Optical properties of refractory TiN, AlN and (Ti,Al)N coatings

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ABSTRACT

Titanium nitride is a golden-colored semiconductor with metallic optical properties. It is already widely used in room temperature spectrally-selective coatings. In contrast, aluminum nitride is a relatively wide-band gap, non-metallic material. Both nitrides have exceptional thermal stability, to over 1000 °C, but are susceptible to oxidation. We will show here that composite coatings consisting of these materials and their complex oxides have considerable potential for spectrally-selective applications, including at elevated temperatures. In particular, we examine the metastable materials produced by magnetron sputtering. The effective dielectric functions of these materials can be tuned over a wide range by manipulation of their microstructure. This provides a strategy to assemble materials with tunable dielectric functions using a 'bottom-up' approach. The results are compared to those achievable by conventional, 'top-down', planar optical stacks comprised of alternating layers of TiN_x and AlN.

Key words: Titanium nitride, aluminium nitride, optical properties, optical simulation, solar absorbers

1. INTRODUCTION

The availability of thermophotovoltaic (TPV) and advanced solar thermal power systems opens up a wide spectrum of possibilities to convert light into electric power. Improvement in the performance of heat-resistant and spectrally-selective coatings is a key requirement for achieving higher efficiencies in both types of technology. Here we will show that composites based on AlN, TiN, TiAlN and their complex oxides are one of the most promising choices for such coatings.

The compound AlN is known for applications in electro-optical devices and optoelectronics. It is a wide-band gap ($E_g = 6.2$ eV) dielectric with good thermal resistance behaviour, high refractive index (about 2 in the visible range) and low-absorption coefficient ($k < 10^{-3}$). This binary system is also transparent to incident light in the visible and infrared region, which makes AlN suitable for applied optics[1]

TiN is widely applied as a wear-resistant coating and diffusion barrier in contact structures. This is due to the fact that TiN exhibits enhanced hardness and high temperature stability (up to 800°C). Interest in this material increased even more when it was noticed that the TiN system is similar to gold in terms of optical behaviour: it has a metallic nature and acts as a strong reflector of thermal radiation in the infrared region. Therefore, like gold, it can be used in heat-mirror applications. In addition, it was discovered that TiN exhibits good plasmonic properties [2,3]. Plasmon resonance in TiN nanoparticles can be used to increase their absorption cross-sections. Although elements such as gold or silver can sustain more intense localized surface plasmon resonances than TiN, they are unsuitable for high temperature applications due to the diffusive mobility of the noble metal atoms. In addition, TiN has a much lower cost.

TiAlN is well-known as a hard coating for elevated temperature applications. It can be considered as a derivative of TiN, where the additional Al content acts to improve resistance to oxidation and increase hardness [4]. There seems to be some controversy regarding the microstructure of such coatings. Some authors reject the formation of a solid-solution compound of the form (Ti,Al)N and insist on the presence of different phases (e.g.TiN, AlN). Indeed, in [5] the author demonstrated that TiAlN compounds obtained by means of physical-vapor deposition (PVD) consisted of three phases, TiN, Ti_2N and AlN. Gago et al. also demonstrate the existence of a TiAlN alloy, obtained by DC

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magnetron sputtering, containing three distinct phases, which are *cubic*-TiN, *cubic*-AlN, and *wurtzite*-AlN. [6]. According to other workers, however, ternary nitride systems like TiAlN represent interstitial alloys and can form a continuous range of NaCl-type structures where the Ti, for example, is partially substituted with another metallic atom of compatible characteristics, such as Al [7,8]. Such a system is considered to be thermodynamically unstable as even slight deviation in stoichiometry prevents the formation of solid solution and leads to formation of several phases [7]. The metastability of the as-deposited TiAlN system has been shown in a number of papers. The decomposition of TiAlN may occur in two ways, by spinoidal segregation into two cubic phases or by precipitation of AlN phase [6]. For example, in[4] it was shown that annealing of TiAlN thin films for 40 minutes at 1000°C led to decomposition of the (Ti,Al)N compound and formation of the *c*-TiN and *c*-AlN phases. TiAlN has also started to receive attention as an optical material for sun control applications [9]. The peculiarity of this ternary nitride system is that its properties can be tuned from dielectric to metallic behaviour by changing the ratio of the elements [9].

There is also a range of related materials which contain oxygen. Aluminium oxynitride (AlON) for example also has a high melting point and a relatively high refractive index (~1.7 to 1.8)[10] This compound is sometimes formed instead of AlN during thin film deposition due to contamination by oxygen or H_2O [11]. Similarly, a material designated 'TiAlON' has been used in spectrally-selective coatings[12], however, is reportedly not a single compound but rather a fine-scale composite of $(Ti,Al)N_x$ and $(Ti,Al)O_2$ [13].

