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Selective Penetration of Metal Atoms — New Evidence and Application for the Simple Ideal Penetration Model of the Long-Chain Close-Packed Alkanethiol Self-Assembled Monolayers on Au(111)

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Abstract

In the study of the vapor deposition of metal atoms onto long-chain alkanethiol self-assembled monolayers (CnSH-SAMs, n>10) on Au(111) substrate, metal atoms with different diameters can penetrate selectively into the CnSH-SAMs or not, which looks like the “size exclusion effect”. In this paper, we proposed a new mechanism for the metal atoms penetration by establishing a simple ideal penetration model of the long-chain CnSH-SAMs with defect-free structure on Au(111). The intrinsic and regular-distributed channels with ~ 3 Å diameters (which are decided by the framework size of the three-aggregate of the CnSH molecules) were deduced. This new penetration mechanism can explain reasonably the selective penetration of metal atoms into the long-chain CnSH-SAMs on Au(111): When the driving force is applied, the metal atoms with diameter smaller than 3 Å will overcome the van der Waals interaction among the neighboring hydrocarbon chains and penetrate into the CnSH-SAMs through the ~ 3 Å channels reaching to the surface of Au(111), while the metal atoms with large diameter (> 3 Å) can not penetrate.

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1. Introduction

The study of the vapor deposition of metal atoms onto alkanethiol self-assembled monolayers (CnSH-SAMs) possesses crucial scientific importance for the development of the molecule–based devices [1–8]. The metal atoms with small size (e.g. Al, Au, Ag, etc.) can penetrate into the CnSH-SAMs on Au(111), and the penetration mechanism is commonly proposed to occur via the “common defect (e.g. pinhole, gauche defect, domain boundary and other diffusion paths) in the SAMs” [9–10] or the “transient defect (transient appearance of holes) caused by the hopping of the adsorbate molecules on the Au substrate lattice” [1–8]. For the former penetration mechanism,
we believe that it is only suitable for the SAMs formed on the substrate with poor pretreatment or the SAMs with disorder structure, but not suitable for the close-packed SAMs with defect-free structure. For the later penetration mechanism, we believe that it is only suitable for the short-chain C\textsubscript{n}SH-SAMs (n \leq 10) because of the sulfur-gold interaction is stronger than the intermolecular interactions of alkanethiols in short-chain SAMs [11–13], which will lead to the hopping of the adsorbate molecules on the gold substrate lattice, and then the holes (defects) appeared transiently. These transit defects are nanometer-scale [1] and sufficiently large to provide enough spaces for metal atom penetration or diffusion to the S/Au interface. However, for long-chain alkanethiols (C\textsubscript{n}SH, n>10), intermolecular interactions dominated and availed the formation of closely packed surface structure [11–13]. Therefore, there will be no transient defect in the long-chain C\textsubscript{n}SH-SAMs and the later permeation mechanism is not suitable for them. In this paper, a simple ideal penetration model was established in order to clarify the permeation mechanism of the metal atoms on the long-chain C\textsubscript{n}SH-SAMs with defect-free structure on Au(111).

2. Results and discussion

2.1 Model description

The simple ideal penetration model was established by the similar principle reported in our previous work [14]:

(1) long-chain alkanethiol hexadecanethiol (C\textsubscript{16}SH) was used as an example to calculate the size of C\textsubscript{n}SH molecule. Figure 1 gives the two proposed expressions for the size of C\textsubscript{16}SH molecule: van der Waals size and framework size [15–18]. Taking into account the covalent radius of the hydrogen atom (0.30 Å) [19], the C\textsubscript{16}SH molecule is considered approximately as a cylinder with the diameter of 2.8 Å (framework size), which is much smaller than the van der Waals size of C\textsubscript{n}SH molecule (4.3 Å) [14].

Figure 1 Schematic diagram of C\textsubscript{16}SH-SAMs on Au(111) substrate. C\textsubscript{16}SH molecule structure is optimized by Gaussian98 program using density functional (DFT) at B3LYP/6-311G+(d,p) level [14]). Van der Waals size: the van der Waals size can be considered as a circle with the diameter of 4.3 Å [14]. Framework size: the distances of the neighbor hydrogen atom in the planform of C\textsubscript{16}SH molecule were calculated to be 1.8 Å and 2.2 Å using GaussView 3.0 based on the covalent radius, respectively. Taking into account the framework size of the hydrogen atom (0.3 Å) [19], the framework size of the C\textsubscript{16}SH molecule can be approximately calculated to be 2.4 Å × 2.8 Å. For simplicity, the C\textsubscript{16}SH molecule studied in our work is considered approximately as a cylinder with the diameter of 2.8 Å. The tilt angle of 25° is chosen in this figure.

