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Titanium dioxide by spray deposition for buried contact silicon solar cells fabrication

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전극함몰형 실리콘 태양전지의 제작시 스프레이 방법에 의한 타이타늄 옥사이드층의 적용에 관한 연구

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Abstract Titanium dioxide (TiO_2) film has been widely used as anti-reflection coating for solar cells but not as masking oxide for metallisation and diffusion of impurities. In this paper we have investigated the properties of TiO_2 for possible incorporation into solar cell processing sequence. Thus the use of a spray deposition system to form the TiO_2 film and the characterisation of this film to ascertain its suitability to solar cell processing. The spray-on TiO_2 film was found to be resistant to all the chemicals used in conjunction with solar cell processing. The high temperature anealing (in oxygen ambient) of the spray-on TiO_2 film resulted in an increased refractive index, which indicated the growth of an underlying thin film of SiO_2 film for the passivation of silicon surface which would reduce the recombination activities of the fabricated device. Most importantly, the successful incorporation of the TiO_2 film will lead to the reduction of the many high temperature processing steps of solar cell to only one.

요 약 타이타늄 옥사이드층을 태양전지의 표면 보호막으로 사용시, 그 적합성에 대해 조사하였다. 스프레이법으로 형성된 타이타늄 옥사이드의 박막층은 태양전지 제조 과정중 사용되는 화학약품에 잘 견디며, 이 층을 사용함으로서 고온산화공정을 줄일 수 있다.

1. Introduction

In the production of solar cells using the buried contact technology (both single sided and double sided buried contact cells), an anti-reflection coating other than the silicon dioxide has not been used. By using an anti-reflection layer of appropriate refractive index an extra 1 % absolute module efficiency is achievable. Titanium dioxide (TiO_2) film is often used as anti-reflection coating because of its high refractive index. Extensive work on TiO_2 film as an anti-reflection layer has been done [5]. However other use of TiO_2 film such as masking layer for both diffusion and metallisation steps have never been studied.

The use of TiO_2 film as a diffusion mask for impurities in conjunction with the spin-on sources would help reduce the many high temperature processing steps to only one. That is, the emitter and the rear junction would be formed, titanium dioxide film sprayed at 200-300℃, front and rear grooves scribed (for the case of double sided cell) and doped with different dopants through spin-on sources and dirven-in at one high temperature. This could be very useful for commerical solar cells that require low cost, mass production and high efficiency.

In order to make such an approach realistic, the feasibility of several steps needs to be demonstrated. These include, spin-on for emitter diffusion, simultaneous groove diffusions using spin-on sources [42]; suitability of TiO_2 as anti-reflection coating, plating and diffusion masks. In this paper, the suitability of the spray-on TiO_2 film using a simple system, figure 1, is reported. The results are preliminary and solar cells incorporating this films are yet to be fabricated.

2. Overview of Titanium dioxide thin film work

The first experimental observation of the anti-reflection coating effect in 1887 was made by Lord Rayleigh, while Bauer offered the first successful theoretical treatment based on interference effects in 1934 [2,3]. Since then the theory and application of optical interference phenomena has been well documented, especially with respect to thin film filters, and has resulted in a multi-million dollar optical coating industry. Anti-reflection coatings for solar cells, however, are relatively recent event beginning primarily in the 1960's with the advent of photovoltaics as remote power sources for spacecrafts and satelites [3].

As a rule of thumb, materials needed for anti-reflection control must be of high refractive index to be able to reduce the reflectance and increase transmittance to a reasonable value. The materials most frequently used for preparing high index films are zinc sulphide (ZnS) and antimony sulphide (Sb_2S_3). These sulphide films, however, even if applied under heat and put through a rather complicated baking process do not have as good mechanical and chemical stability as films of various metal oxides [5]. Titanium dioxide seems to be most satisfactory with mechanical and chemical durability as well as possessing high index of refraction.

Titanium dioxide exist at room temperature in three forms-rutile, anatase, and brookite, each of which occurs naturally. Each contains six coordinate titanium but rutile is most commom form, both in nature and as produced commercially, and others transform into it on heating [1]. All forms have a rather high index of refraction in the visible and the useful infra-red region, and are chemically and mechanically very resistive [5]. The rutile structure is based on a slightly distorted hcp (hexagonal closed packing) of oxygen atoms with half the octahedral interstices being occupied by titanium atoms [1]. Anatase and brookite are both based on cubic rather than hexagonal close packing of oxygen atom, but again the titanium atoms occupy half the octahedral interstices. Titanium dioxide, if evaporated in high vacuum, reduces slightly at the high temperature required for its evaporation and condenses as an absorbing film.