For solar selective applications, the AlN, TiN and TiAlN compounds and the complex oxide phases derived from them seem to be suitable candidates, where the main requirements are 1) high temperature stability and oxidation resistance from the point of view of response to temperature changes and 2) low reflectivity in the visible range of the spectrum, but high in the near-infrared to prevent energy losses and achieve low emittance. However, the latter requirement is not always achievable by applying only a single layer. To increase the spectral selectivity further, a multilayer or graded structure could be applied. The first one usually implies using three-layer stack, which can be further modified by using additional layers. This basically includes 1) strongly reflective bottom layer, 2) highly absorbing middle layer and 3) anti-reflecting top layer. Graded structures enable improved solar absorbance as the gradual changes in refractive index throughout the medium allow minimisation of the reflectance that would otherwise occur due to step changes in index [14].

In the present study, AlON, TiN and TiAlN films of various types were made and characterized in order to investigate their response to light, in particular as a function of chemical composition. We also modelled the optical properties of hypothetical multilayer stacks or graded layer structures of these materials in order to determine whether combinations of several materials would improve spectral selectivity further.

2. EXPERIMENTAL DETAILS

Ti, Al, and Ti plus Al were deposited in a vacuum chamber evacuated to a base pressure of 1.1×10^{-6} Pa on rotating Si (101) and glass substrates by means of direct current (DC) magnetron sputtering in the presence of Ar and N_2 reactive gases. A Ti target with purity of 99.999% and an Al target with purity of 99.999% were used. The Si substrate was first cleaned with acetone and methanol in an ultrasonic bath. The glass substrate was cleaned in a soap solution in the ultrasonic bath. Films were deposited at room temperature or onto a heated substrate (nominally 350°C**)**. The target-to-substrate distance was kept constant. Thickness of the coatings was monitored during the deposition process with a quartz crystal microbalance. The deposition parameters are presented in Table 1.

X-ray diffractometry (XRD) using Cu Kα radiation was used to determine the crystal structure of the films. Film thickness was determined by profilometry and spectroscopic ellipsometry. Optical constants *n* and *k* were extracted from spectroscopic ellipsometry by fitting theoretical and experimental data obtained at 65°, 70° and 75° angles of incidence. Transmittance and reflectance spectra were obtained by spectroscopy at normal incidence. The elemental composition and elemental distribution were investigated using a scanning electron microscope with energy dispersive spectrometer (EDS). The size of the grains was estimated using SEM. Woollam Co. WVASE ellipsometric software was employed to simulate the optical response (R%)of multilayer stacks and graded layers on different substrates.

Table 1 –Deposition conditions

Sample	N_2 pressure, SCCM	Ar pressure, SCCM	Current for Ti target, Amps	Current for Al target, Amps	Deposition rate, Å/s	Nominal substrate temperature, ºC	Profilometer thickness, nm	Optical thickness, nm
AlON	50.7	62.8	-	0.4	0.2 – 0.3	24	140	140
TiN	11.5	32.5	0.518	-	0.5	460	440	n.d. §
TiAlN	52	59.5	0.329	0.333	0.3 – 0.4	24	460	406

§no measurable transmittance

3. RESULTS

3.1 ‘AlON’ thin film

3.1.1 Structure and elemental composition

The thin film that was originally intended to be AlN did not show any XRD peaks and was evidently amorphous. This is a similar result to that obtained by Sharma et al. for AlN samples made at room temperature by reactive ion beam sputter deposition [15]. The formation of a non-crystalline structure can be explained by the lack of activation energy to drive the sputtered atoms into a crystalline configuration at room temperature [16]. In contrast, it has been shown [15] that crystalline structures will generally be achieved if a material is deposited onto a heated substrate.

EDS analysis of the film indicated that its composition was about 24 at% Al, 9 at% N and 67 at% O. This indicates a deficiency of nitrogen atoms and significant oxidation of the sample. At face value the film must therefore consist of a mixture of a small amount of amorphous AlN plus a larger amount of amorphous Al_2O_3, or a mixture of AlON and Al_2O_3. This sample will, for convenience, be referred to as the ‘AlON’ sample in the discussion that follows. EDS mapping showed that the distribution of these three elements was homogenous across the film at the magnification used (266×).

