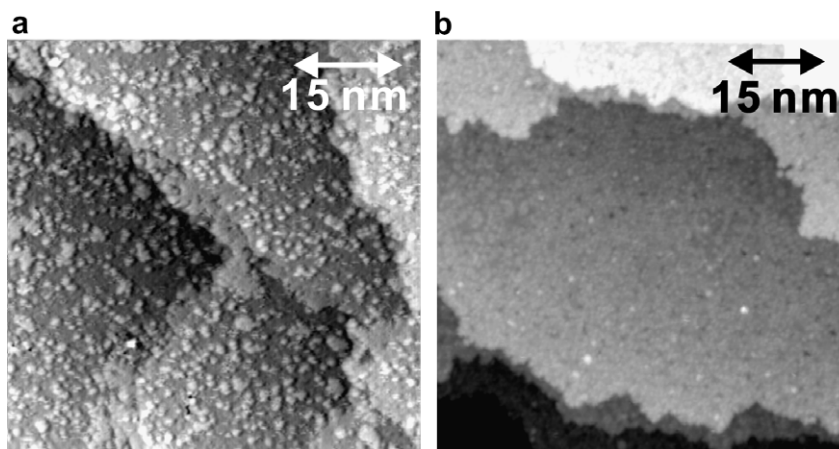
Fig. 2. STM images of the formation of the Pb film on d-Al–Ni–Co. (a); 57 nm × 57 nm, 0.32 MLE, Z_{rms} = 45 pm; (b); 64 nm × 64 nm, 1 MLE, Z_{rms} = 27 pm.
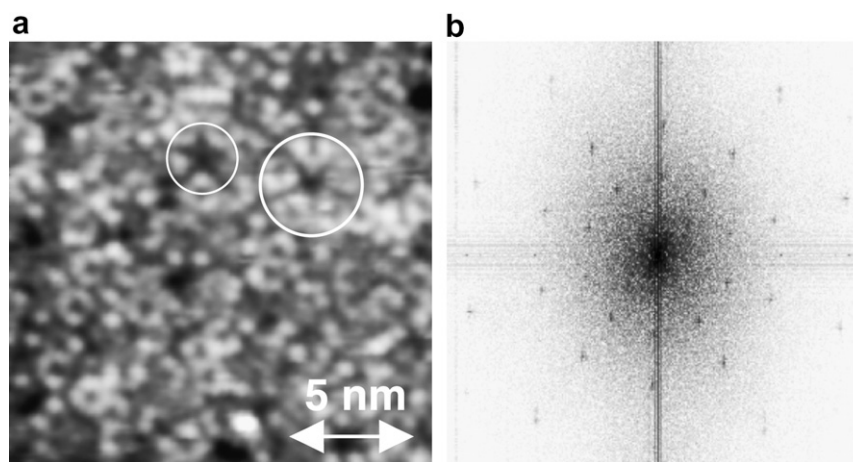
Fig. 3. (a) Quasicrystalline Pb film on d-Al–Ni–Co before annealing. The circles indicate pentagonal motifs. 15 nm × 15 nm, 1 MLE, Z_{rms} = 25 pm. (b) FFT of a 50 nm × 50 nm image from this coverage, showing several decagonal rings indicating excellent quasiperiodic ordering.
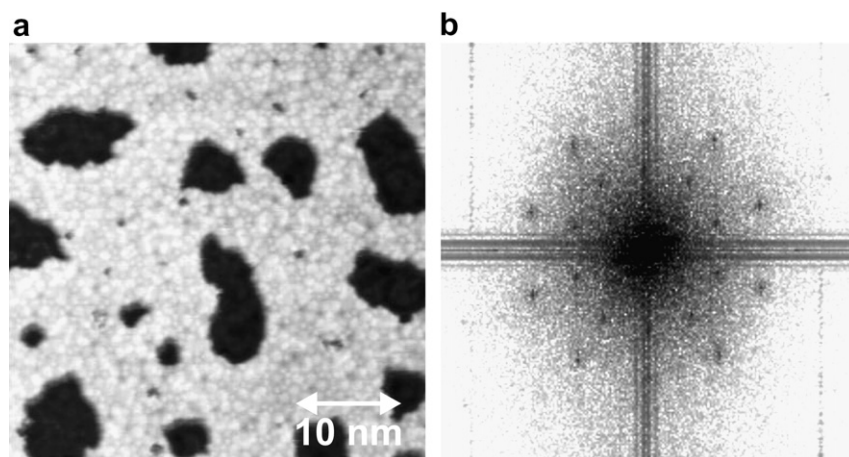