Various deposition methods have been used for the fabrication of TiO_2 coatings of desirable optical, mechanical and chemical properties. Such methods include : chemical vapor deposition [8, 9, 26-32], oxidation of the metal in air [5, 10-13, 33], vacuum evaporation [14], anodic oxidation [15-20], sputtering [21-25, 38, 39], spray deposition [3, 34, 35] and this work. The properties of the deposited film depends on the method of deposition and the temperature at which the material is deposited.

3. Properties of TiO_2

The properties of titanium dioxide has been studied by many workers and a comprehensive literature search was done by Grant [5] and Samsonov [36] and Clark [40]. The properties that are vital to solar cell are the optical (which varies according to the structure of the film), electronic and the chemical properties. A review of literature on the properties according to the different method of deposition and temperature is given in table 1.

4. Experimental

The experimental set up for spray deposition is simple and consists of a hot plate, the spray gun, nitrogen gas and the tetra-iso-propyl titanate (TPT)-$Ti(C_3H_6OH)_4$, as the TiO_2 source. TPT makes an excellent precursor for deposition of the film. It can be high-

Table 1
Variation of refractive index with temperature of deposition and author at 0.6 micron wavelength

Author	Method	Structure	Refractive index	Density (g/cm^3)
Haas [5]	Thermal oxidation of Evaporated Ti film at 300℃ - 700℃	Rutile(Electron diffraction)	2.7	—
Feursanger [8]	Hydrolysis of $TiCl_4$ above 160℃	—	2.55	—
Yokozawa et al [9]	Thermal decomposition of $Ti(C_3H_7O)_4$ in N_2 at 410 - 750℃	Anatase(X - ray diffraction)	2.08 - 2.13	2.5 - 2.6
		Amorphous(X - ray diffraction)	2.00 - 2.05	2.4 - 2.49
	Thermal decomposition of $Ti(C_3H_7O)_4$ in N_2+O_2 at 320℃ - 540℃	Anatase(Electron diffraction)		
Szlufeik et al [7]	Screen printing di - isopropoxides titanium and firing at 500℃ - 900℃	Rutile(X - ray diffraction)	2.76	4.26
		Anatase(X - ray diffraction)	2.56	3.84
Rausch & Burte [30]	Vapor deposition of tetra - iso - propyl titanate at 350℃ and annealed to 900℃	Anatase (Electron diffraction)	2.2 - 2.6	4.1
Toshiro & Arai [29]	Vapor deposition of $Ti(C_5H_7O_2)_4$ and $Ti(C_3H_7O)_2\ (C_5H_7O_2)_2$ at 300℃ - 600℃	Anatase(X - ray diffraction)	1.71 - 1.91	—
Fitzgibbons et al [27]	Vapor deposition of Tetra - iso - propyl titanate at 150℃ and annealed at 350℃ - 1000℃ in air	Amorphous Anatase	2.0	—
		Rutile	2.4	3.9
			2.6	4.24

Author	Method	Structure	Refractive index	Density (g/cm^3)
Sladek & Herron [28]	Hydrolysis of Tetra-iso-propyl titanate above 160℃	—	—	—
Lehoczky et al [33]	Oxidation of evaporated Ti in air at 100℃ - 300℃	Rutile	2.75	—
Wernr & Tracy [3]	Spray deposition of TPT at 95℃ and baked at 200℃ - 450℃	Anatase(X-ray diffraction)	2.18 - 2.20	—
Fuyuke & Matsunami [26]	Vapor deposition of TPT at 200℃ - 400℃	Anatase (X-ray diffraction)	2.24	—
Zang et al [35]	Spray pyrosis of $TiCl_4$ at 330℃	Anatase(X-ray diffraction)	2.1 - 2.1	—
Williams & Hess [38]	Rf. Sputtering of $TiCl_4$ and O_2 at 250℃ - 700℃	Anatase and rutile	—	—
Demiyont & Sites [39]	Ion-beam sputtering of pure titanium	Anatase(X-ray diffractoin)	2.52	—
This work	Spray-on TPT at 200℃ - 390℃	Amorphous and anatase(X-ray diffraction)	2.4 - 2.8	.

ly purified, which is most important for optical properties and, being non-corrosive, it can be stored almost indefinitely if kept in a dry atmosphere. Also, TPT reacts with water to form titanium dioxide leaving behind only volatile by-product [43].