3.1.2 Optical properties

Optical transmission spectra of the ‘AlON’ sample deposited on a glass substrate are presented in Figure 1. The film reveals high transmittance in the visible and near infrared spectrum. The highest transmittance value is ~90% across both the visible and infrared spectrum, which is essentially the same result as for the glass on its own. Indeed, it is the glass substrate that is responsible for the fall off in transmittance in UV. Figures 2 and 3 show *n* and *k* coefficients for the film of ‘AlON’ on silicon extracted using WVASE software. They were obtained by fitting measured ellipsometric parameters Δ and Ψ to a theoretical model. In this three-layer model the film indices were adjusted to fit the experimental data. The bottom layer represented the Si substrate, the middle layer silicon dioxide, which takes into account native oxidation of the substrate, and the top layer represented AlON for which a Lorentz oscillator model was used to fit the measured optical response. The value of the refractive index of the top later varied between 1.74 and 1.63. These values are significantly lower than those usually reported in the literature for AlN in the visible part of the spectrum (where *n* is generally reported to lie between 1.9-2.1 [17]) but a reasonable match for AlON [10] or Al_2O_3 [18] provided some porosity in the films is accepted. However, a similar range of values (*n* = 1.67-1.84) was reported by Choudhary et al. [19] and Hajakbari et al.[20] for AlN. One explanation for a reduced value of *n* in an AlN layer may be the presence of voids [19]. In the present instance, however, the more probable explanation is that the film is simply a mixture of amorphous AlON and Al_2O_3. The value of extinction coefficient *k* approached zero, pointing to the absence of the absorption in the material.

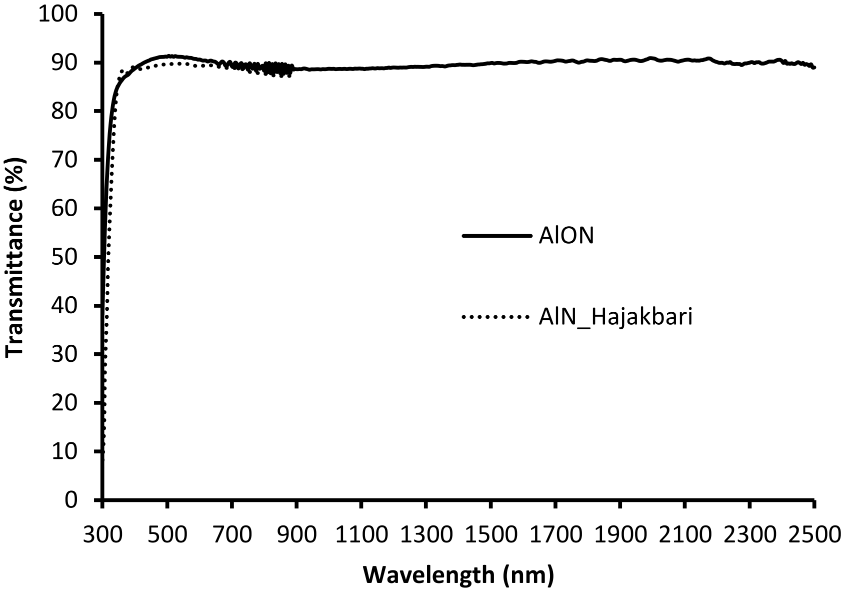


Figure 1 – Transmittance spectrum of amorphous thin film of 'AlON' of 160 ± 20 nm thickness on a glass substrate.

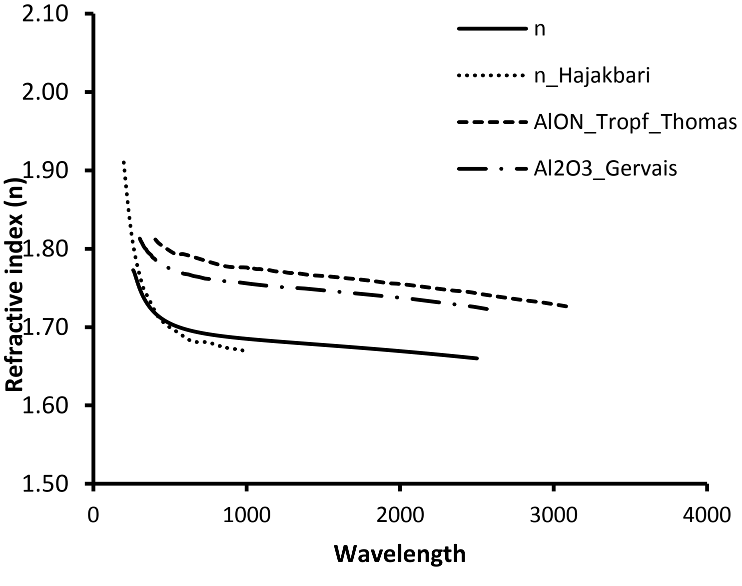


Figure 2 – Refractive index *n* and extinction coefficient *k* of AlON thin film of the present work. For comparison data for AlN [20,21], Al_2O_3 [18] and AlON[11] are also shown.

