Growth morphology of Pb films on Ni$_3$Al(111)

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Abstract
Scanning tunnelling microscopy (STM) and Auger electron spectroscopy (AES) have been used to investigate the growth morphology of ultra-thin Pb films on the Ni$_3$Al(111) face at room temperature. A previous study [Miśków K, Krupski A. Appl Surf Sci 2013;273:554] using low-energy electron diffraction (LEED) and real time Auger intensity recording has demonstrated that an initial two-dimensional growth of the first Pb monolayer thick ‘wetting layer’ takes place. With further deposition and for T = 300 K, flat three atomic-layer-high islands are grown. Above 350 K, the Stranski–Krastanov growth mode was observed. In the current study, the analysis of STM measurements indicate and confirm that for coverage $\theta = 1.0$ ML two-dimensional growth of the first Pb monolayer took place. Above $\theta > 1.0$ ML, a three-dimensional growth of the Pb islands was observed with a strongly preferred atomic scale ‘magic height (N),’ hexagonal shape and flat-tops. At coverage $\theta = 3.5$ ML, only islands containing N = 3, 5, 7 and 11 atomic layers of Pb are observed. At the higher coverage $\theta = 5.5$ ML, three types of regular hexagonal islands with side lengths of 25, 30 and 45 nm are observed. Furthermore, three different island adsorption configurations rotated by ±1° and 30° ± 6° with respect to each other were observed. After an annealing at T = 400 K of 5.5 ML of lead deposited at RT on the Ni$_3$Al(111) the morphology of the surface changes. Post-anneal, islands of Pb are observed above the ‘wetting layer’ with an estimated average size and diameter of 768 nm $^2 \pm$ 291 nm $^2$ and 38.17 $\pm$ 6.56 nm and constant uniform height of two atomic layers (N = 2).

Keywords:
Lead; Nickel; Aluminium; Alumina; Crystal growth; Thin film growth; Metal–metal interfaces; Low index single crystal surface; Crystalline structure; Auger electron spectroscopy (AES); Scanning tunneling microscopy (STM).
1. Introduction

Recently a novel growth mode has been observed in epitaxial growth of Pb on Si(111) [1-13], Ni(111) [14-16], Cu(111) [13,17-18] and Mo(110) [19-20] surfaces. Pb islands grow to specific heights ‘magic heights,’ which are stable due to quantum size effects (QSE) [21-24] associated with electron confinement in the vertical direction within the islands. The growth conditions of temperature and flux when these uniform height islands are observed is different for the different substrates which shows that kinetic factors play a role.

The adsorption of Pb on Ni$_3$Al(111) is an additional system used to understand the interplay between kinetics and electronic effects in height selection. The system has been investigated both theoretically [25] and experimentally [26]. Kośmider [25] studied adsorption of CO molecules and Pb atoms on Ni(111) and Ni$_3$Al(111) substrates using *ab initio* density functional theory (DFT) calculations. The calculations suggested that Pb atoms would adsorb on a stoichiometric Ni$_3$Al(111) surface preferentially on the threefold fcc ($E_b = 3.98$ eV) and hcp ($E_b = 3.75$ eV) sites, both being about 1 eV energetically more favorable than the top$_{Al}$ position ($E_b = 2.92$ eV). In contrast, it was shown that the top$_{Ni}$ site is unstable.

Experimentally [26] there was a study of the atomic structure and morphology of ultrathin Pb layers deposited on the Ni$_3$Al(111) face. Films were deposited in ultrahigh vacuum at substrate temperatures ranging from 200 K to 950 K. Auger electron spectroscopy, low-energy electron diffraction (LEED) and directional elastic peak electron spectroscopy (DEPES) were used for in-situ characterization. The analysis of AES measurements showed that a two-dimensional growth of the first Pb monolayer took place for substrate temperatures $200 \, \text{K} \leq T \leq 650 \, \text{K}$. For $T = 200 \, \text{K}$, Pb on the Ni$_3$Al(111) grows layer-by-layer, while for $T = 300 \, \text{K}$ flat three atomic-layer-high islands are seen to grow after the completion of the first lead monolayer. Above 350 K, Stranski–Krastanov growth mode was observed. Ordered LEED patterns corresponding to $p(\sqrt{3} \times \sqrt{3})R30^\circ$ and $p(4 \times 4)$ structures for coverages $\Theta < 0.6$
ML and 0.7 ML < Θ < 2.4 ML were observed, respectively. AES and LEED data indicated the formation of a surface alloy between lead atoms and the Ni₃Al(111) face. DEPES results show that a stacking fault is formed in the Ni₃Al(111)-Pb interface region.