(2) Figure 2 shows the schematic diagram of the surface structure for C\textsubscript{16}SH molecules on Au (111) and it’s surface parameters were calculated (Table 1) [20–23], and then the intrinsic and regular–distributed penetration channels with ~ 3 Å diameter (which is decided by the framework size of the three-aggregate of the C\textsubscript{n}SH molecules) in the long-chain C\textsubscript{n}SH-SAMs were deduced. Therefore, we can conclude that the metal atoms with small diameter (< ~ 3 Å) have the possibility to penetrate into the SAMs, whereas the metal atoms with large diameter (> 3 Å) can not penetrate.
Gold atom; C_{16}SH in van der Waals size; Channel; C_{16}SH in framework size;

Figure 2 Schematic illustration of the C_{16}SH-SAMs on Au(111). For clarity, the chain tilt and rotation are ignored. Hereby, the S head groups are arranged in a ($\sqrt{3} \times \sqrt{3}$) R30° structure, $a = b = 5.0$ Å, $a = 120°$.

Table 1 Summary of the surface characteristic parameters for the long-chain C_{n}SH-SAMs on Au(111) (Channel diameters $d_c$ in framework size).

| Items                               | Parameters                                      |
|-------------------------------------|-------------------------------------------------|
| Unit mesh                           | $a=b=5.0$ Å, $a=120°$                           |
| Lattice ($\sqrt{3} \times \sqrt{3}$)R30° | $a=5.0$ Å, $a=120°$                            |
| Chain tilt ($\varphi$)              | 20–30°                                          |
| Experimental surface coverage ($I_e^d$) | $\sim 7.9 \times 10^{-10}$ mol/cm$^2$         |
| Theoretical surface coverage ($I_t^d$) | $7.6 \times 10^{-10}$ mol/cm$^2$               |
| C_{n}SH molecule number ($N_m$)     | 4.6$\times 10^{14}$ per cm$^2$                 |
| Channel number ($N_c$)              | 9.2$\times 10^{14}$ per cm$^2$                 |
| Channel diameter ($d_c$)            | $3.2$ Å$^f$                                     |
| Ignore the chain tilt ($\varphi = 0°$) |                                                  |
| Consider the chain tilt ($\varphi = 25°$) | $2.9$ Å$^g$                                     |
| Measured from STM/AFM images        | $\sim 3$ Å$^{[14]}$                             |

[a] $a$ and $b$ are the magnitudes of the real space unit mesh parameters, $a$ is the angle between the neighbour unit mesh vectors $a$ and $b$ $^{[20]}$.
[b] The fcc hollow site (alkanethiol molecule adsorption on the hollow of three gold atoms) is adopted to draw the simple ideal model $^{[21]}$.
[c] The $\varphi$ is obtained from IR spectroscopic and ellipsometric data $^{[22]}$.
[d] The $I_e$ is obtained from C_{12}SH-SAMs on Au(111) $^{[23]}$.
[e] The $I_t$ is calculated from the area per alkanethiol molecule on Au(111) ($21.7$ Å$^2$) $^{[20]}$.
[f] Channel diameter $d_0$ is the diameter of the dash circle in Figure 2 (B).
[g] Under this condition, channel diameter $d_c = d_0 \cos \varphi = d_0 \cos 25°$.

(3) The penetration process can be clearly understood by this simple ideal model as shown in Figure 3. When there is no driving force, the van der Waals interactions among the neighboring hydrocarbon chains block the channels, thus the metal atoms whose diameters larger than 1.4 Å (Figure 2 (A)) can not penetrate into the SAMs; When the driving force is applied (e.g. under a certain vapor pressure, thermally evaporated metal atoms will reach the C_{n}SH-SAMs surface), the metal atoms with small diameter ($< \sim 3$ Å) will overcome the van der Waals interactions among the neighbouring hydrocarbon chains and penetrate into the C_{n}SH-SAMs through the $\sim 3$ Å channels reaching to the Au(111) surface, then form a 1:1 metal adlayer between the Au substrate and the C_{n}SH-SAM $^{[1]}$. 
2.2 The explanation of the experimental phenomena in the vapor deposition process of metal atoms on the C_nSH-SAMs on Au(111) by this simple ideal penetration model