Fig. 4. (a) STM image, 20 nm × 20 nm of the 1 MLE film following annealing to 600 K, showing the porosity developed in the film, Z_{rms} = 20 pm both in the pores and outside the pores. (b) FFT of the image in (a), showing that decagonal rings remain after annealing.

face shown in (a), obtained from an image 50 nm × 50 nm in dimensions. In this case only two distinct rings of spots are observed; the radii of the rings are τ-scaled. These rings have the same radii as for the unannealed film shown in Fig. 3. The FFTs from images of the annealed film with pores removed by thres-

holding is indistinguishable from FFTs of the annealed film with pores.

In order to determine the chemical nature of the pores, C_{60} was used as a probe molecule. C_{60} deposited on the clean surface of d-Al–Ni–Co at room temperature forms a disordered overlayer with-
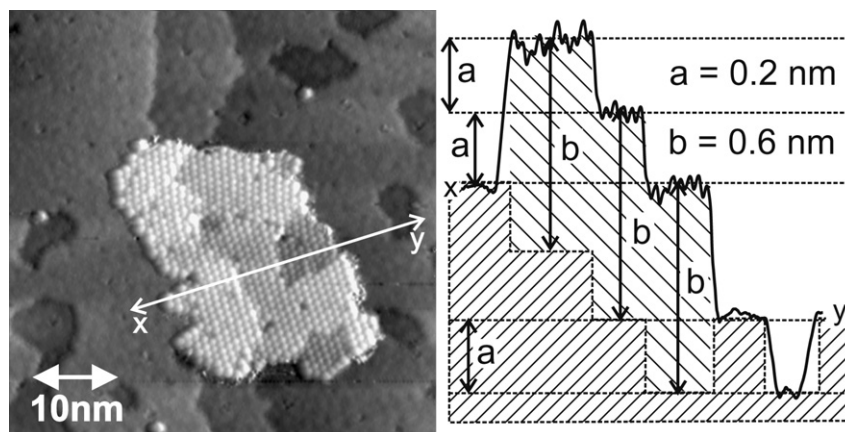
Fig. 5. (Left): A 50 nm × 50 nm STM image of a C_{60} island on the annealed Pb/AlNiCo surface. (Right): A profile scan taken along the line *xy* in the STM image. Dark-hatched areas denote the Pb-covered substrate, lighter hatched areas denote C_{60}.

out formation of hexagonal structures [30]. Fig. 5 shows the resulting structure when C_{60} is deposited on the annealed Pb-covered surface. The left panel shows an island of C_{60} molecules in an hexagonal structure. The right panel shows the *z*-profile of the line indicated on the left.

The information in the line profile is consistent with C_{60} adsorption on the Pb-covered substrate both inside and outside of the pores. The step height on the d-Al–Ni–Co surface is 0.2 nm [26]. C_{60} molecules image as protrusions of apparent height 0.6 nm. The profile from *x* to *y* is therefore consistent with C_{60} adsorbed on two successive terraces, followed by C_{60} adsorbed in a pore. The last part of the profile follows the interior of one of the empty pores. The observation of this island, where C_{60} sits on both Pb-covered terraces and on pores is evidence that the material within the pores is chemically the same as the material on the terraces. Furthermore the C_{60} molecules form hexagonal structures, both on the flat part of the surface and within the pores. Therefore we conclude that the pores are also Pb-covered.

Fig. 6a shows LEED data recorded at 75 eV from the clean surface after the preparation procedure described in Section 2. Quasicrystals are aperiodic materials, which means that the reciprocal lattice is infinitely dense and fills reciprocal space; in practice LEED patterns are dominated by the most intense reflections. In this case several rings of spots of 10-fold symmetry are observable. The radii of the two most intense rings are related by a factor τ, which is indicative of quasiperiodic ordering. The LEED pattern after adsorption of 1 MLE is shown in Fig. 6b at the same beam energy. Again the diffraction spots are quasiperiodically spaced. The pattern is similar to that observed for the clean surface, although some

of the fainter spots in Fig. 6a are no longer visible. The LEED pattern recorded after annealing to 653 K is shown in Fig. 6c. Compared to the unannealed surface, the intensity of the spots has increased, suggesting an improvement in structural quality. There are also changes in the relative intensities of some of the rings, suggesting some structural changes may take place upon annealing. As the data for the clean surface and the Pb-dosed surface were taken with different instruments, quantitative comparison of Fig. 6a with b and c is not possible. However the FFTs from the clean surface STM data (not shown), the as deposited Pb film (Fig. 3b) and the annealed Pb film (Fig. 4b) show that the most intense rings of spots have the same radii and orientation.

