



# Plasma polymerised thin films for flexible electronic applications



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## ABSTRACT

The significant advancement and growth of organic and flexible electronic applications demand materials with enhanced properties. This paper reports the fabrication of a nonsynthetic polymer thin film using radio frequency plasma polymerisation of 3,7-dimethyl-1,6-octadien-3-ol. The fabricated optically transparent thin film exhibited refractive index of approximately 1.55 at 500 nm and rate of deposition was estimated to be 40 nm/min. The surface morphology and chemical properties of the thin films were also reported in this paper. The optical band gap of the material is around 2.8 eV. The force of adhesion and Young's modulus of the linalool polymer thin films were measured using force-displacement curves obtained from a scanning probe microscope. The friction coefficient of linalool polymer thin films was measured using the nanoscratch test. The calculated Young's modulus increased linearly with increase in input power while the friction coefficient decreased.

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## 1. Introduction

The past few years have seen a significant and rapid growth in research and development of organic thin film devices [1]. The advancement is especially noticeable in organic electronic systems such as flexible and large-area electronic displays, thin film transistors and thin film organic photovoltaic devices [2,3]. Many commercial organic devices are already in the market, typically organic light emitting diodes, organic field effect transistors (OFETs), organic solar cells, and polymer coated contact lenses or catheters [4,5]. One of the major driving forces behind the organic semiconductor research is the desire to replace inorganic materials, such as silicon and germanium, with lower fabrication cost and environmentally friendly alternatives [6]. Other significant advantages of organic semiconductors include the reduced number of physical defects compared to inorganic semiconductors, and ability to tailor material properties by choosing a precursor from a vast array of available volatile organics. This work focuses on the development of an insulating material, which can be incorporated in the OFET structure to make the flexible electronics a reality [7,8].

Surface modification of bulk materials using plasma enhanced technologies is an attractive method of imparting desirable surface properties onto the material while preserving its bulk characteristics [9,10]. Plasma polymerisation is broadly used for fabricating polymer thin films from volatile monomers [11,12]. A high degree of material diversity of the polymer thin films can be attained by changing the

deposition parameters such as pressure, monomer flow, and input radio frequency (RF) power, to name a few [13–15]. As such, physico-chemical, mechanical, and surface properties, as well as the degradation behaviour of the plasma polymerised thin films can be tailored to suit a desired application [16]. Furthermore, the technique can be effectively employed for the polymerisation of organic compounds that may not undergo conventional thermo-chemical polymerisation [17].

The precursor 3,7-dimethyl-1,6-octadien-3-ol (linalool) is a naturally occurring terpene alcohol. Linalool is a key constituent of several essential oils, including lavender oil, basil, rosewood and coriander and is primarily used in fragrances and aromatherapy for its perfume and medicinal characteristics that include anti-stress, antibacterial and sedative properties [18–20]. This work investigates the feasibility of using plasma polymerisation to fabricate thin solid films from this naturally occurring compound without the need for additional processing. Research has previously been conducted to investigate properties of polymer thin films fabricated from eucalyptus and lavender oils [21–24] and components such as terpene-4-ol [8,14,25,26] and linalyl acetate [27,28].

## 2. Experimental details

### 2.1. Film fabrication

Polylinalool thin films were fabricated in accordance with the experimental procedure outlines elsewhere [29]. Typically the fabrication system consists of an RF plasma generator, a custom-made glass tube ( $l$  70 cm,  $d$  5 cm), a vacuum pump and vacuum gauges. We have fabricated the thin films at a pressure of 13 Pa, RF frequency of 13.56 MHz and at various RF power levels. For each deposition,

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1 ml of linalool was placed in the monomer flask. The monomer flow rate,  $F$ , was measured using a procedure outlined in previous studies by Gengenbach and Griesser [30]. The reactor chamber was first evacuated, then the monomer was released into the chamber. Once the pressure had stabilised, the vacuum valve was closed; the pressure was recorded every 5 s for 1 min. The flow rate  $F$  was calculated as  $F = dp/dt \times 16172 V/T$ , where  $V$  is the volume of the chamber,  $T$  is the temperature,  $p$  is the pressure inside the reactor chamber and  $t$  is time.  $F$  was estimated to be  $13.75 \text{ cm}^3/\text{min}$  during the polymerisation process.

## 2.2. Material characterisation

Optical constants and thickness of poly(linalool) thin films were estimated from ellipsometer parameters  $\Psi$  and  $\Delta$  collected over the wavelength range of 190–1000 nm using a variable angle spectroscopic ellipsometer (J.A. Woollam Co., Inc., model M-2000D). Gaussian and Tauc–Lorentz oscillators were applied to both reflectance and transmission data to obtain the best fit to the experimental data. Direct and indirect optical band gap of poly(linalool) was calculated using the Tauc relation applied to the absorption data obtained using an Avantes AvaSpec 2048 UV–vis spectrometer. Tauc relation is described as  $\alpha h\nu = B (h\nu - E_o)^p$ , where  $\alpha$  is the absorption,  $h\nu$  is the energy of light,  $B$  is related to the length of localised state tails,  $E_o$  is the optical band gap and  $p$  is a constant that determines the type of transmission. For direct band gap, the Tauc relation is applied for  $p = 1/2$ , whereas for indirect band gap,  $p = 2$  is used.