This new penetration mechanism can easily explain the following experimental phenomena in the vapor deposition process of metal atoms on the C_nSH-SAMs on Au(111):

2.2.1 Selective deposition of different metal atoms on the long-chain C_nSH-SAMs

Table 2 lists the metal atomic diameter \(d_m\) of different metal atoms [19] and penetration behaviors of these metal atoms in the vapor deposition process on the long-chain C_nSH-SAMs on Au(111). From Table 2, one can see that the metal atoms with relatively small \(d_m\), such as Al (2.86 Å) [1], Ag (2.88 Å) [9,10], Au (2.88 Å) [3], Cu (2.56 Å) [24] and Hg (3.02 Å) [5,25], can penetrate into the long-chain C_nSH-SAMs. However, metal atoms with relatively large \(d_m\) can not penetrate (e.g. Na (3.72 Å) [6] and K (4.64 Å) [7]). Considering that the metal atoms have become condensed matter when they reach the surface of the C_nSH-SAMs and form a metallic film, therefore, the diameter of metal atoms in vapor phase and condensed matter state is different. Literature reported that van der Waals diameters of metal atoms are usually much longer than those corresponding metal atoms in condensed matter [26], then \(d_m\) can be used to estimate the penetration ability of the metal atom, whereas the van der Waals diameter of metal atom is not suitable [27]. The different penetration behaviors of the metal atoms with different \(d_m\) values on the long-chain C_nSH-SAMs are just like the “size exclusion effect” [28] and ~ 3 Å might be the critical diameter for the metal atoms to penetrate. Since both the common defect and transient defect proposed by the previous two penetration mechanisms as penetration channels are all nanometer-scale [1,14], which is large enough for all metal atoms to penetrate (Table 2), therefore, there will be no “size exclusion effect” based on these two “defects” penetration mechanisms. The diameter of the intrinsic penetration channels (~ 3 Å) in the long-chain C_nSH-SAMs deduced from our simple ideal penetration model is in good agreement with the critical diameter (~ 3 Å) for the metal atoms to penetrate reported by literature. Therefore, this “size exclusion effect” can be explained easily by the new penetration mechanism proposed by us.

2.2.2 Formation of a highly uniform ~ 1:1 Al adlayer on Au when the Al doses up to ~ 12 atoms/nm²

There are 8 channels and 4 alkanethiol molecules in the same area of 10 Å × 10 Å × sin60° (0.87 nm²) (Figure 2(B)). These 8 Al atoms can occupy firstly the bottom of the channels and then 4 Al atoms will deposit continuously under the alkanethiol molecules and replace them. Finally there will be 12 Al atoms locating in this 0.87 nm² area. It is in agreement with the literature reported that a highly uniform ~ 1:1 Al adlayer on Au was formed when total Al doses up to ~ 12 atoms/nm² [1]. This can also be proved by secondary ion mass spectrometry (SIMS). When the average dose of Al (dAl) is < 1 Å, 8 Al atoms are in the 8 channels and only Al⁺ and Al₂⁺ peaks occur. When 1 Å < dAl < 2 Å, Al–S peak occurs, indicating that the Au–S bond is replacing by the Al–S; When
Table 2 The metal atomic diameter ($d_m$) of metal atoms and the penetration behaviors of these metal atoms on the long-chain C$_n$SH-SAMs on Au(111).

| Atoms | $d_m$(Å)[19] | C$_n$SH-SAMs | Penetration behaviors |
|-------|--------------|--------------|----------------------|
| Li    | 3.04         | –            | –                    |
| Na$^+$| 3.72         | C$_{22}$SH-SAMs | × [6]               |
| K$^+$ | 4.64         | C$_{16}$SH-SAMs | × [7]               |
| Rb$^+$| 4.96         | –            | –                    |
| Cs$^+$| 5.30         | –            | –                    |
| Be    | 2.23         | –            | –                    |
| Mg    | 3.20         | –            | –                    |
| Ca    | 3.94         | –            | –                    |
| Sr    | 4.30         | –            | –                    |
| Ba    | 4.35         | –            | –                    |
| B$^+$ | 1.72         | –            | –                    |
| Al    | 2.86         | C$_{16}$SH-SAMs | √ [1]              |
| Ga    | 2.70         | –            | –                    |
| In    | 3.34         | –            | –                    |
| Tl    | 3.40         | –            | –                    |
| Cu$^*$| 2.56         | C$_{16}$SH-SAMs | √ [24]             |
| Ag    | 2.88         | C$_{16}$SH-SAMs | √ [9,10]           |
| Au    | 2.88         | C$_{16}$SH-SAMs | √ [3]              |
| Ti    | 2.94         | –            | –                    |
| Hg$^*$| 3.02         | C$_{16}$SH-SAMs | √ [5,25]           |

