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Al-neal Degrades Al₂O₃ Passivation of Silicon Surface

Olli E. Setälä,* Toni P. Pasanen, Jennifer Ott, Ville Vähänissi, and Hele Savin

Atomic layer deposited (ALD) aluminum oxide (Al₂O₃) has emerged as a useful material for silicon devices due to its capability for effective surface passivation and ability to generate p⁺ region underneath the oxide as active or passive component in semiconductor devices. However, it is uncertain how Al₂O₃ films tolerate the so-called Al-neal treatment that is a necessary process step in devices that also contain silicon dioxide (SiO₂) passivation layers. Herein, it is reported that the Al-neal process is harmful for the passivation performance of Al₂O₃ causing over eightfold increase in surface recombination velocity (SRV) (from 0.9 to 7.3 cm s⁻¹). Interestingly, it is also observed that the stage at which the so-called activation of Al₂O₃ passivation is performed impacts the final degradation strength. The best result is obtained when the activation step is done at the end of the process together with the Al-neal thermal treatment, which results in SRV of 1.7 cm s⁻¹. The results correlate well with the measured interface defect density, indicating that the Al-neal affects defects at the Si/SiO_x/Al₂O₃ interface. The root causes for the defect reactions are discussed and possible reasons for the observed phenomena are suggested.

1. Introduction

Surface defects are well known to limit the performance of silicon devices. These defects cause increased recombination and leakage current, and therefore, their avoidance is an important factor in device processing.^[1] The detrimental effect of surface defects has traditionally been reduced by passivating them with thermally grown silicon dioxide (SiO₂). The passivating effect of SiO₂ relies mostly on elimination of interface defects,^[2] which is typically achieved optimally by annealing the film with a thin aluminum layer on top in a process called the Al-neal.^[3,4] The improved passivation performance obtained by Al-neal is based

on a chemical reaction between the Al and hydroxyl ions (OH⁻) in the oxide, which generates atomic hydrogen that neutralizes dangling bonds at the Si/SiO₂ interface.^[5]

In addition to thermal SiO₂, the recent development of materials and processes has led to increasing utilization of other surface passivation thin films, including aluminum oxide (Al₂O₃) grown with atomic layer deposition (ALD).^[6–8] Unlike with thermal SiO₂, no Al layer is needed to activate the surface passivation of Al₂O₃; instead, a pure anneal in nitrogen or forming gas (FG) ambient at around 400 °C is typically conducted to improve the passivation.^[9] It is well known that during the ALD a thin 1–2 nm SiO_x layer is formed between Si and Al₂O₃ and in the subsequent activation anneal hydrogen that is present in the Al₂O₃ film (≈1–3 at%) diffuses to Si/SiO_x interface and passivates the dangling bonds, thus improving the


chemical passivation.^[6,10–12] The Al₂O₃ activation anneal often improves also the field-effect passivation by increasing the magnitude of the negative charge (≈10¹² cm⁻²) formed at the SiO_x/Al₂O₃ interface,^[13,14] the effect being most prominent for water-based thermal ALD.^[7,15]

While Al₂O₃ thin films are today commonly used in silicon devices, thermal SiO₂ films are still often needed alongside them to provide, e.g., high-quality neutral (or small positive) charge insulator in the same device, a typical example being an induced junction photodiode.^[16,17] Consequently, when both Al₂O₃ and SiO₂ are present on the same wafer, it can be difficult to perform optimized passivation anneal for both films. One good example is the Al-neal of SiO₂ that is usually performed as one of the last steps in device fabrication to allow performing it alongside contact metallization. While it can be difficult to perform deposition and patterning of the Al₂O₃ film after metallization and Al-neal due to, e.g., etch selectivity between Al and Al₂O₃, it needs to be studied how such Al-neal would affect the passivation performance of Al₂O₃.

In this work, we study the aforementioned issue, i.e., the impact of the Al-neal process on Al₂O₃ passivation in high-resistivity silicon wafers that are typical for detector fabrication. First, we investigate how the Al-neal affects the performance of ALD Al₂O₃ passivation film using standard minority carrier lifetime measurements. Interface defect density and total charge of the film are then measured to gain understanding about the interface and oxide properties that are affected by the Al-neal. Next, we study how the stage at which the activation anneal of Al₂O₃ is performed affects the passivation and oxide interface properties. Finally, the results and underlying causes for the observed phenomena are discussed.

