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# Self-Assembled Monolayer Compatible with Metal Surface Acoustic Wave Devices on Lithium Niobate

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The formation of a siloxane self-assembled monolayer (SAM) film on a lithium niobate substrate was investigated for surface acoustic wave (SAW) sensor devices for the detection of hydrogen. The most widely used SAM coupling reagent, octadecyltrichlorosilane, etches aluminum metal features that are integral to sensor devices, due to the formation of high local concentrations of hydrochloric acid. An alternative coupling reagent, octadecyltrimethoxysilane (OTMS), does not show any etching of metal parts. OTMS and related molecules are compatible with conventional SAW device manufacturing techniques and other devices that contain metal features susceptible to etching by acid released in the SAM formation process.

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## 1. Introduction

Self-assembly of molecular films from solution has been extensively employed to construct monolayer thin organic layers because a molecularly ordered structure can be prepared very easily without expensive and sophisticated equipment.[1] The most widely studied SAMs are alkanethiols on gold[2] and alkylsilanes on glass ($SiO_2$).[3] The diverse applications of molecular self-assembly range from simple adhesion control[4,5] and polymer grafting[6,7] to more complex integrated functionalities such as organic light emitting diodes,[8,9] molecular electronics,[10,11] and biosensors.[12]

SAM coatings have recently been used in hydrogen ($H_2$) gas sensor applications.[13] Palladium (Pd) is well-known to absorb $H_2$, and this property has been used for $H_2$ sensor applications.[14] Xu et al. suggested that the response of the Pd-based sensor to $H_2$ was enhanced when the glass substrate surface was modified by an octyldimethylchlorosilane SAM prior to the Pd deposition.[13]

In recent times, the growth of alkylsilane SAMs has been extended to various kinds of surfaces such as $TiO_2$,[15] chromium,[16] indium tin oxide,[17,18] indium nitride,[19] and tantalum pentoxide.[20] To our knowledge, siloxane SAMs have not been grown on lithium niobate ($LiNbO_3$).

Surface acoustic wave (SAW) devices have been utilized for a wide range of applications such as mobile communication systems[21,22] and chemical sensors in gas[23] and liquid.[24] An SAW device consists of microelectrodes, the so-called interdigital transducer, which transforms an applied ac bias to an acoustic wave on a piezoelectric substrate (and vice versa).[21] These devices can be manufactured using a variety of piezoelectric substrates, including quartz, $LiNbO_3$, or lithium tantalate ($LiTaO_3$). $LiNbO_3$ and $LiTaO_3$ have been widely used as piezoelectric substrates due to their high electromechanical coupling coefficients.[25] The electrodes are normally made from aluminum (Al) or an Al alloy.[23] SAW-based sensors consist of acoustic wave devices incorporating sensing materials, such as indium oxide layers deposited on $LiTaO_3$ with an SAW device made of Al electrodes[23] and a polysiloxane layer grafted on quartz with a gold electrode.[26,27]

It is tempting to combine the small, wireless features of the SAW device with self-assembly techniques to control wettability of the device to achieve controlled deposition of a metal layer or achieve direct grafting of biopolymer- and polymer-sensing materials or even the design of more complex nanostructures. Since most SAW devices are fabricated on oxide substrates, one strategy is to form siloxane SAMs on the surface. Conventional SAW device manufacturing processes, including deposition and photolithographic definition of metal-interdigitated electrode and bus bar structures, preferably precede the SAM coating process. Under these conditions, it is important to examine the effect of

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SAM formation on SAW device features and properties, e.g., the possibility of corrosion in the wet chemical process.

In the present study, the formation of a siloxane SAM on a $LiNbO_3$ substrate with aluminum SAW devices was investigated. The most widely used coupling reagent, octadecyltrichlorosilane (OTS), etches Al metal features due to the formation of high local concentrations of hydrochloric acid. On the other hand, an alternative coupling reagent, octadecyltrimethoxysilane (OTMS), does not show any etching of metal parts. This SAM has characteristics that make it compatible with conventional SAW device manufacturing techniques.