3.2 TiN thin film

3.2.1 Structure and elemental composition

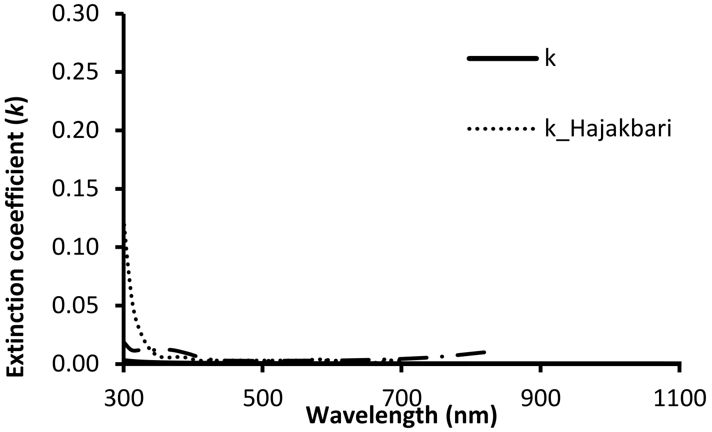


Figure 3 – Extinction coefficient *k* of 'AlON' thin film of the present work. For comparison, data for the AlN thin film deposited by Hajakbari [20] and as reported for the bulk phase by Loughin and French [22] are shown.

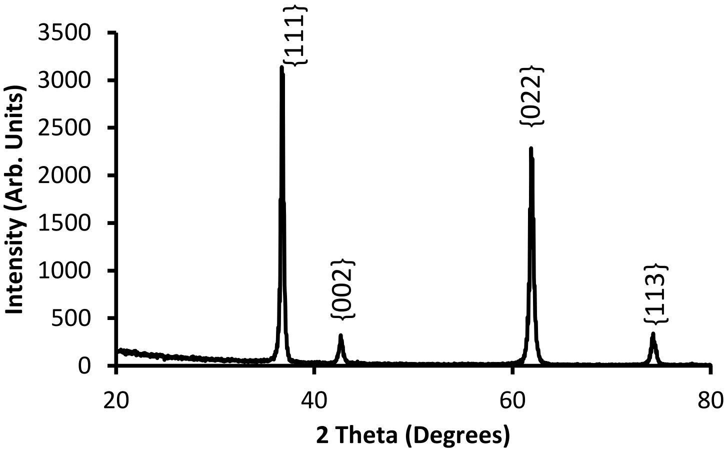


Figure 4– XRD pattern of TiN film

An XRD pattern of the as-deposited TiN film is shown in Figure 4. The XRD peaks matched with the cubic structure reported for TiN_x. The lattice parameter was found to be in the range 4.2143 ± 0.0004 Å by Rietveld refinement. The lattice parameter of TiN_x is known to vary with *x* and the substrate on which it had been deposited (due to residual stresses). Generally, a value in the range of 4.236 to 4.239 Å seems appropriate for a compound with stoichiometry in the range $TiN_{0.90}$ to $TiN_{0.95}$ (see entry # 1101046 in the Crystallography Open Database (http://www.crystallography.net/) as well as Wahlstrom et al.[23]). A reduced nitrogen content is correlated with a smaller lattice parameter. For example, in [24] a lattice parameter of 4.221 Å was reported for $TiN_{0.61}$. At face value, and assuming Vegard's law holds, then lattice parameter of the present film suggests that it could be sub-stoichiometric in nitrogen. In contrast, EDS analysis of the TiN film indicated 44 at% Ti, 52 at% N and a negligibly small amount of oxygen (3.6 at%). In this case, if the analyses are considered to be accurate, then a super-stoichiometric TiN_x is indicated. Taking into account the well-known measurement inaccuracies of both thin film GIXRD and of EDS of elements of low atomic number, we conclude that the TiN in our film may be taken to be close to stoichiometric as a first order approximation.

3.2.2 Optical properties

The optical reflectance curve for thin film TiN on a Si substrate is presented in Figure 5. The reflection spectrum for this film has a distinct minimum in the blue-green region, a sharp rise in the mid and far (red) visible spectrum, and reaches a maximum in the near-infrared. The highest value of reflection exhibited in the infrared was 82%.

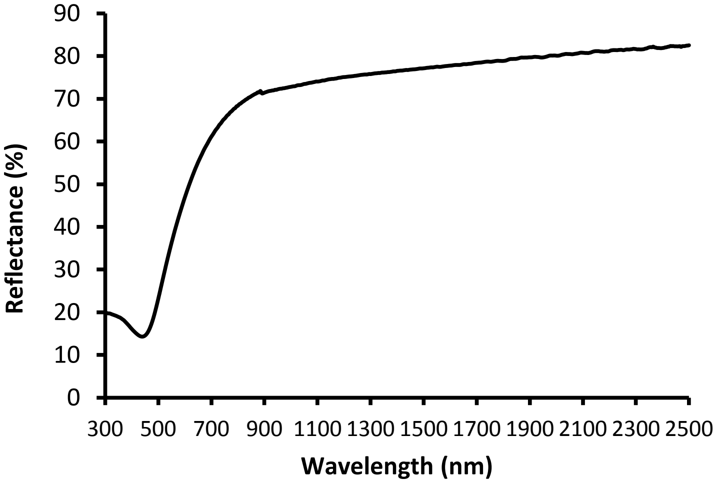


Figure 5 – Reflection spectrum of as-deposited TiN film deposited on Si substrate