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2. Experimental Section

To investigate the impact of the Al-neal on the passivation performance of Al₂O₃, the quality of surface passivation in various processing scenarios, which are shown in **Figure 1**, is assessed. The wafers used in these experiments were 300 μm-thick high resistivity (≈5 kΩcm) n-type single-side polished Float Zone (FZ) silicon wafers with (111) crystal orientation and 100 mm diameter. Before the actual processing, the lifetime-limiting bulk defects in the FZ wafers were deactivated by a typical anneal at 1050 °C in an oxygen atmosphere for 30 min,^[18,19] followed by a subsequent oxide removal. Two wafers (dashed and solid blue arrows in **Figure 1**) were RCA-cleaned followed by the double-sided deposition of a 50 nm-thick Al₂O₃ film with ALD in 200 °C with trimethylaluminum (TMA) and water as the precursors. Surface passivation was activated by annealing the wafers in FG at 425 °C for 30 min (“Al₂O₃ activation” in **Figure 1**). On one of the wafers, the Al-neal was performed. As shown in **Figure 1**, it consisted of the following three steps: 1) sputtering of a 300 nm-thick Al-layer on both sides of the wafer, 2) annealing in FG at 425 °C for 30 min, and 3) removal of the Al layer with a commercial phosphoric acid-based Al etchant.

To investigate how the stage of the process at which Al₂O₃ passivation is activated affects the passivation performance, another Al₂O₃ sample was prepared, where a separate Al₂O₃ activation anneal prior to Al deposition was omitted (dotted blue line in **Figure 1**). Otherwise the fabrication process followed the steps of the earlier Al-nealed Al₂O₃ wafer.

The level of surface passivation that can be obtained by SiO₂ with and without the Al-neal process (solid and dashed red arrows, respectively, in **Figure 1**) was evaluated for reference.

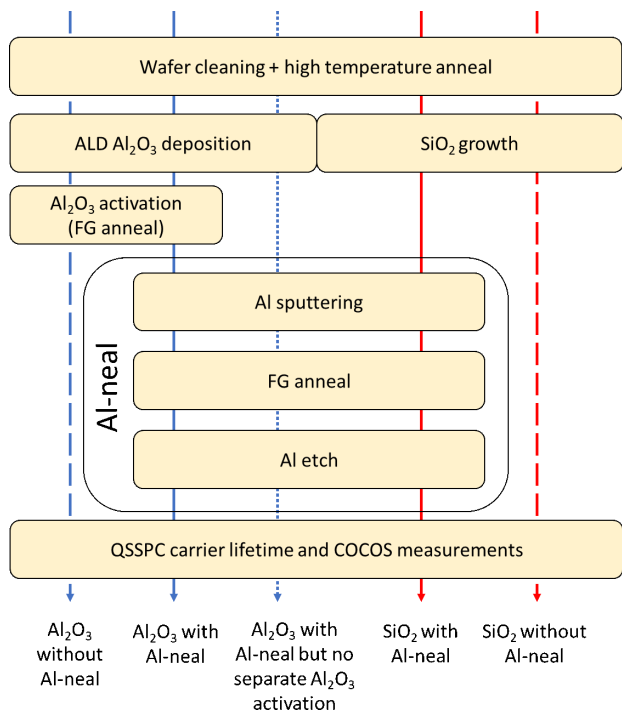


Figure 1. Process flows for each fabricated test sample containing the most important processing steps.

After standard cleanings, two SiO₂ samples were oxidized in O₂ atmosphere at 1000 °C for 90 min, which resulted in an 85 nm-thick oxide. The Al-neal was then performed for the other SiO₂ sample the way described before.