## 2. Experimental Section

All glassware was cleaned by piranha solution (3:1 (v/v) concentrated sulfuric acid/30% hydrogen peroxide) (*caution: piranha solution is extremely corrosive and can react violently with organic compounds*) and dried by a stream of hot air prior to use. ST-cut quartz (Sawyer) and Y-cut $LiNbO_3$ (Crystal Technology, Inc.) substrates were $O_2$ plasma treated (100 mW rf power, 400 mTorr of $O_2$, 5 min). $O_2$ plasma treatment does not degrade Al devices but maximizes the number of hydroxyl groups on the substrates as indicated by the wettability. The rms roughness of the $LiNbO_3$ wafer, evaluated for regions of ~2 $\mu$m × 2 $\mu$m, was measured by AFM cross section and found to be ~0.2 nm for the polished front surface and ~75 nm for the unpolished backside. The rms roughness of the quartz wafer was found to be ~0.3 and ~40 nm for the polished and unpolished surfaces, respectively. OTS (Gelest Inc.) and OTMS (Gelest Inc., >95%) were used without further purification. The OTS SAM was made by immersing a clean substrate into a 0.1% (2.5 mM) OTS solution in a mixture (4:1) of hexadecane (Sigma, >99%) and chloroform (Sigma-Aldrich, 99.8%) at room temperature (ca. 22 °C) in an ambient atmosphere (typical RH ≈ 40%) for 20 min.[28,29] The OTS sample was rinsed with chloroform and methanol. The OTMS SAM was made by immersing a clean substrate into a 1% (24 mM) OTMS solution in hexanes (Mallinckrodt, ACS grade) or anhydrous ethanol (Pharmco-AAPER, ACS/UPS grade) at room temperature in an ambient atmosphere for 4 h unless otherwise indicated. The OTMS sample was rinsed with hexanes and methanol (Pharmco-AAPER, ACS/UPS grade). Ultrasonic cleaning, which is often used to remove excess physisorbed polymeric layers,[30] was avoided since ultrasonic cleaning sometimes breaks $LiNbO_3$ substrates. After being dried with a stream of $N_2$, the SAM-coated sample was stored in a Petri dish. The quality of the SAMs on quartz and $LiNbO_3$ surfaces was characterized by FTIR and water contact angle measurement. FTIR spectra were measured using a Bruker Optics TENSOR 27 spectrometer equipped with a DTGS detector in transmission geometry at a normal incidence.[31] All the FTIR spectra were background corrected by subtraction of a spectrum of the clean, bare (i.e., SAM-free) substrate. All of the spectra were recorded by integrating 200 scans with a resolution of 4 $cm^{-1}$. Degradation of Al circuits on the $LiNbO_3$ substrate was visually examined by an optical microscope using various magnifications of up to 400×. Electrical testing was performed on wafer-level OTS- and OTMS-coated SAW device samples using an rf probe station connected to an HP-8753 network analyzer to measure the reflected response ($S_{11}$) over a 100 MHz frequency range centered at 250 MHz.

## 3. Results and Discussion

The quartz and $LiNbO_3$ substrates coated with an OTS SAM showed water contact angles around 105° at the polished surface and 120° at the unpolished backside surface, indicating that OTS SAMs were constructed on both quartz and $LiNbO_3$ substrates. FTIR spectra of the OTS SAM on quartz (dashed line) and $LiNbO_3$ (solid line) surfaces in the CH stretching region (Figure 1) show the two major peaks associated with the symmetric and asymmetric stretching modes of the methylene groups. For OTS on quartz these appear at 2850 and 2918 $cm^{-1}$, respectively. These frequencies indicate that the alkyl chains are in a crystalline-

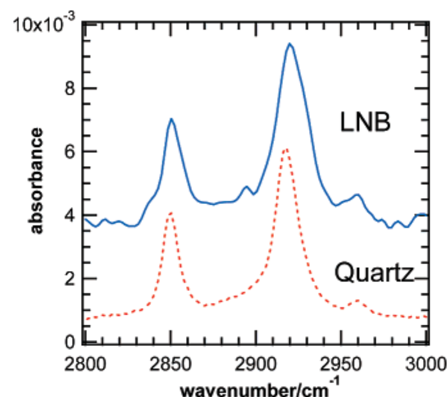


**Figure 1.** FTIR spectra of an OTS SAM on quartz (dashed line) and $LiNbO_3$ (solid line) in the CH stretching region. Distortion of the spectral shape in the dashed line was caused by an incomplete compensation of background subtraction.