Figure 6 shows the dispersion of refraction index *n* and extinction coefficient *k* for the TiN thin film. Both optical constants increase with a wavelength. An increase of reflection at $\lambda > 500$ nm is caused by the onset of the plasmonic response with $k > n$ and arises from the Drude response of electrons in the conduction band of TiN. Absorption is still significant at $\lambda < 500$ nm and is dominated there by inter-band transitions.

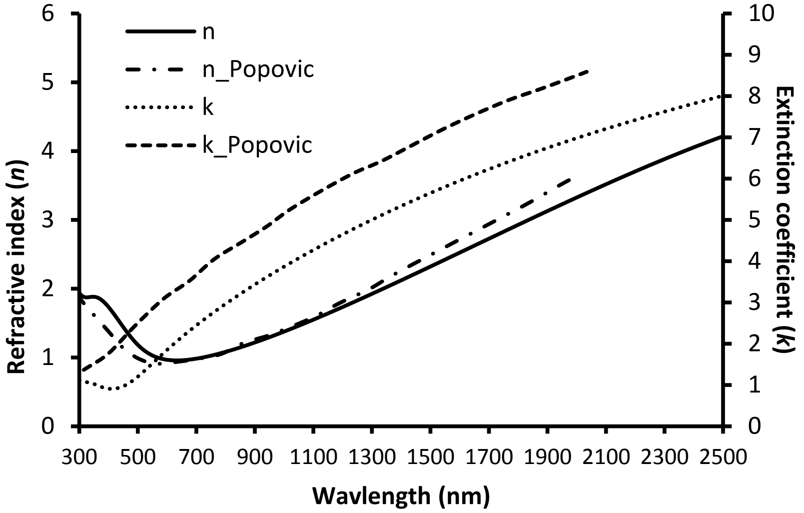


Figure 6- Refractive index, *n*, and extinction coefficient, *k*, of TiN film and reference TiN film deposited by Popovic et al. [25]

3.3 TiAlN film

3.3.1 Structure and elemental composition

Grazing incidence X-ray diffraction patterns, Figure 7, showed that the TiAlN film had a nanocrystalline polycrystalline structure with some amorphous inclusions. Broadening of the peak between 30° and 40° indicates the possible presence of amorphous phase in the thin film.

The EDS results showed that the major elements in the coating are 30 at% Al, 46 at% N, Ti 10 at% Ti and 15 at% O. The presence of oxygen suggests the formation of one of the complex oxygen-containing phases mentioned previously.

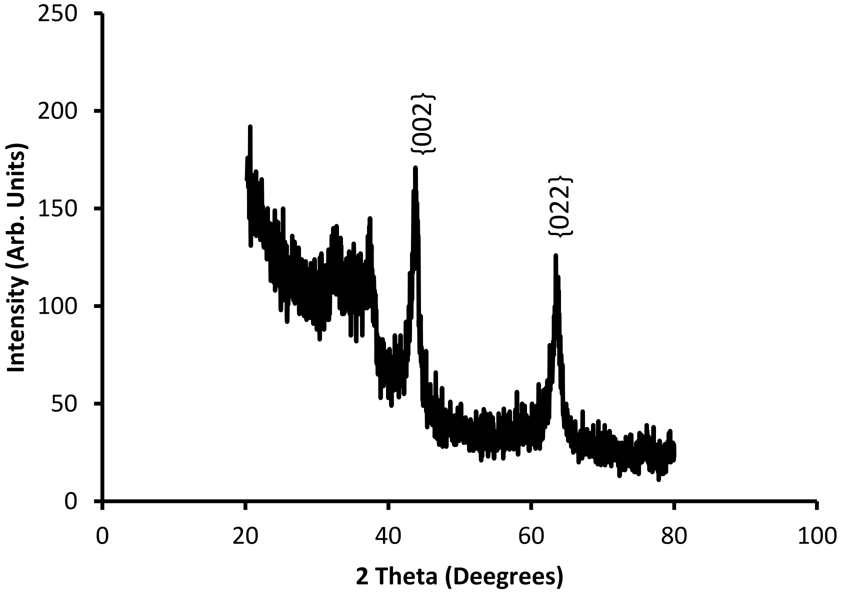


Figure 7 – XRD pattern of TiAlN film. The positions of the reflections for TiN are indicated.