The present work investigates the growth morphology of lead adsorbed on Ni₃Al(111) surfaces in ultrahigh vacuum at room temperature. These results were obtained using of STM and AES.

2. Experiment

The experiments were performed in two different UHV chambers, each with a base pressure below 2.0×10⁻⁸ Pa. The Ni₃Al(111) single crystal was supplied by MaTeck, Jülich. In this paper, AES(t) measurements were done in Wroclaw with reverse view LEED optics (OCI Vacuum Micro engineering) while STM images were taken in Bonn using a home-built liquid nitrogen cooled beetle type STM [27]. Owing to the geometry of the Wroclaw system it was possible to record continuously in time the Auger peak heights during the deposition of lead onto the sample surface, under computer control, for the nickel MVV transition at 61eV and the lead NVV transition at 94 eV. This possibility allows to follow atom rearrangement on the surface in real time especially if very fast processes operate. The nickel Auger peak height was corrected for background, created by the secondary electrons, in the low-energy part of the dN/dE versus electron energy curve. In our experiments, the metal deposition is not interrupted for Auger peak measurement. Therefore, the AES(t) plots continuous evolution of the deposited layer. Plotting the AES peak heights of the substrate and of the adsorbate as a function of deposition time (AES(t) plots) enables the determination of the growth mechanism as well as monolayer formation [16, 20, 26, 28-33]. Namely, if α_S = h_SI/ h_S0 defines the coefficient of attenuation of the substrate Auger peak, owing to the presence of a monolayer of adsorbate, then the expected height of the substrate Auger peak (h_{Sn}) for layer-by-layer
growth (Frank–van der Merwe (FM)) [34-35], after completion of the \( n = 2^{nd}, 3^{rd}, 4^{th}, \ldots \) layer is given by the equation

\[
h_{Sn} = h_{S0} (\alpha S) \alpha^{n} = h_{S0} (h_{S1}/h_{S0})^{n} = (h_{S1})^{n},
\]

where \( (h_{S0}) = 1 \), and corresponds to the height of the substrate Auger peak without adsorbate \( (n = 0) \). Equation (1) enables also calculation of the expected height of the substrate Auger peak \( (h_{Sn}) \) for the growth of flat islands composed of \( l = 2, 3, 4, \ldots \) atomic layers [16, 26, 33], as it is presented in Fig. 1. The variable temperature VT-STM was constructed using components copied from the original design by Stipe et al. [36]. The clean and ordered \( \text{Ni}_{3}\text{Al}(111) \) surface was prepared by repeated cycles of neon ion sputtering, at a sample temperature of 600K (40 min, 500 eV, 9 \( \mu \)A), with subsequent annealing to 1150 K (7 min) and 1000 K (7 min), respectively. This procedure was repeated until the carbon peak was no longer visible in the AES spectrum, and a LEED pattern with sharp spots and low background appeared. Lead (99.999\%) was evaporated onto the crystal surfaces from the same quartz crucible surrounded by a tungsten resistive heater in a vacuum of \( 5.0 \times 10^{-8} \) Pa or better, and at a deposition rate of \( 1.46 \times 10^{-3} \) ML/s. All STM measurements were performed on the same sample at appropriate deposition times (marked by blue arrows as “STM” in Fig. 2(a)) at room temperature with W tips, in constant current mode. STM data were processed by freeware image-processing software [37].