The passivation performance in all samples was evaluated by the measurement of minority charge carrier recombination lifetime as a function of injection level using quasi-steady-state photoconductance (QSSPC) method with Sinton Instruments WCT-120TS tool. The maximum surface recombination velocity (SRV_{max}, hereinafter referred to simply as SRV) was calculated from the carrier lifetimes assuming infinite bulk lifetime with^[20]

$$SRV_{\max} = \frac{W}{2 \cdot \tau_{\text{eff}}} \quad (1)$$

where W is the thickness of the wafer and τ_{eff} is the measured effective carrier lifetime. Finally, thin film properties, such as the total charge (Q_{tot}) and interface defect density (D_{it}), were assessed with a method called corona oxide characterization of semiconductor (COCOS),^[21] using Semilab SDI PV-2000 Lifetime Scanner. Linear dependence between deposited corona charge and the measured contact potential difference indicates that no charge leakage occurred during any of the measurements, ensuring reliable determination of Q_{tot} and D_{it} .

3. Results

Figure 2 shows the impact of the Al-neal process on the carrier lifetimes of Al₂O₃, and as a reference, SiO₂ passivated silicon samples. It is evident that the Al-neal process has a large effect on the lifetimes achieved with both films and due to identical substrates, the differences in lifetimes reflect changes in surface passivation. As expected, passivation performance of SiO₂ increases vastly from microsecond range to milliseconds. Interestingly, a totally opposite phenomenon is observed in the case of Al₂O₃ passivation. The Al-neal causes the lifetime of the Al₂O₃-passivated wafer to decrease from 16.45 to 2.05 ms

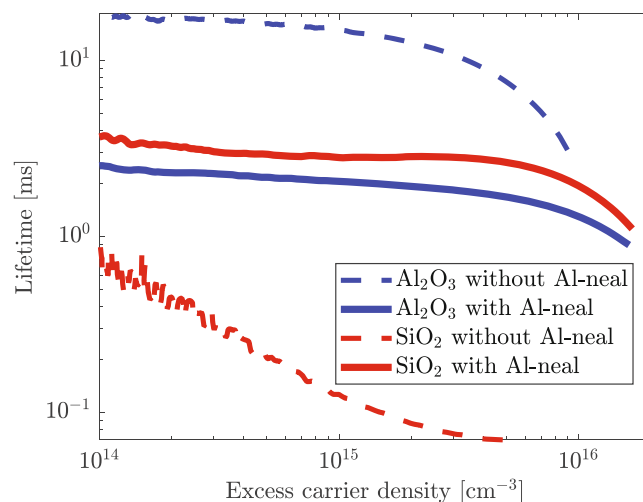


Figure 2. The effect of the Al-neal process on the minority charge carrier lifetimes on Al₂O₃ and SiO₂ passivated Si. Contrary to SiO₂, passivation performance of Al₂O₃ is impaired by the Al-neal.

Table 1. The impact of the Al-neal process on the interface defect density (D_{it}) and charge density in the thin film (Q_{tot}) on Al_2O_3 - and SiO_2 -coated n-type 5 k Ω cm Si with 300 μ m thickness. Lifetime and SRV at 10^{15} cm^{-3} injection level are presented for reference.

Film material	Treatment	Midgap D_{it} [$eV^{-1} cm^{-2}$]	Q_{tot} [cm^{-2}]	Lifetime [ms]	SRV [$cm s^{-1}$]
Al_2O_3	Without Al-neal	4.1×10^{11}	-1.3×10^{12}	16.45	0.9
Al_2O_3	With Al-neal	2.3×10^{12}	-3.2×10^{12}	2.05	7.3
SiO_2	Without Al-neal	5.7×10^{11}	3.5×10^{11}	0.13	115.4
SiO_2	With Al-neal	1.2×10^{11}	1.5×10^{11}	2.83	5.3

(or SRV to increase from 0.9 to 7.3 $cm s^{-1}$) at 10^{15} cm^{-3} carrier density, deteriorating the passivation performance below that of SiO_2 .

To understand the mechanism of passivation degradation during the Al-neal process, we need to study the behavior of D_{it} and Q_{tot} , which are the two major components constituting the passivation performance of a film.^[22] Table 1 shows these values measured from Al_2O_3 - and SiO_2 -passivated samples with and without the Al-neal. Similar to the lifetime results, the behavior of Al_2O_3 film properties during the Al-neal is completely opposite to SiO_2 . A substantial increase in D_{it} (from $\approx 10^{11}$ to $\approx 10^{12}$ $eV^{-1} cm^{-2}$) is observed in the Al_2O_3 samples after the Al-neal process, which indicates that the Si/ SiO_x / Al_2O_3 interface seems to degrade due to the deterioration of chemical passivation. COCOS results reveal also that there is a slight increase in the film charge due to the Al-neal; however, the higher field-effect passivation cannot compensate the declined chemical passivation. The observed lifetime degradation with Al-nealed Al_2O_3 can be thus explained by increased recombination at the Si/ SiO_x / Al_2O_3 interface. In SiO_2 passivated reference samples, on the contrary, D_{it} behavior is opposite and the Al-neal reduces it considerably. Consequently, the observations imply that the Al-neal process affects the chemistry of the Si/film interfaces differently in SiO_2 than in Al_2O_3 films.