Figure 1 is a schematic of the spray system. In this system, the nitrogen gas (carrier gas) and the TPT meet externally and are then mixed for deposition on the

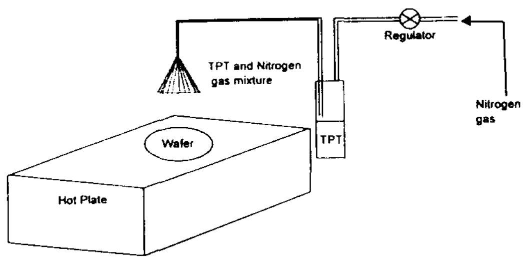


Fig. 1. Schematic showing the set up for the TiO_2 spray-on system.

wafer that is already heated to the required temperature (200 - 390℃). The sprayed TPT on the heated wafers transforms to form TiO_2.

The deposition of TiO_2 film is through hydrolysis and not by reaction of TPT with the ambient oxygen in the air. The chemical reaction is as follows

$$Ti(C_3H_6OH)_4 + 2H_2O \rightarrow TiO_2 + 4(C_3H_7)OH$$

This reaction is endothermic and occur in the gas or liquid phase. At low temperatures (< 150℃) polymers containing the repeat unit TiO_2 can be formed [34].

The formation of titanium dioxide film becomes apparent by the colour changes on the top surface. As the film thickens, it first turns light brown to a darker brown, then seemingly little colour change till it begins to turn to deep royal blue whcih indicates the correct thickness for a quarter wave for anti-reflection coating. Further spraying will change the film colour to light blue and following other colours of the spectrum (yellow, green, red etc.) are visible until the film turns blue again signifying a thickness for half wave interference.

4.1. *Film uniformity*

In carrying out the spray, for a uniform and good quality (in terms of the structure) film the time of spraying matters as well as the temperature. Therefore, to maintain the temperature at reasonably stable value, the spraying has to be done in bits. When the gun is directed to the substrate for too long a time, the temperature comes down and this will result in white specs on the substrate which will peel off during metallisation and will reflect light away as an anti-reflection layer. The uniformity of the film is gotten by spraying with the gun quite close to the substrate with narrow jet for easy control.

5. Characterisation of TiO_2 film

The spray-on titanium dioxide was characterised to ascertain its properties, such as ; structure, refractive index, corrosion resistance as applicable to the intended use. It should be noted that, the film thickness for individual test was different from the other.

5.1. *Structure*

The structure of TiO_2 films was examined by x-ray diffractometry. Films of several hundreds angstroms were deposited on p-type and n-type silicon substrates. No crystalline diffraction was recorded for samples deposited at 200 - 250℃ indicating an amorphous structure. Samples deposited at temperature of 300 - 390℃ exhibited the presence of a crystalline phase which was identified as anatase, the tetragonal form of TiO_2.

5.2. *Refractive index*

Elipsometric measurement of the refractive index was obtained for the as-deposited films and the results summarised in the graph of figure 2, the refractive index of spray-on TiO_2 film as a function of deposition temperature. However, the method explored did not permit the deposition of TiO_2 film at a temperature lower than 200℃. Even at temperature of 200℃, there were so many white specs and as the temperature went higher the white specs reduces. Note that, in figure 2, the refractive index value was measured from 250℃ temperature because, it was easier to obtain specs free films at this temperature than those of lower deposition temperatures (200℃).

5.3. Corrosion resistance

An important requirement of the anti-reflection coating is its resistance to corrosion, stress relief, delamination, cracking or chemical changes over an extended time frame. A simple but rigorous functional test that has shown good correlation with long-term degradation of refractory oxide films in field conditions is the hot water test [3]. In this procedure the sample is immersed in boiling diionized (DI) water for 10 to 20 minutes. The samples of TiO_2 spray-on films on polished both n-type and p-type silicon wafers were boiled for periods of up to 60 minutes to simulate exceptionally severe conditions. Ellipsometric measurements of film thickness and index of refraction showed no change after these tests. The films remain clear, without cracks or microscopically visible defects, and did not peel or delaminate of Scotch tape testing, indicating excellent stability.