The Al 2*p* and Pb 4*f* core levels were also recorded upon adsorption of a monolayer of Pb. Fig. 7a shows that the shape of the Al 2*p* core level measured is identical to that of the clean quasicrystal surface for the monolayer as deposited on the quasicrystal at room temperature or annealed to 653 K for 5 min. The Pb 4*f* core level recorded from the quasiperiodic Pb monolayer has an identical shape to that measured from one 1 MLE of Pb grown on Al(1 1 1) (Fig. 7b). The lack of new components and chemical shifts within the core level peaks is consistent with the immiscibility of Pb and Al [31,32]. No changes were detected in the Ni 2*p* core level either. A comparison of the ratio of the intensities of the Pb and Al photoemission peaks indicates that the density of Pb atoms is 0.09 atoms/$Å^2$, identical to that found on the i-Al–Pd–Mn surface and on the Al(1 1 1) surface. For comparison, the value found using LEED I(V) for the clean surface is 0.123 atoms/$Å^2$ [26].

The data from the diverse techniques presented above therefore present a coherent picture. STM and LEED show that Pb adopts a
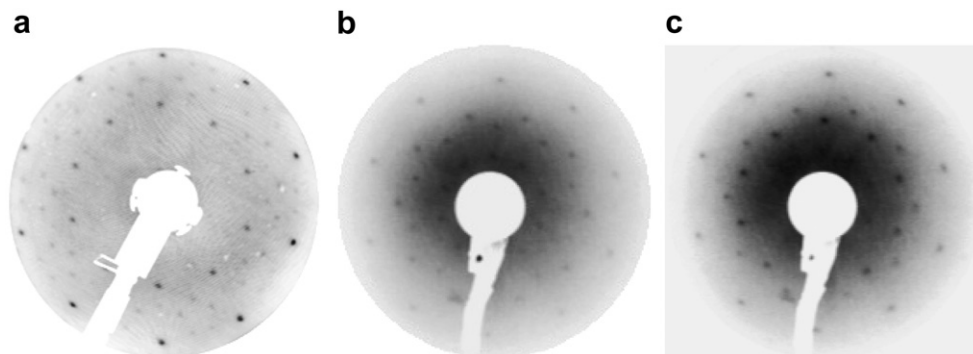
Fig. 6. LEED patterns recorded at 75 eV on (a) the clean surface, (b) with 1 MLE Pb as deposited on the d-Al–Ni–Co quasicrystal surface and (c) after annealing this film to 653 K.

a

b
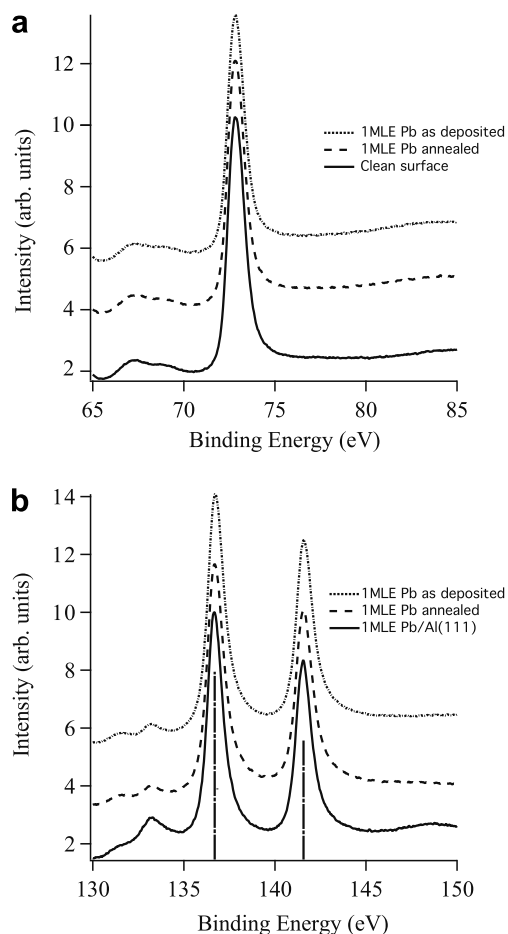
Fig. 7. (a) XPS spectra of the Al 2p core levels for the clean d-Al–Ni–Co surface (solid line), for 1 MLE of Pb as deposited (dotted line), and 1 MLE of Pb annealed to 653 K (dashed line); (b) XPS spectra of the Pb 4f core levels for 1 MLE of Pb adsorbed on Al(1 1 1) (solid line), 1 MLE of Pb as deposited on the d-Al–Ni–Co surface (dotted line) and 1 MLE after annealing to 653 K (dashed line). The markers at 136.6 and 141.5 eV indicate the position of the core levels for elemental Pb. The satellite peaks at low binding energy are due to the MgKα source.