3.3.2 Optical Properties

Some compositions of TiAlN may be suitable for solar-thermal applications [26], although, as mentioned above, decomposition of the material into two or more individual phases is a possibility during service at elevated temperatures. The reflectance and transmittance of a 280 nm TiAlN film deposited on a glass substrate are shown in Figure 8. The coating exhibits the characteristic thin film interference oscillations of a dielectric (low *k*) material at longer wavelengths but good absorptance below 400 nm as observed previously[27]. The latter aspect is also clearly visible in the plot of *n* and *k* , Figure 9. A steep rise in the blue-green part of the spectrum affects color and an increase through the far visible and NIR, modulated by both interference oscillations and additional absorption processes, is observed. This film has a composition ratio of around 75% of AlN to 25% TiN, and so contains more Al than can be taken up into the $(Ti_yAl_{1-y})N_x$ solid solution. Therefore, it is probably a nanoscale composite of semiconducting $(Ti_yAl_{1-y})N_x$ dispersed within an insulating AlN or AlON matrix. The *k* spectrum in Figure 8 indicates a much reduced band gap for the composite relative to pure AlN, which is why it is green. This spectrum also indicates that strong, below-the-band-gap, absorption occurs.

4. SIMULATIONS

Multilayer spectrally selective stacks for solar absorber and cool color coating applications were modelled using WVASE software by JA Woollam Co. The stacks were simulated using optical constants of the deposited TiN and AlN thin films (Figure 2, 3, 6). Two different concepts of selective absorbers were used. The first model implies building a multilayer with exploitation of alternate metallic and dielectric layers. Such design enables an increase an absorptance of the coating in a select band due to multiple reflection of the light, Figure 10(a) [14]. The second approach is a graded metal -dielectric composite layer, where absorptance can be achieved due to optical transitions in the metallic part of the structure, Figure 10(b). [14]. Three models are presented here for discussion; one multilayer stack and two graded composition structures on 1 mm thick Si and Cu substrates. Optimal optical thickness of the each layer was reached by minimizing reflectance in the absorbing range and maximizing reflectance in either the infra red beyond 2.5 μm for solar absorbers (which need metal substrates) or NIR for cool colors (which would have an overlayer of a solar transmitting high emittance material).

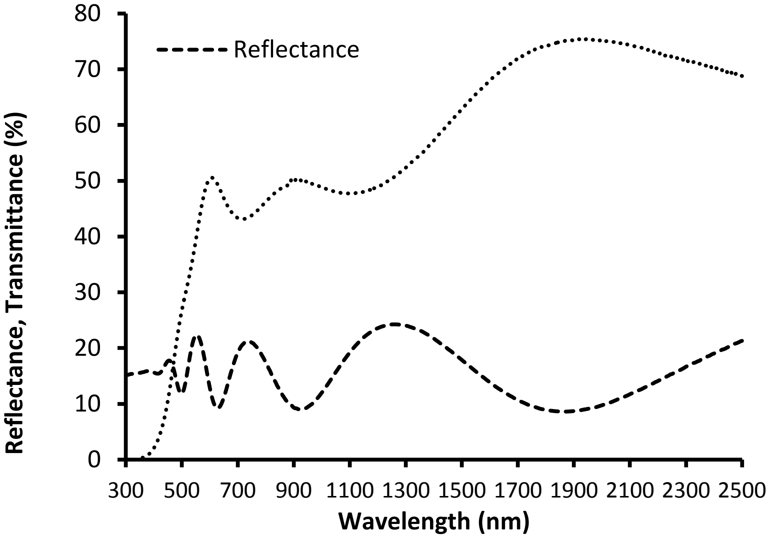


Figure 8 – Reflectance and transmission spectrum of a TiAlN thin film on a glass substrate

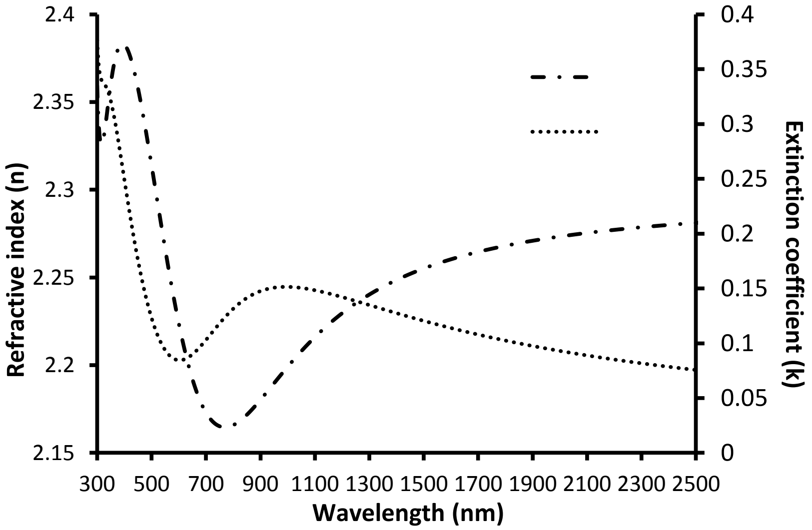


Figure 9 - Refractive index and extinction coefficient of TiAlN film

3	aln_g	38.100 nm
2	tin_nk	120.447 nm
1	alon_sell	80.200 nm
0	si_jaw2	1 mm

a)