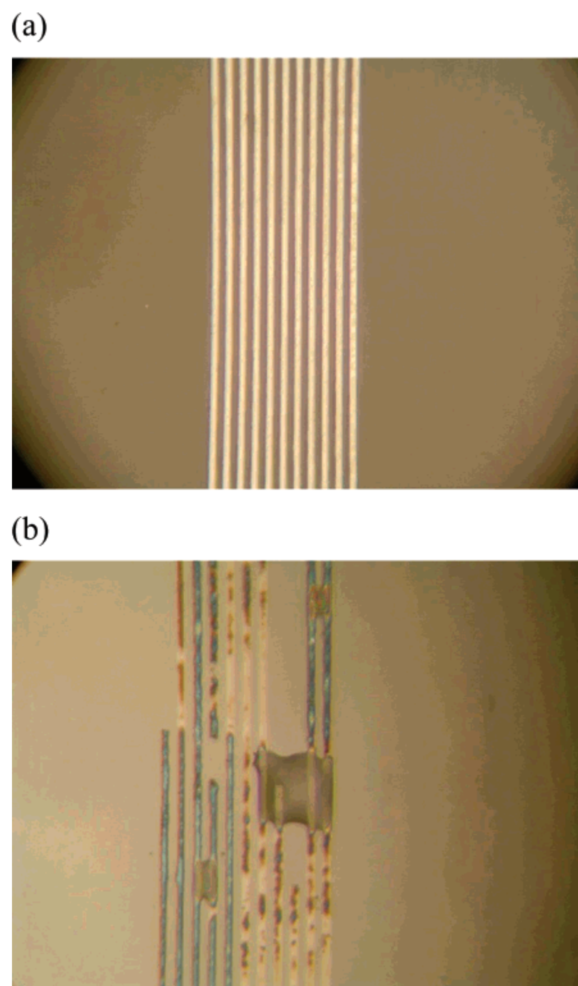


**Figure 2.** Typical optical microscope images of an Al device on a $LiNbO_3$ substrate (a) before and (b) after OTS treatment. The Al lines are nominally 3.5 $\mu$m wide.

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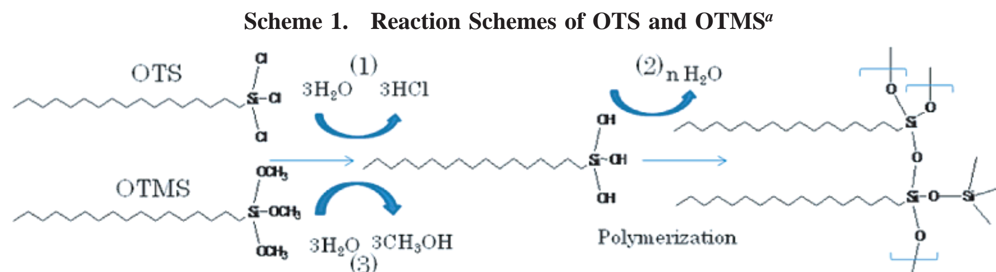
**Scheme 1. Reaction Schemes of OTS and OTMS[a]**




[a] Key: (1) SiCl of OTS is replaced by SiOH to release HCl. (2) SiOH groups react with each other and with surface silanol to form a siloxane linkage. (3) $SiOCH_3$ of OTMS is replaced by SiOH to release $CH_3OH$. Part 2 is essentially the same for both OTS and OTMS.

like structure rather than the liquid phase.[32] The peak absorbance of ~3 mOD suggests the formation of a monolayer of octadecyl groups on the substrate.[33] The larger absorbance for $LiNbO_3$ (~5 mOD) is probably associated with either some multilayer formation or the larger surface area due to the larger surface roughness of the unpolished backside. Interestingly, when a quartz surface was cleaned by piranha solution, the asymmetric stretching mode was observed at 2917 $cm^{-1}$ with a peak absorbance of ~3 mOD for OTS on quartz. The two major peaks were observed at 2850 and 2921 $cm^{-1}$ for OTS on $LiNbO_3$, which indicates a relatively disordered alkyl layer. Thus, we conclude that it is possible to form a hydrophobic OTS SAM on a $LiNbO_3$ surface pretreated with $O_2$ plasma but with a slightly disordered monolayer structure compared to an OTS SAM on a quartz surface.