quasiperiodic structure. AES, XPS and STM show that the coverage saturates at 1 MLE. Upon annealing, STM shows that the terraces develop pores. XPS indicates that Pb does not desorb upon annealing, and C$_{60}$ adsorption experiments indicate that the pores are also Pb-filled. LEED indicates some improvement in structural quality of the overlayer after annealing.

4. Discussion

The principal result is the observation of quasiperiodic ordering of Pb on the 10-fold d-Al–Ni–Co surface. It was not possible with STM to determine possible adsorption sites as this requires simultaneous observation of atomically resolved adsorption atoms and the underlying clean surface at sub-monolayer coverages. However the observation of 4.9 Å pentagonal clusters in the 1 MLE film suggests that these features are a key building block of a quasicrystalline monolayer, whether on an icosahedral or a decagonal quasicrystalline substrate [33].

It is also interesting that pentagonal clusters of two different orientations rotated by 36° are observed on a single terrace in the Pb film (Fig. 3a). In several STM studies of the clean d-Al–Ni–Co surface, on each terrace, 5-fold features of only a single orientation are observed [34,35,26,2]. However for Si adsorption on d-Al–Ni–Co [17], pentagonal clusters of both orientations are observed

on the same terrace. It appears that the inversion symmetry evident on successive terraces on the clean surface is broken when considering the structure of adsorbed layers.

The saturation in coverage at 1 MLE is similar to that found for Pb adsorption on i-Al–Pd–Mn [23]. This behaviour appears to be unusual for supported metal growth: for example at room temperature, Pb follows the Stranski–Krastanov growth mode on Si(1 1 1) [36]. This could be due to a low binding energy for Pb on the quasiperiodic Pb film, a possibility which could be investigated using density functional theory calculations of the adsorption of Pb atoms on a Pb monolayer. Such calculations are now quite feasible [37,38,29]. A second explanation is that the effect could be due to a limitation on the dissipation of the kinetic energy of the adsorbing Pb atoms leading to a vanishing sticking coefficient for Pb on the quasiperiodic Pb monolayer. A low sticking coefficient could result from a high phonon frequency for energy transfer from impinging Pb atoms to the quasicrystalline surface Pb layer [23].

There are other similarities and differences between the d-Al–Ni–Co/Pb and i-Al–Pd–Mn/Pb systems. During growth extended Pb islands are observed on d-Al–Ni–Co, whereas on i-Al–Pd–Mn/Pb a dispersed network of pentagonal clusters is clearly seen. This indicates higher mobility for adsorbing atoms on the former substrate, which is supported by the observation of tip-induced diffusion at the island edges during growth. Annealing improves the order of the overlayer on both substrates, although in the case of d-Al–Ni–Co this produces shallow pores in the Pb-covered terraces. These pores have been shown to also contain Pb. The production of these pores clearly involves a significant restructuring process. However the details of this process are not determined from the data available in this study.

5. Concluding remarks

Lead has been found to form a well-ordered quasiperiodic overlayer on the 10-fold surface of the decagonal Al–Ni–Co quasicrystal. This work extends the number of known systems where an adsorbate adsorbs pseudomorphically on a quasicrystal substrate. It is now apparent that chemistry is one of the most important factors in determining adsorbate structure: the elements which form pseudomorphic quasiperiodic overlayers are clustered in Groups III–V of the periodic table [7]. Further studies are needed to determine the structure of these overlayers; they present a unique challenge to current quantitative surface structural techniques.

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