3	Graded (EMA)	1000.000 nm
2	EMA alon_sell/10% (tin_nk)	0.000 nm
1	tin_nk	0.000 nm
0	cu_palik_g	1 mm

b)

Figure 10 – Simulated models of a) multilayer stack on Si substrate and b) graded AlN - TiN composite on Cu substrate

Figure 11 shows the reflectance one of the model stacks, in this case designed on a Si substrate. To achieve spectral selectivity, a TiN layer was used as a near infrared reflecting layer. The top dielectric AlN layer was added to broaden the absorption in the visible spectrum range. A four-layer configuration enabled the design to reach higher reflectance in the infrared spectrum. Addition of more layers did not lead to further rise in reflectance. The maximum value of reflectance was 82% in the NIR. An ideal colored NIR reflector requires the reflection transition to be even more abrupt but that in Figure 11 is already potentially useful.

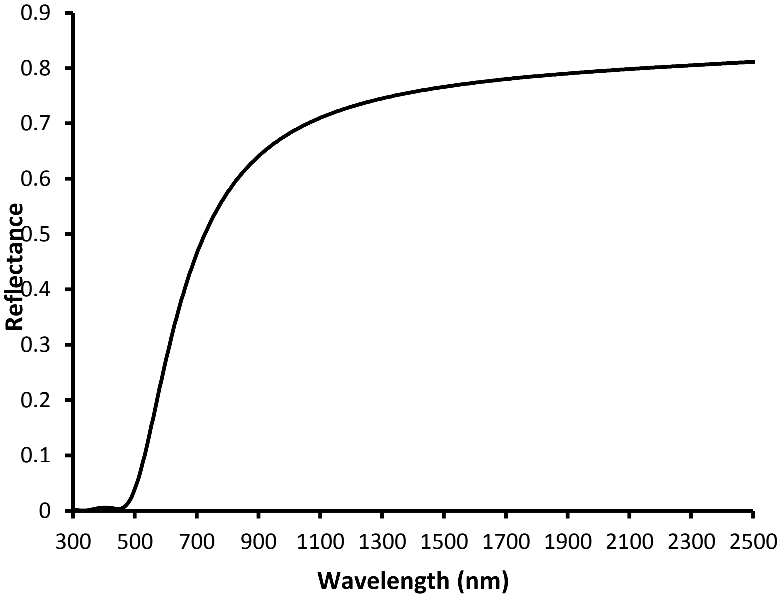
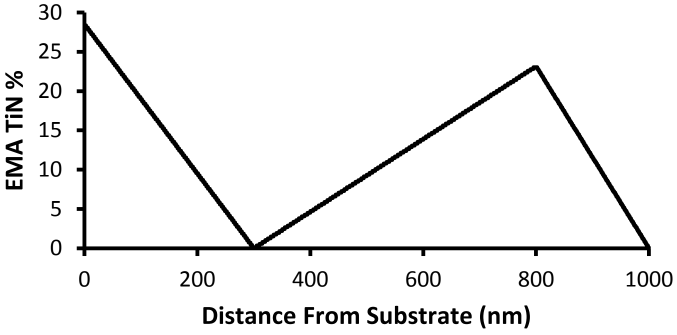


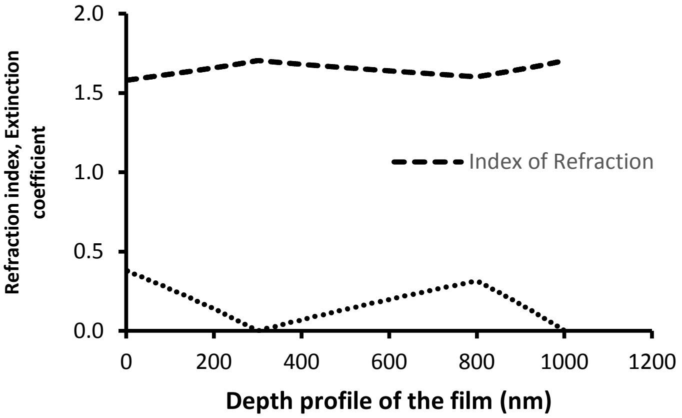
Figure 11 – Reflection spectrum of modelled multilayer stack AlN/TiN/Si

The second model applies a dielectric of aluminum oxynitride containing titanium nitride onto a copper substrate. A graded profile of the compositionally graded film is presented in figure 13 and indicates a nonlinear optical response to the incident light. The thickness of the coating was chosen to be 1000 nm. A graded profile of the compositionally graded film is presented in Figure 12 and its response to light in Figure 13, and indicates a nonlinear optical response to the incident light.