Contrary to our expectation, no acoustic response was observed upon electrical testing of these SAW devices. To identify the impact of the OTS SAM formation procedure on performance, optical microscope images were acquired (a) before and (b) after OTS treatment as shown in Figure 2. These images clearly show that OTS treatment causes significant degradation of the Al metal features, which are critical for the device operation. This is probably because during SAM formation, the OTS releases HCl near the surface of the wafer when it hydrolyzes (see the reaction in Scheme 1a), and HCl etches the delicate Al device elements, which are approximately 150 nm thick and nominally 3.5 $\mu$m wide. OTS in solvent at first hydrolyzes with a trace amount of water adsorbed on the substrate, releasing three molecules of HCl per OTS molecule hydrolyzed near the surface (reaction 1, Scheme 1). Then the condensation reaction takes place, releasing $H_2O$ back to solution in the near-surface region (reaction 2, Scheme 1). The cycles of reactions 1 and 2 may result in a low effective surface pH. Clearly, OTS is not an appropriate reagent to form a SAM on the substrate with Al devices. A similar etching effect has been reported for chlorosilanes on stainless steel, suggesting that the chloride ion is the source of the corrosion.[34]

A silane coupling agent without chloride such as a trialkoxysilane has been suggested to be an appropriate alternative for coating of corrosion-susceptible materials.[34] As shown in Scheme 1b, OTMS releases only methanol during hydrolysis. In contrast to monolayer formation from chlorosilane precursors, methoxysilane SAMs have been studied less extensively. Some groups have reported OTMS SAM formation in nonpolar solvents such as toluene[35,36] and bicyclohexane,[16] while other groups used polar solvents such as 2-propanol solutions for OTMS[37] and ethanol for N-[3-(trimethoxysilyl)propyl]ethylenediamine.[38] To justify these protocols, we examined hexanes and ethanol as solvents for OTMS SAM formation. The SAM coating was carried out by immersing $LiNbO_3$ substrates into 24 mM OTMS (i.e., 1%) solution in hexanes (Figure 3a) and anhydrous ethanol (Figure 3b) for various times. The relatively high concentration of the OTMS solution was chosen to form a good monolayer without using an acid/base catalyst.[16] Many research groups have used acids[34] or bases[35] as catalysts to promote hydrolysis of meth-

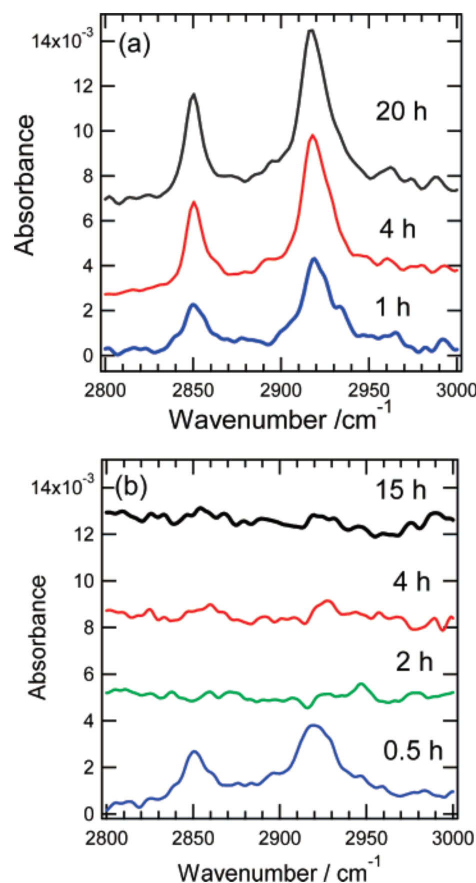



**Figure 3.** FTIR spectra of an OTMS SAM on $LiNbO_3$ in the CH stretching region. OTMS deposition was done in (a) hexanes and (b) ethanol solutions of 24 mM OTMS for various periods of time as indicated in the figure.