3. Results and discussion

For the growth of lead on \( \text{Ni}_{3}\text{Al}(111) \) at room temperature, one linear component of the AES(t) plots for both 61 eV nickel and 94 eV lead peaks was observed, see Fig. 2(a). This component of the AES(t) plots indicates a two-dimensional (2D) growth of the first lead layer. After the first monolayer, the quasi-linear shape of the second region of the AES(t) suggested that a two-dimensional growth continued that was not of the standard Frank – van der Merwe (FM) type. In this region the calculated AES(t) plot for a substrate with FM
growth did not fit the experimental data (see curve (1) in Fig. 2(a)). A much better fit was obtained, until the 2.5 ML, for an AES(t) plot calculated for the growth of flat Pb islands composed of three atomic layers (curve (3) in Fig. 2(a)). Curves (1)-(4) in Fig. 2(a) were calculated using the formula described in the experimental part of the present paper, under the assumption that the first lead layer was completed at \((h_{S1}; t_1) = (0.56\pm0.03; 685\pm88)\). The scatter of \(h_{S1}\) and \(t_1\) values in room temperature AES(t) plots was not large, thus, it will be further assumed that the first linear region of the curve corresponded to the formation of the first layer \((\theta = 1.0\text{ ML})\) of lead, where a 1 ML Pb(111) film corresponded to an atomic packing density of \(9.43\times10^{14}\) atoms/cm\(^2\). Conversely, for growth at a deposition temperature \(T = 400\text{ K}\), Fig. 2(b), two linear regions were clear in the AES(t) plots that corresponded to the first monolayer formation and to uniform Pb island growth. The traces are flatter and remain constant with Pb deposition indicating that the relative fractions of areas with Pb and areas with the wetting layer must be unchanged so growth is 3DIM and of the same height. Otherwise the Pb intensity should be increasing monotonically and the substrate intensity decay to a very low level. Fig. 3(a) displays an STM image, taken on a low-index Ni\(_3\)Al(111) substrate with terraces between 29 and 120 nm wide separated by monoatomic steps. The height of the steps on the Ni\(_3\)Al(111) surface were measured by STM to be \(2.06 \pm 0.3\) Å, see Fig. 3(b). STM images of Ni\(_3\)Al(111) surfaces with various Pb coverages presented in Fig. 3, Fig. 4 and Fig. 5, illustrate the evolution of growth morphology of the Pb layer deposited on Ni\(_3\)Al(111) at room temperature. Fig. 3(c) shows a typical STM image corresponding to the submonolayer coverage of \(\theta = 0.5\) ML. The bright features represent lead islands. Height profile (B), taken across the Pb islands (Fig. 3(d)), show that the height and estimated average diameter of the islands was \(5.72\) Å, \(11.44\) Å and \(5.8 \pm 0.6\) nm \(7.3 \pm 0.9\) nm, respectively. These Pb island heights correspond to 2 and 4 atomic layers of Pb(111). It is suggested that the observed conical shape of lead islands on the substrate was a consequence
of tip convolution and the finite island size [38-39]. At sample temperatures between 200 and 300 K, for coverage \( \Theta < 0.6 \text{ ML} \), the structure \( p(\sqrt{3}\times\sqrt{3})R30^\circ \) was observed in LEED [26]. As Pb coverage approached \( \theta = 1.0 \text{ ML} \), Pb was seen to wet the \( \text{Ni}_3\text{Al}(111) \) surface almost completely, as can be seen in Fig. 3(e). At this coverage only LEED spots associated with the \( p(4\times4) \) structure were observed [26]. The density of Pb atoms of the \( p(4\times4) \) structure is known to be \( 10.42\times10^{14} \text{ atom/cm}^2 \), which is 110\% of the density of the close-packed (111) Pb layer. The almost perfect wetting in this system is due to the high specific surface free energy of the \( \text{Ni}_3\text{Al}(111) \) surface as compared with that of the Pb(111) surface. Since the total specific surface free energy should be minimised, a covered \( \text{Ni}_3\text{Al}(111) \) surface is favoured. As seen in the inset of Fig. 3(e), the first Pb layer on the \( \text{Ni}_3\text{Al}(111) \) surface was not free from dislocation defects. The arrows in Fig. 3(e)-(f) indicate areas where holes with an average depth of \( d_h = 1.02 \pm 0.35 \text{ Å} \) in the ‘wetting layer’ are observed. The two-dimensional growth of the first Pb ‘wetting layer’ is thermodynamically driven by the lower surface free energy of Pb \( (0.377 \text{ J/m}^2 < \gamma_{\text{Pb}} < 0.600 \text{ J/m}^2) \) [40-43] compared to \( \text{Ni}_3\text{Al} \) \( (\gamma_{\text{Ni}_3\text{Al}} \approx 1.887 \text{ J/m}^2) \) [44]. This wetting is still favorable despite the elastic energy resulting from the large lattice mismatch about 24\% between \( p(4\times4)\)-Pb structure and \( \text{Ni}_3\text{Al}(111) \) surface. A