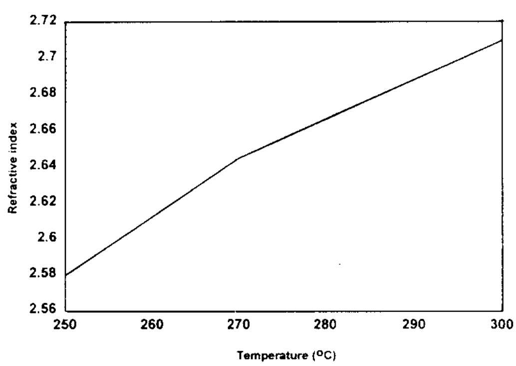
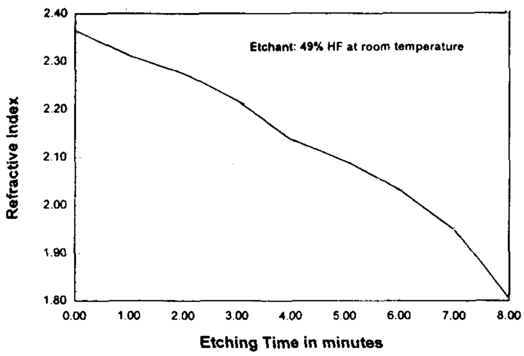


Fig. 2. Refractive index of TiO_2 film as a function of deposition temperature.

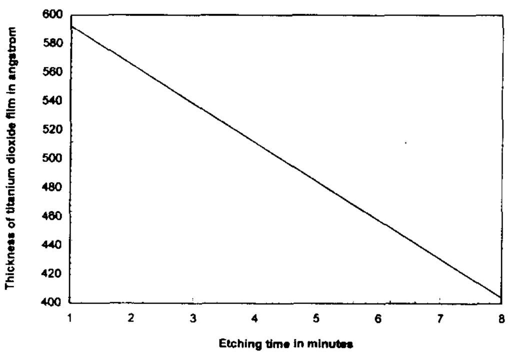
The resistance of the film toward corrosive chemicals was testd by immersing coated silicon for one hour at a temperature of 100℃ in the following aqueous reagents, HCl (49 %), HNO_3 (49 %), H_2SO_4 (97.8 %), H_3PO_4 (49 %), and NaOH. No deterioration or visual changes of any sort occurred on the samples during these tests.

Further test was carried out with 49 % HF and BHF (buffered hydrofluoric acid with ammonium fluoride in the ration of 1 : 5) at room temperature. The coated silicon was immersed in BHF for 30 minutes without any appreciable change in the thickness and the refractive index of the film. The coated samples immersed in the pure HF completely deglaze after about 11 minutes and the etch rate was determined as 36 Å/min. Figures 3a and 3b show the refractive index and thickness of the film as functions of the etching time.

Some of the TiO_2 spray-on silicon samples



(a)



(b)

Fig. 3. (a) Refrective index of TiO_2 as a function of etching time (Note that this sample was sprayed at a lower temperature than the previous figure, that accounts for the descrepancy in the film refractive index), (b) TiO_2 film as a function of etching time (starting thickness of 58.6 nm).

underwent the standard RCA-1 and RCA-2 [41] chemical cleaning and at the end the refractive index and thickness of oxide measured to show no change.

6. Application of TiO_2

The aim of this work is to verify the possible use of TiO_2 film for solar cell processing. These include ; anti-reflection coating, nickel/copper plating mask and mask for diffusion of impurities. The spray-on TiO_2 was, therefore, tested for each of these applications.

6.1. *Anti-reflection coating*

The reflectivity test for the spray-on TiO_2 film was carried out to ascertain its anti-reflection properties. Three p-type silicon wafers with surfaces randomly textured were sprayed with TiO_2 film of different thicknesses for this test. The result is summarised in figure 4, which shows the absolute reflectance as a function of wavelength. This reflectance measurements for these film thicknesses were in agreement with those TiO_2 films deposited with sophisticated equipment [5]. As can be seen in the figure, the thicker the TiO_2 film the more reflectance from the surface. Thus for an effective reduction of reflection from the silicon surface, TiO_2 film of less than 70 nm would be necessary.

6.2. *Nickel/copper plating mask*

The metallisation of solar cell, especially the buried contact solar cell, involves the use of nickel as the first metal layer to form nickel silicide which blocks the copper metal that follows. The electroless nickel/copper (Enplate Cu-704) supplied by Ethone OMI International in Sydney, contains some NaOH which at the plating temperature of

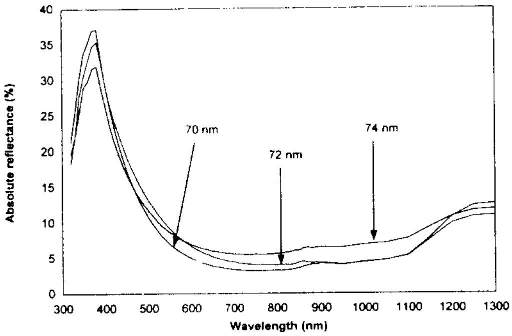


Fig. 4. Absolute reflectance measurements of spray-on TiO_2 film on various thicknesses on textured wafers.