Δ: van der Waals diameter: Na: 4.62 Å; K: 4.62 Å; Rb: 4.88 Å; Cs: 5.24 Å; B: 4.16 Å [27]. van der Waals diameter values of other metal atoms are not available.
×: no experiment has been done;
√: atoms can penetrate into the C$_n$SH-SAMs;
*: C$_n$SH-SAMs on polycrystalline gold.

$d$Al $\geq$ 2 Å, Al$_1^+$, Au$_{x}$Al$_y$S$_z^-$ and AlS(CH$_2$)$_x^+$ peaks are also observed, indicating the formation of a highly uniform $\sim$ 1:1 Al adlayer on Au(111) [1]. If the metal atoms penetrate into the C$_n$SH-SAMs via common defect or transient defect, Al adlayer on Au(111) should be irregular distribution and ununiform shape due to the irregular distribution of these defects, rather than forming a highly uniform $\sim$ 1:1 Al adlayer on Au(111).

3. Conclusions

The physical meanings and scientific significances of the proposed new mechanism are clear. “Void”, which is often ignored in the past, actually plays an important role in many research field [20,29–31]. The intrinsic $\sim$ 3 Å channels existing in the C$_n$SH-SAMs are just these regular voids (free volume [9] or pore [32]) among the entities of C$_n$SH molecules in the close-packed long-chain C$_n$SH-SAMs on Au(111). The nature of the penetration behaviors of metal atoms on the long-chain C$_n$SH-SAMs through the intrinsic $\sim$ 3 Å channels is similar to the behavior of liquids or melts flowing through nanoscale channels [33], translocation of DNA [34] and proteins [35] through the solid-state nanopores, and the permeation of gas (e.g. CO$_2$, CH$_4$ and N$_2$) across the zeolite membrane [36]. The characters of the new penetration mechanism proposed by us and the previous two penetration mechanisms are summarized in Table 3, we can find that this new penetration mechanism is differ obviously from
the previous two “defects” mechanisms, which can be considered as the third intrinsic penetration mechanism [37]. The merits of the application of this simple ideal model are as follows:

(a) It can easily elucidate that the penetration process of metal atoms on $C_n$SH-SAMs on Au(111). It solves the problem in explaining the metal atoms penetration and avoids using the “defects” mechanism: the random and irregular distribution of the defects or pinholes are in different sizes with nanometer range [1,14], which can not select the metal atoms with different diameters for penetration or not.

(b) In the Å-scale and nm-scale microcosm, the classical physical principles are still valid by effective combination of structural chemistry and quantum chemistry, thus many complicated things can be treated much simpler.

(c) This model gives useful insights to the further study of the metal/SAMs systems (such as the study of the correlation of vapor process and penetration of metal atoms, the interaction mechanism between metal atoms and SAMs) as well as the artificial design of the SAMs–based devices (e.g. different metal atoms, metal substrates and function-group of adsorbate molecules). Furthermore, this model provides valuable references to the basic studies in the SAMs and the electrode’s surface and interface (such as defects, permeation, electron tunneling, conduct channel, nano–gap and the various influence factors for SAMs’ preparation, etc.) [38–48].

Table 3 The characters of the previous two penetration mechanisms and the new penetration mechanism proposed in this work.

| Penetration characters | (I) The first mechanism | (II) The second mechanism | (III) This work |
|-----------------------|------------------------|--------------------------|-----------------|
| (1) Penetration channel | Pinhole, gauche defect, domain boundary and other diffusion paths | Transient appearance of holes (defect) caused by the hopping of the adsorbate molecules on Au | Intrinsic ~ 3 Å channels |
| (2) Position of channel | Determinate | Random | Determinate |
| (3) Distribution of channel | Irregular | Irregular | Regular |
| (4) Size of channel | Nanometer-scale | Nanometer-scale | ~ 3 Å |
| (5) Application range | SAMs formed on the substrate with poor pretreatment or the SAMs with disorder structure | Short-chain $C_n$SH-SAMs ($n \leq 10$) | Long-chain $C_n$SH-SAMs ($n > 10$) with defect-free structure |
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