similar ‘wetting layer’ growth with dislocations for Pb adsorption on Mo(110) has been observed [19]. The presence of compressed wetting layers with density higher than the metallic Pb(111) density by approximately 5\% has been identified as the key factor that provides unusually fast and non-classical kinetics in the uniform height island [11,14]. It is not unusual to expect that the \( p(4\times4) \) phase plays the same role and superfast diffusion can be present in this system as well. At coverages of \( \Theta > 1 \text{ ML} \), three-dimensional growth (Stranski-Krastanov) is assumed, as can be seen in Fig. 4 and Fig 5(a). Figure 4 shows STM images taken after 3.5 ML of Pb was deposited on the \( \text{Ni}_3\text{Al}(111) \) surface at room temperature. Height profile (D) taken across the Pb islands where the Pb islands had a hexagonal shape, flat top and thickness of about 9,
14, 20, or 32 Å in Figure 4(b) is presented. The interlayer spacing along the [111] direction of Pb was measured to be $d_{111} = 2.86$ Å and therefore the islands had a thickness corresponding to three, five, seven or eleven atomic layers, respectively. In Fig. 4(a), the heights of the Pb islands in atomic layers are indicated. Closer inspection of the flat tops of Pb islands, smaller Pb islands were observed distributed in an ordered fashion, see Fig. 4(c). The white arrows in Fig. 4(c) point to the smaller Pb islands in the beginning stages of forming two atomic layer rings with diameter $9 \pm 0.41$ nm. Height profile (E) was taken across these smaller Pb islands and the height of these were measured to be $5.72 \pm 0.3$ and $2.86 \pm 0.3$ Å. The height distribution of Pb islands corresponding to Fig. 4(a) is presented in Fig. 4(e). As Pb coverage was increased to 5.5 ML, three types of regular hexagonal islands with length side 25, 30 and 45 nm are measured (Fig. 5(a)). Smaller irregularly shaped Pb islands were also observed between the main regular hexagonal islands. Furthermore, three different adsorption configurations of Pb are distinguishable, rotated by $\alpha = 10^\circ \pm 1^\circ$ and $\beta = 30^\circ \pm 6^\circ$ with respect to each other. It should be noted that the different orientation of these hexagonal Pb islands is not correlated with their size. The presence of these multilayer lead islands agreed with the island growth deduced from AES analysis at room temperature [26]. During post-deposition annealing ($T = 400$ K, 60 sec.) of Pb films ($\theta = 5.5$ ML), a change of the height of the lead ‘magic islands’ was observed as seen in Fig. 5(c)-(d). Only islands with the estimated average size and diameter of $768$ nm $^2 \pm 291$ nm $^2$ and $38.17 \pm 6.56$ nm containing two atomic layers of Pb are observed above the ‘wetting layer.’ The heights of the Pb islands in atomic layers are indicated in Fig 5(c) with the corresponding height distribution seen in Fig. 5(e). LEED patterns corresponding only to weakly ordered $p(4 \times 4)$-Pb structure were observed after post-deposition annealing. These STM results confirm our present AES studies at $T = 400$ K concerning a 3DIM growth mode of the same height above the ‘wetting layer.’ Table 1 shows the addition of the Pb-Ni$_3$Al(111) adsorption system to the known group of systems where
uniform island height selection during metal thin film growth has already been observed and interpreted in terms of quantum size effects (QSE) [21-24]. Typically, these electronic effects have been observed on semiconducting (e.g. Pb/Si(111) [1-13], Pb/Ge(111) [8] or Ag/GaAs(110) [45]) or metal substrates (e.g. Ag/Fe(100) [46], Pb/Ni(111) [14-16], Pb/Cu(111) [13,17-18] or Pb/Mo(110) [19-20]). For example, scanning tunnelling microscopy and spectroscopy observations of Pb islands on the Cu(111) surface face [18] indicated the equilibrium distribution of island heights showed some heights appearing much more frequently than others. In all these systems a confining barrier restricts the electron to be within the film because of the misaligned electronic bands in the metal film and the substrate so no states are available to move from the film to the substrate. ‘Magic’ preferred heights correspond to islands with a quantum well state far from the Fermi energy, while the ‘forbidden’ heights appear to be those that have a quantum well state close to the Fermi level. Another typical example of uniform island growth is Pb deposited on Si(111) at a temperature between 170 and 250 K and typically results in the formation of Pb islands with N = 4(unstable), 5, 6, 7 and 9 atomic layer heights above the wetting layer. Nevertheless, the occurrence of ‘magic heights’ is related to the increased stability associated with islands of specific thickness.