42℃ normally has etching effect on SiO_2. Some spray-on TiO_2 samples were immersed in this solution for various times ranging from 5 hours to 35 hours. At the end of the experiment the thickness and the index of refraction of TiO_2 film were measured and there was no change. Also the hot water test was carried out at the end and this had no effect on the properties of TiO_2.

6.3. Diffusion mask

As a diffusion mask, the titanium film should also posses a passivating quality to reduce the possible surface recombination velocity which would normally degrade the electrical performance of the cells. Also, the bulk lifetime of the cells have to be preserved after the high temperature processing. Thus it was necessary to carry out the minority carrier measurements of the wafers with spray-on titanium dioxide films. Three samples of different film thicknesses were prepared using 80-100 ohm-cm, n-type silicon wafers. The minority carrier lifetimes of these samples were measured immediately after TiO_2 film had been sprayed-on, than after the dry oxidation for 30 minutes at 1000℃ in oxygen ambient. The fourth sample was a control wafer. The summary of results is given in table 2. The results given in this table (table 2) includes both the surface and bulk minority carrier lifetimes.

7. Discussion

As has been seen with all the tests, the TiO_2 film adhered well, and showed no cracks even after high temperature annealing. The slight increase in the oxide thickness after dry oxidation is consistent with the work of Rausch and Burte [30] following the growth of very thin silicon dioxide film underneath TiO_2 film. The minority carrier lifetime for the three samples and control wafer were observed to remain unchanged before and after dry oxidation. This indicates that, the sprayed TiO_2 film using the simple system does not exhibit any contaminating properties. It also suggests that, the underlying silicon dioxide can act as a surface passivation layer for solar cell. Of particular importance is the slight increase in the refractive index which is useful for encapsulated cells. The increase in the refractive index is mainly due to the densification of the film. Thus a possible change of structure, from anatase to rutile which exhibits such refractive indices.

Table 2
Minority carrier lifetime measurements of spray-on TiO_2 film before and after high temperature annealing in nitrogen ambient

Sample identification	TiO_2 thickness (nm)		Refractive index		Minority carrier lifetime (micro sec)	
	before	after	before	after	before	after
Control sample	–	10	–	1.45	22.5	22.5
Sample 1	69.5	70.5	2.38	2.65	37.5	37.5
Sample 2	72.4	73.3	2.41	2.66	58.8	58.8
Sample 3	71.7	72.4	2.43	2.67	28.8	28.8

More work is being done on the spray-on titanium dioxide for incorporation into the processing sequence of the buried contact solar cell. The possble processing sequence for buried contact solar cells with the inclusion of this film as masking layer would be as follows :

1. Emitter and rear floating or back surface field junction formation using spin-on diffusion sources,
2. Titanium dioxide film spray-on at 200-300℃,
3. Front and rear grooves scribing (for the case of double sided cell) and cleaning,
4. Application of spin-on phosphorus and boron to front and rear grooves respectively,
5. Driven-in at one high temperature,
6. Metallisation,
7. and Testing.

Comparing the proposed processing sequence with the conventional sequence for buried contact solar cells, there is enormous advantage, especially in cost savings in both equipment and time. This will mean that a buried contact solar cell can be processed at a time less than half the conventional processing sequence. This will reduce the cost of solar cells drastically and the use of low lifetime wafers can be processed with reasonable energy conversion efficiency because of the reduction in time of exposure to high temperature.

8. Conclusion

The properties of the spray-on titanium dioxide have been found to be consistent with other deposition methods using sophisticated equipment. The spray-on TiO_2 has been exposed to several tests including the corrosion and high temperature tests which would enable the spray-on film to be used as plating and diffusion masks respectively. The film passed all the tests without showing any crack nor deterioration in the minority carrier lifetimes of the silicon substrates. Also the reflectance test was consistent with the results of other workers.

The formation of a thin film of SiO_2 during the high temperature annealing makes the spray-on TiO_2 film very compatible with solar cell processing. This film (SiO_2) would passify the silicon surface to reduce the possible recombination activities at both front and rear surfaces of silicon substrates. This would enable the solar cells using the spray-on TiO_2 film to perform as well as the conventional oxidised cells.

On the whole, despite the simplicity of the deposition technique, the resulting spray-on TiO_2 film was of good quality which would be very useful for the intended application in solar cell processing.

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