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**Table 1. Summary of FTIR, Contact Angle (CA) Measurements, and Rms Roughness of the Substrate: Peak Absorbance, Peak Frequency (freq), and Full Width at Half-Maximum (fwhm) of the $CH_2$ Asymmetric Mode[a]**

| sample | abs (mOD) | freq ($cm^{-1}$) | fwhm ($cm^{-1}$) | CA (deg) (polished) | CA (deg) (unpolished) |
|---|---|---|---|---|---|
| Q/OTS | 5.0 | 2917.9 | 15.3 | 105 | 115 |
| LNB/OTS | 5.0 | 2920.9 | 20.4 | 105 | 120 |
| Q/OTMS(4) | 3.0 | 2918.5 | 16.7 | 100 | 120 |
| $Q^p$/OTMS(4) | 2.4 | 2919.5 | 19.0 | 105 | 115 |
| LNB/OTMS (4) | 6.1 | 2918.7 | 19.0 | 105 | 120 |
| LNB/OTMS (4)′ | 6.4 | 2918.3 | 17.9 | 105 | 120 |
| Q/OTMS (20) | 3.0 | 2918.0 | 15.6 | 100 | 120 |
| $Q^p$/OTMS (20) | 3.4 | 2919.1 | 18.3 | 105 | 117 |
| LNB/OTMS(20) | 6.2 | 2918.5 | 18.3 | 105 | 120 |

| | rms roughness (nm) (polished) | rms roughness (nm) (unpolished) |
|---|---|---|
| Q | 0.3 | 40 |
| LNB | 0.17 | 75 |

[a] Key: Q, quartz; LNB, $LiNbO_3$; (4), 4 h of immersion; (20), 20 h of immersion; $Q^p$, quartz cleaned by piranha solution. The uncertainty of the CA measurements was ca. ±5°. The uncertainty of frequencies in FTIR measurements was $< \pm 0.2$ $cm^{-1}$. LNB/OTMS (4)′ is a duplicate experiment.

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oxysilane. However, it was also reported that good monolayers on chromium were obtained from 0.1 M OTMS in bicyclohexyl without catalyst in 48 h.[16] Good monolayers were not obtained from 1 mM OTMS solutions.[16]

FTIR spectra of an OTMS SAM on $LiNbO_3$ grown from hexane solutions (Figure 3) showed the two major peaks at 2850 and 2919 $cm^{-1}$ with absorbance of 6 mOD, indicating that OTMS in hexanes forms densely packed SAMs on $LiNbO_3$. The peak absorbance is larger than that of OTMS on a quartz substrate (3 mOD) presumably because of the higher roughness of the unpolished backside surface. The water contact angle is around 105°, which is slightly less than that expected for a perfect alkyl monolayer,[34] indicating that this OTMS SAM may include some defects. Comparing the 4 h immersion sample with the 20 h sample, 4 h seems long enough for almost complete OTMS monolayer formation. Since we have on occasion seen larger absorbance (ca. 10 mOD) for samples immersed more than 14 h (not shown), longer immersion appears to occasionally cause multilayer formation or deposition of bulk polymerized OTMS. Thus, we conclude that 4 h of immersion in hexanes solution is an effective protocol to form an OTMS SAM.

The 4 h immersion seems rather short for OTMS formation without a catalyst, compared with reported literature procedures.[16,34] The FTIR data (Table 1) suggest that 4 h is not enough time for the OTMS SAM to grow on piranha-cleaned quartz. Saturation occurs at a longer time (20 h). Thus, the faster growth kinetics in the present study is attributed to the cleaning process. The different cleaning process leads to different numbers of surface silanol groups and/or physisorbed water molecules that affect the growth kinetics.[39] The effect of the cleaning process was also seen in OTS coating of a quartz surface as mentioned above.