In the aforementioned samples, surface passivation was activated by annealing the Al_2O_3 film directly after the deposition of the thin film. As the sample experiences similar heat treatment also during the Al-neal process, it raises a question if the negative effect of the Al-neal could be reduced by changing the stage of the process when the Al_2O_3 passivation is activated. To study this, another Al_2O_3 -passivated sample was fabricated without a separate Al_2O_3 activation step (blue dotted line in Figure 1). Figure 3 compares the carrier lifetimes of such sample (dotted line) with the ones presented earlier (solid and dashed lines). At 10^{15} cm^{-3} carrier density, lifetime of 8.66 ms (SRV 1.7 $cm s^{-1}$) is achieved after the Al-neal process, which is a significant improvement from 2.05 ms (SRV 7.3 $cm s^{-1}$) that was measured from the sample that experienced a separate Al_2O_3 activation before the Al-neal process. A slight reduction in D_{it} (1.8×10^{12} vs 2.3×10^{12} $eV^{-1} cm^{-2}$) and increase in charge density (-3.9×10^{12} vs -3.2×10^{12} cm^{-2}) is achieved by removing the separate activation step. Although both values change to favorable direction for improved surface passivation, the magnitude of change is surprisingly small to completely

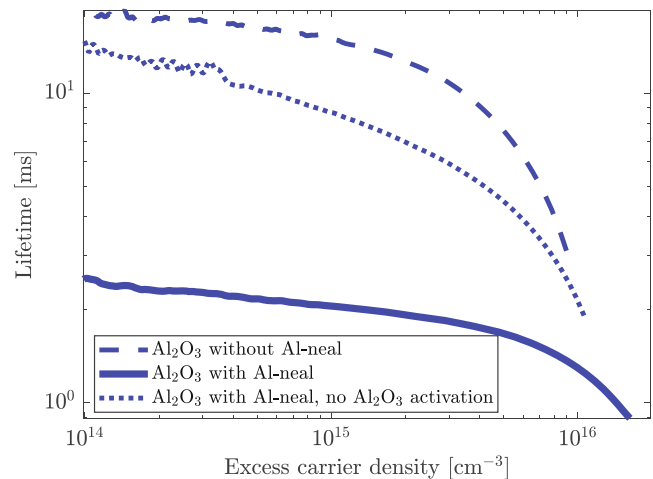


Figure 3. Carrier lifetime of Al_2O_3 sample with no separate Al_2O_3 activation compared with samples with separate activation anneal performed directly after thin film deposition. The passivation performance with the Al-neal is significantly better than earlier but not quite as good as achieved without the Al-neal.

explain the clear improvement seen in passivation performance. Interestingly, the lifetimes do not reach the initial values measured without the Al-neal process. This indicates that indeed something harmful happens during the Al-neal process that cannot be recovered during the Al_2O_3 activation anneal integrated in the Al-neal step.

4. Discussion

The Al-neal process was observed earlier to be harmful for the passivation performance of an ALD Al_2O_3 film. The result raises questions about the physical and chemical mechanisms taking place during the Al-neal of Al_2O_3 . In case of thermal SiO_2 , a chemical reaction takes place between the oxide and aluminum, in which atomic hydrogen is released.^[5] As hydrogen is known to be effective at passivating defects both at silicon surface and in the bulk,^[23,24] this reaction explains the increase in the passivation performance of SiO_2 . Indeed, Table 1 shows that Al-nealing of SiO_2 sample decreased D_{it} by a factor of four.