From Figure 13 it can be seen that a graded AlN-TiN structure on a Cu substrate provides a nearly ideal solar absorbing coating with an abrupt transition near 2700 nm. The reflectance of the coating/substrate combination was around 87% in the IR. Such a high value of reflection was achieved in part due to selection of Cu substrate. Cu is famous for its strong reflection in the IR spectrum and is widely applied in the optical areas [28]. To collect solar thermal energy efficiently the maximum absorption of the incident light must span the solar range.



(a)



(b)

Figure 12 – A graded profile of the graded AlN-TiN composition structure on a Cu substrate, at 500 nm. (a) Design of film, (b) Optical properties of film.

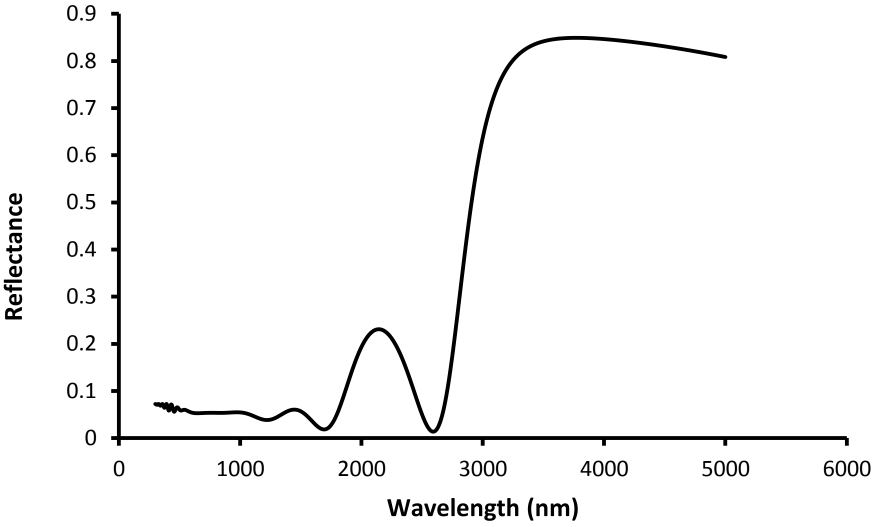


Figure 13 – Reflection spectrum of modelled AlN-TiN graded layer on Cu substrate

All of the modelled optical structures showed technologically attractive responses to incident light. The optimal values of thickness (20 – 80 nm) for each layer in the model are completely feasible to achieve with current deposition techniques. The best model of those examined was the graded model AlN-TiN (10%) on Cu. This exhibited the best optical behaviour in terms of correspondence to spectral selectivity requirements. The minimum of reflection (R ~ 0.03 - 0.05) was reached in the near IR range (1000 – 1600 nm and around 2530 nm). After that the reflection trend started to increase sharply reaching a maximum of about 83% at 3400 nm. The proposed simulated model film design is suitable for solar selective applications as it satisfies the main requirements mentioned in the introduction.

6. CONCLUSIONS

This study explored the optical properties of thin films of crystalline TiN, amorphous 'AlON' and TiAlN. Experimental samples were deposited by DC magnetron sputtering. This method enables thin films of different stoichiometries, composition and thicknesses to be fabricated. The formation of crystalline phase was facilitated by deposition on a heated substrate (460°C) and relatively high deposition rate (0.5 Å/s). The experimental TiN_x film was close to stoichiometric composition, which resulted in high reflectance and Drude-like properties. The deposited 'AlON' film formed an amorphous phase, which was attributed to the deposition conditions. This sample revealed high transmittance (about 90%) and reduced values of optical constant *n.* The deposited Ti_xAl_{1-x} film contained mixed phases, which was also proved by transmittance and reflectance spectra, where absorption due to TiN phase was observed.

We also used simulation techniques to explore solar absorbers made using the above materials. The main idea was to achieve minimum reflectance in the solar range and maximum reflectance in the infrared. Overall, one can notice that improved spectral selectivity can be achieved by combining several materials with dielectric, semiconducting or metallic behavior into a stack. Modelling showed that the already good performance of a simple multilayer stack could be further improved by minimizing reflection in the solar spectrum with a graded composite. The optimum performance was obtained using a combination of multilayer stack and graded composite.

Acknowledgements

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