When the SAM coating was carried out in an ethanol solution of 24 mM OTMS, the band intensity of the FTIR spectrum was quite variable and irreproducible. Furthermore, the time evolution of the OTMS immersion was nonmonotonic and never showed saturation. Typically, an absorbance of a few mOD was observed for a relatively short immersion time (30 min), but with continued immersion the absorbance decreased to almost zero (2 h) and then increased to 0.5 mOD (4 h). Further immersion in ethanol solution up to 15 h did not improve the SAM formation. Since the same trend was observed for OTMS on quartz surfaces, ethanol was considered to be the cause of the poor SAM formation. This is probably because ethanol hydrogen bonds with surface silanol more strongly than hexanes, preventing OTMS molecules from reacting with the surface silanol. Although ethanol has been used as a solvent for reactions of aminomethoxysilane due to its good solubility,[38] the present results indicate that using a non-hydrogen-bonding solvent seems to improve the coverage and reproducibility of the methoxysilane SAM.

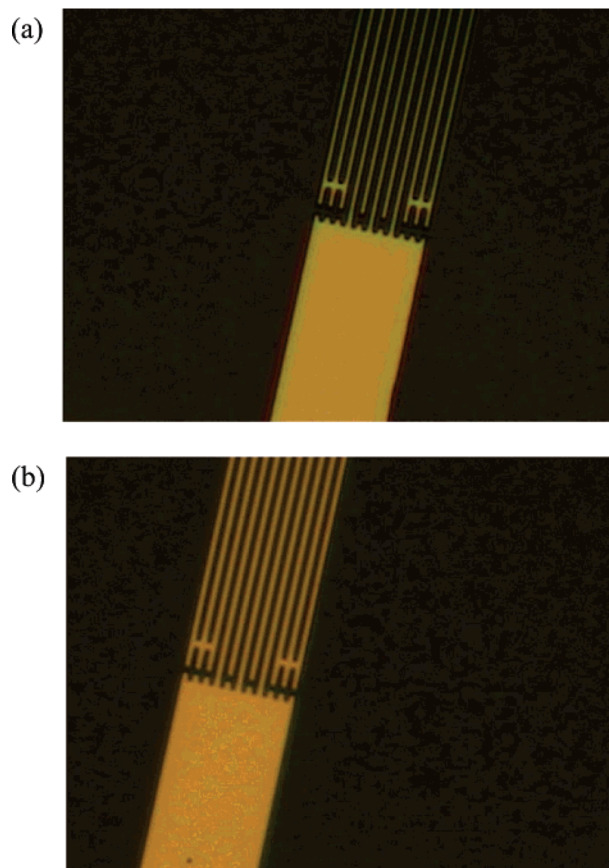


**Figure 4.** Typical optical microscope images of an Al device on a $LiNbO_3$ substrate (a) before and (b) after OTMS treatment. The Al lines are nominally 3.5 $\mu$m wide.

As we expected, there was no etching effect of OTMS on the Al device on the $LiNbO_3$ substrate (Figure 4), demonstrating the compatibility of OTMS formation with delicate metallic devices. The fact that OTMS did not show any etching effect supports our hypothesis that the release of HCl is the cause of Al device degradation upon OTS treatment. The expected acoustic response was also observed upon electrical testing of these devices, indicating that the OTMS SAM layer also did not interfere with the acoustic wave generation and propagation.

## 4. Conclusion

This work demonstrated that OTS forms densely packed SAMs on both $LiNbO_3$ and quartz. The formation process of OTS SAMs degrades Al devices significantly, while that of OTMS SAMs does not. OTMS SAMs were constructed on $LiNbO_3$ substrates with good reproducibility from hexane solutions, while OTMS did not form reproducible SAMs on $LiNbO_3$ or quartz from ethanol solutions. OTMS SAM formation is potentially useful as one step in a procedure that can be used to manufacture SAW-

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based hydrogen sensors or the coating of any device whose metal parts are susceptible to corrosion. These findings suggest that although alkyltrichlorosilane SAMs have been widely used and investigated, alkyltrimethoxysilanes may be of more practical importance when self-assembly is required on surfaces with delicate metal components.

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