The effect of the Al-neal seems to, however, be completely opposite on Al_2O_3 . The root cause for the difference between the behavior of Al_2O_3 and SiO_2 films during the Al-neal cannot be concluded from the aforementioned experiments, but we will discuss here some hypotheses along with further experimental results. The obvious starting point is the D_{it} and Q_{tot} values shown in Table 1. The immense increase in the D_{it} during the Al-neal of Al_2O_3 indicates that the deteriorated passivation performance is caused by weakened chemical passivation resulting from additional defect states that are formed to the Si/ SiO_x / Al_2O_3 interface. It is known from the literature that chemical passivation can be destroyed by sputtering of the Al film, as it can cause damage to the silicon lattice and substrate/film interface.^[25] Indeed, this was confirmed to be the case here as well because the measured lifetime dropped to 30 μ s directly after sputtering. Such large changes in interface quality may be

unrecoverable by anneal afterward. The D_{it} of the Al_2O_3 samples could be thus related to the damage inflicted to the interface by sputtering, as the density of interface defects is five times larger in both cases where the Al-neal (and consequently sputtering) has been performed compared with the situation after only Al_2O_3 activation ($\approx 2 \times 10^{12}$ vs $4.1 \times 10^{11} \text{ eV}^{-1} \text{ cm}^{-2}$). Conversely, the Al-neal seems to increase the negative charge in Al_2O_3 (and SiO_2 but in its case the change is far less prominent and seen as decreased positive charge), which should be advantageous regarding field-effect passivation. Indeed, the generated interface defect states have been reported to trap charges and therefore can affect the amount of field-effect passivation.^[26,27] In our case, this effect was still rather modest and the increased D_{it} dominated in the measured lifetime.

To assess the damage resulting from the mere sputtering step speculated earlier, additional experiments were conducted. The processing sequence for these extra samples was the following: ALD Al_2O_3 deposition, optional Al_2O_3 activation anneal, Al sputtering, Al etch, FG anneal, and characterization. In other words, in these wafers, both with and without a separate Al_2O_3 activation anneal preceding the sputtering, the Al layer was immediately etched away after sputtering and an FG anneal was then performed without Al on the wafers. The results, also shown in Table 2 (rows 1 and 2), were as follows. If the Al_2O_3 activation anneal was done preceding the sputtering, the lifetime and SRV were 7.1 ms and 2.5 cm s^{-1} , respectively. If there was no Al_2O_3 activation anneal done preceding the sputtering, the lifetime and SRV were 10.59 ms and 1.7 cm s^{-1} , respectively. As shown earlier in Table 1 (and also in Table 2 as row 3), the corresponding values for Al_2O_3 without Al-neal were 16.45 ms and 0.9 cm s^{-1} , respectively. Thus, as hypothesized earlier, the SRVs of the sputtered wafers without Al-neal (2.5 and 1.7 cm s^{-1}) differ from the wafer that experienced only Al_2O_3 deposition and postdeposition activation (0.9 cm s^{-1}), indicating that sputtering does indeed cause some unrecoverable damage.

To see the effect of the presence of Al layer during the FG anneal, one needs to compare the aforementioned extra results (Table 2, rows 1 and 2) to their counterparts (Table 2, rows 4 and 5). The D_{it} and the lifetime are much worse if the Al layer is present during the FG anneal, indicating that the sputtering step cannot be solely responsible for degraded passivation. Furthermore, there is a clear disparity between the passivation performances of Al-nealed Al_2O_3 films with and without a separate Al_2O_3 activation step (Table 2, rows 4 and 5). In both of these samples, the Al_2O_3 film experiences the same sputtering

process, but the sample without separate Al_2O_3 activation recovers from the sputtering-induced damage considerably better. Interestingly, the same effect is seen in the results obtained from the additional experiments as there is similar disparity (Table 2, rows 1 and 2). Consequently, more efficient passivation is again achieved without a separate activation step.