4. Conclusions

In summary, the growth behaviour of ultra-thin Pb films on Ni₃Al (111) surfaces at room temperature was investigated by means of Auger electron spectroscopy and scanning tunnelling microscopy. Two-dimensional growth of the first lead layer was observed agreeing with previous studies [26]. For submonolayer coverage (θ = 0.5 ML), lead nucleated randomly and created either two or four atom high islands with a conical shape on Ni₃Al (111) terraces. The average diameter of the observed lead islands at θ = 0.5 ML was 5.8 ± 0.6 nm and 7.3 ± 0.9 nm for the 2 and 4 atom high islands, respectively. Coverages θ > 1 ML
showed a transition three-dimensional growth of the Pb islands with a strongly preferred atomic-scale ‘magic height’ and flat top. At coverages $\theta = 3.5$ ML, STM images indicated growth of three, five, seven and eleven-atom high lead islands with a hexagonal shape on top of the first lead layer ‘wetting layer.’ At the higher coverage $\theta = 5.5$ ML, three types of regular hexagonal islands were seen with sides of 25, 30 or 45 nm. In addition, three different adsorption configurations of Pb were distinguishable, rotated by approximately 10° or 30° with respect to each other. After annealing at 400 K, the morphology of the surface of 5.5 ML of lead deposited on the Ni$_3$Al (111) changes. The island height distribution exhibited a peak at the relative height corresponding to $N = 2$ Pb atomic layers above the ‘wetting layer.’ The estimated average area and diameter of the two-atomic lead islands were $768 \pm 291$ nm$^2$ and $38.17 \pm 6.56$ nm, respectively. Future theoretical and experimental work is required to study the electronic structure and stability of these uniform Pb ‘magic islands’ on Ni$_3$Al(111). This type of growth could have impact on the engineering of stable materials and devices with nanometer-scale dimensions.

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References


Caption of figures

Fig. 1. (a) Schematic view of layer-by-layer growth (1), two- (2), three- (3), and four (4) atomic layer thickness flat islands growth on the first layer, respectively as a function of deposition time. (b) AES(t) substrate plots expected for the growth modes presented in (a).

Fig. 2. AES(t) plots of Ni MVV and Pb NVV peak heights for lead deposition on Ni$_3$Al(111) face at: (a) T = 300 K, and (b) T = 400 K. (1) – AES(t) plot calculated for Frank–van der Merwe growth. (2), (3) and (4) – AES(t) plots calculated for the growth of two-, three- and four atomic layer thickness flat islands on the first lead layer, respectively.