The surface morphology of the poly(linalool) thin films was investigated using the NT-MDT NTEGRA Prima atomic force microscope (AFM). A Hysitron Triboscope Nanomechanical unit in conjunction with the AFM system was used for nanoscratch test to determine the friction coefficient of the poly(linalool) material. Scratches were performed using a Berkovich tip, with a ramped load with the upper limit varied from 1000 nN to 2000 nN over 30 s. The cross hatch test using the Elcometer 107 kit was also performed to obtain a standardised (ASTM D3359) qualitative assessment of the adhesion between poly(linalool) films and glass substrate.

The Veeco TMX-2000 Explorer scanning probe microscope (SPM) was used in force-versus-distance mode ( $f$ - $d$  mode) to generate force–displacement curves measuring the interactions between the tip and sample surface [31]. For each poly(linalool) sample,  $f$ - $d$  curves were generated at 15 locations using a conical tip and a cantilever spring constant of 0.08 N/m. The adhesion force of the sample was calculated as  $F_{ADH} = (SP + D_0 + D_A) \times k/m_s$ , where  $F_{ADH}$  is the adhesive force,  $SP$  a set point,  $D_0$  the zero level for the curve,  $D_A$  the adhesive deflection and  $m_s$  the gradient of the approach curve for silicon (in this case  $m_s = 0.14$ ). Young's modulus was calculated as  $F_{cone}(\sigma) = 2E^* \sigma^2 / (\pi \tan \alpha)$ , and  $1/E^* \approx (1 - \mu^2) / E_{sample}$ , where  $F_{cone}$  is the applied force of the conical tip,  $\sigma$  the indentation,  $E^*$  the relative Young's modulus,  $\alpha$  the half angle of the tip (in this case,  $\alpha = 11^\circ$ ),  $E_{sample}$  the Young's modulus of the sample, and  $\mu$  Poisson's ratio for the sample. Poisson's ratio of 0.35 is assumed for poly(linalool) thin films.

## 3. Results and discussion

### 3.1. Deposition rate and films thickness

The spectroscopic ellipsometry studies demonstrate that the poly(linalool) thin film thickness increases linearly with time and the rate of deposition was approximately 40 nm/min. With increase in input RF power, the rate of deposition decreased, which can be attributed to the higher degree of fragmentation of the monomer molecule. Therefore, films fabricated at higher input RF power were characterised by higher density and increased degree of cross-linking. Furthermore, the process of ablation may be more prominent in the case of high

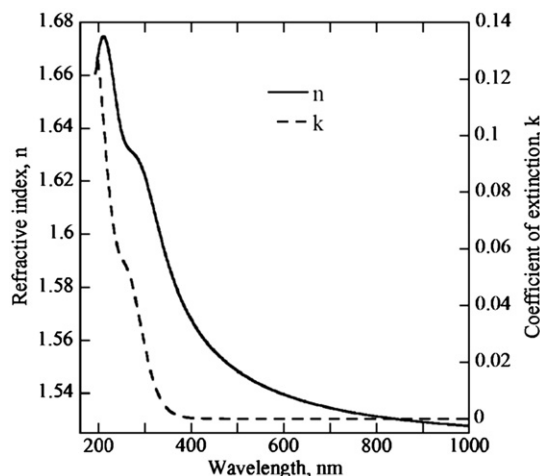


Fig. 1. Refractive index and extinction coefficient of poly(linalool) thin films deposited at 25 W.

power conditions, etching back the newly formed thin layer [15]. These results are in agreement with previously reported studies for similar compounds [21,22,29]. It is therefore possible to fabricate thin films of desired thickness to suit given application by controlling such deposition conditions as time and applied RF power.

### 3.2. Optical properties

Spectroscopic ellipsometry was used to measure the optical absorption of poly(linalool) thin films fabricated at different RF powers and estimate the refractive index, extinction coefficient and optical band gap. Fig. 1 shows the typical refractive index and extinction coefficient profiles for poly(linalool) thin film. The refractive index of poly(linalool) was slightly greater than that of the glass substrate, at 1.55 at 500 nm. Above 400 nm, the extinction coefficient was negligible, confirming that poly(linalool) is optically transparent. For films fabricated at 25 W under varied time, a deviation in measured optical parameters occurred over the lower wavelength region, with both indices increasing with thickness. However, the increase was insignificant. Similarly, there was no significant difference between optical profiles for the samples deposited at different RF power levels.

The effect of applied deposition power on the optical band gap  $E_o$  of poly(linalool) was investigated using UV–vis spectroscopy data. Thin films were manufactured for 10 min and their absorption profile data were obtained. Increase in the input RF power level had no significant effect on the general shape of the absorbance profiles, with all curves displaying a single peak at approximately 310 nm. The variance in height of the peak was attributed to the differences in film thickness.

The optical band gap  $E_o$  values are obtained from the UV–vis data using the Tauc relation and the results of films deposited at seven different power levels are summarised in Table 1. It was found that  $p = 1/2$ , which is commonly assumed for semiconducting plasma

Table 1

The influence of RF power on the refractive index and optical band gap of poly(linalool) thin films.

RF power level (W)	Thickness (nm)	Refractive index (at 500 nm)	Optical band gap (eV)
15	313.7	1.56	2.81
25	289.0	1.55	2.82
35	245.3	1.57	2.85
50	243.7	1.57	2.74
75	197.4	1.55	2.64

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