The aforementioned results confirm that in addition to sputtering damage there are other harmful mechanisms taking place during the Al-neal process. One possibility could be the depletion of hydrogen in the film. During standard activation of Al_2O_3 passivation, hydrogen is created in atomic form in Al_2O_3 bulk in a mechanism, where OH-groups and Al atoms in the film form Al—O bonds and release hydrogen.^[28] This hydrogen together with hydrogen already present in the as-deposited film diffuses toward the Si surface and is responsible for the significantly reduced D_{it} .^[29] Such process should occur in the film regardless of the presence of the Al capping layer, as the thermal treatment during the Al-neal can be considered only as an additional Al_2O_3 activation. Due to the existence of OH-groups, the presence of Al layer could actually even increase the amount of atomic hydrogen in a reaction that is similar to SiO_2 Al-neal passivation. However, when combining the activation steps with the damage caused by sputtering, the situation may become different. In such circumstances, the amount of hydrogen in the film may not be enough for effective passivation. Indeed, notable release of hydrogen has been reported in ALD Al_2O_3 films deposited in $200 \text{ }^\circ\text{C}$; 15% of the total hydrogen content was released during 10 min thermal treatment in $400 \text{ }^\circ\text{C}$.^[29] Considering the similar Al_2O_3 activation temperature ($425 \text{ }^\circ\text{C}$) and longer duration (30 min) in our experiments, even larger depletion of hydrogen can be expected. Therefore, the hydrogen content in the once activated Al_2O_3 film might be too low to allow complete repassivation of the Si/ SiO_x / Al_2O_3 interface after it has been damaged by sputtering. This could explain why separate Al_2O_3 activation has such detrimental impact on the passivation performance of Al-nealed Al_2O_3 .

Based on the observations made in this study, solutions for the declined passivation performance can be suggested. First, the most obvious solution is to avoid the Al-neal of Al_2O_3 altogether, although this might not be feasible in every application. Second, the deposition of additional protective layers on Al_2O_3 during Al-neal could be considered but this may add complexity to the process. Third, alternative possibly less detrimental metal deposition methods, such as evaporation, may also be tried. Performing the Al-neal with gentler deposition methods could even raise the passivation performance above the level achieved

Table 2. Collection of the experimental results obtained for Al_2O_3 deposited samples. Thickness of the wafers annealed without Al on top is higher (350 vs $300 \mu\text{m}$) explaining the same SRV with different lifetimes.

Al_2O_3 activation prior sputtering	Anneal after sputtering	Midgap D_{it} [$\text{eV}^{-1} \text{ cm}^{-2}$]	Q_{tot} [cm^{-2}]	Lifetime at 10^{15} cm^{-3} [ms]	SRV [cm s^{-1}]
Yes	FG anneal without Al	7.8×10^{11}	-2.1×10^{12}	7.10	2.5
No	FG anneal without Al	3.2×10^{11}	-1.7×10^{12}	10.59	1.7
Yes	No sputtering	4.1×10^{11}	-1.3×10^{12}	16.45	0.9
Yes	Al-neal	2.3×10^{12}	-3.2×10^{12}	2.05	7.3
No	Al-neal	1.8×10^{12}	-3.9×10^{12}	8.66	1.7

without the Al-neal, due to its positive impact on the Q_{tot} . In cases where sputtering of Al on Al_2O_3 cannot be avoided the best result is achieved by etching the metal off from Al_2O_3 before annealing or alternatively by performing the Al-neal without separate Al_2O_3 activation step.

5. Conclusions

We have investigated the effect of the Al-neal process on the ALD Al_2O_3 passivation. We observed that the Al-neal can be harmful for the passivation performance of Al_2O_3 causing over eightfold increase in SRV (from 0.9 to 7.3 cm s^{-1}). Omitting a separate Al_2O_3 activation anneal between deposition of Al_2O_3 and the Al-neal was presented as a partial solution to the problem. Such fabrication scheme resulted in SRV of 1.7 cm s^{-1} , which is already an adequate value for most applications. Possible reasons for the compromised passivation were discussed, with the D_{it} results pointing toward sputtering damage (D_{it} increases from 4.1×10^{11} to $\approx 2 \times 10^{12} \text{eV}^{-1} \text{cm}^{-2}$) and problems with Al_2O_3 activation during the Al-neal. To achieve the optimal passivation performance with Al_2O_3 , additional modifications to the device fabrication process are likely needed. The simplest way is to avoid the Al-neal step altogether, but another possible solution could be to use alternative metal deposition methods, such as evaporation, to prevent excessive damage at the Si/ SiO_x / Al_2O_3 interface.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords

Al-neal, aluminum oxide, silicon devices, surface passivation

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