Fig. 3. STM images of Pb deposited on Ni$_3$Al(111) at T = 300 K at coverage $\theta \leq 1.0$ ML: (a) clean Ni$_3$Al(111) (2330 Å × 2330 Å, $I_T = 125$ pA, $U_{bias} = 1.8$ V); (b) The line scan A shows terraces approximately between 29 and 120 nm wide; (c) $\theta = 0.50$ ML, $(h_{Pb}/h_{Ni})_{AES} = 0.27$ (2070 Å × 2070 Å, $I_T = 123$ pA, $U_{bias} = 1.85$ V), the height of the Pb islands corresponds to 2 and 4 atomic layers of Pb(111); (d) Height profile along line B from the image in (c) demonstrating that the height of the conical shape islands corresponds to 5.72 and 11.44 Å, respectively; (e) $\theta = 1.00$ ML, $(h_{Pb}/h_{Ni})_{AES} = 0.80$ (2070 Å × 2070 Å, $I_T = 122$ pA, $U_{bias} = 2.0$ V); The inset presents zoom (392 Å ×392 Å) in the area marked by a square. Arrows indicate holes in the wetting layer; (f) Height profile along line C from the image in (e) showing that the depth of the holes is $d_h = 1.02 \pm 0.35$ Å.

Fig. 4. STM images of Pb deposited on Ni$_3$Al(111) at T = 300 K at coverage $\theta = 3.5$ ML, $(h_{Pb}/h_{Ni})_{AES} = 1.99$: (a) (1520 Å × 1520 Å, $I_T = 100$ pA, $U_{bias} = 3.2$ V). The height of the Pb islands corresponds to 3, 5, 7 and 11 atomic layers of Pb(111). Hexagonal shape of Pb islands is marked; (b) Height profile along line D from the image in (a); (c) (582 Å × 582 Å, $I_T = 100$ pA, $U_{bias} = 3.2$ V). An arrow indicates set of small Pb islands growing on diameter 9 nm; (d) Height profile along line E from the image in (c) demonstrating presence of additional smaller Pb islands (height 5.72 and 2.86 Å, respectively) on the flat tops of Pb islands, (e) a height distribution of Pb islands corresponding to image (a).

Fig. 5. STM images of Pb deposited on Ni$_3$Al(111) at T = 300 K at coverage $\theta = 5.5$ ML, $(h_{Pb}/h_{Ni})_{AES} = 2.50$: (a) (4400 Å × 4400 Å, $I_T = 146$ pA, $U_{bias} = 7.1$ V). The Pb islands have regular hexagonal shape I, II and III with the length of the side 25, 30 and 45 nm, respectively. The different orientation of the Pb islands with respect to each other is marked; (b) Height profile along line F from the image in (a); (c) post annealed at T = 400 K, $(h_{Pb}/h_{Ni})_{AES} = 0.85$ (6600 Å × 6600 Å, $I_T = 130$ pA, $U_{bias} = 7.6$ V). The height of the Pb islands corresponds to 2 atomic layers of Pb(111) is measured from the wetting layer. The inset present differentiated STM images with enhanced contrast; (d) Height profile along line G from the image in (c), (e) a height distribution of Pb islands corresponding to image (c).
Caption of table 1

The table summarizes the observed layer thicknesses (N) of ‘magic height’ Pb islands for different adsorption systems. Layer thicknesses are measured with respect to ‘wetting layer.’ (u) – describes unstable Pb islands. The exact island height distribution depends on the substrate reconstruction, deposited amount, growth conditions and annealing sample history, coarsening times etc. The sharpest Island height distribution is obtained on Si(111)-7×7 with a practically a δ-function observed of 7-layer height [2].
FIGURES

FIG. 1.

(a)

(b)
FIG. 2.

(a)

(b)
FIG. 3.

(a)  

(b)  

(c)  

(d)  

(e)  

(f)
FIG. 4.

(a) (b)

(c) (d)

(e)
Fig. 5.

(a) 

(b) 

(c) 

(d) 

(e)
<table>
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<tr>
<th>Pb / Substrate</th>
<th>Layer Thickness (N)</th>
<th>Temperature (K)</th>
<th>Methods</th>
<th>Ref.</th>
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<td>240</td>
<td>STM</td>
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<td>$T &gt; 175$</td>
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<td>[14,15]</td>
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<td>AES, LEED, DEPES</td>
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