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Original

Estimation of the elastic modulus of Au nanofilms by subtracting the polymeric substrate influence

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Abstract: The elastic modulus of Au nanofilms deposited onto a polymeric substrate was estimated by subtracting the substrate mechanical influence from the Au/polymer system under axial tensile test. To select the most appropriate substrate between polyethylene-terephthalate (PET) and Kapton (polyimide), the crystallinity and structural isotropic conditions were previously examined by X-ray diffraction. Kapton shows a lower % of crystallinity and enhanced isotropic conditions than PET. The elastic modulus of 60 nm-thick Au films deposited onto Kapton was measured, resulting in an average value of 37 GPa, which is lower than the Au bulk value. Crystalline structure and elastic properties of polymeric substrates are found to be of great relevance to be considered for the determination of the mechanical properties of metallic films.

Keywords: **polymeric substrates, gold nanofilms, PET, Kapton, elastic modulus.**

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1. Introduction

The synergistic properties of metal/polymer systems make them ideal candidates to be incorporated as core elements in different technological fields. Among the most relevant features of this type of material, the excellent combination of mechanical robustness, electrical conductivity, and thermal stability is a desired property, making them suitable for a wide range of applications, from energy storage to advanced flexible electronics (Tayyab et al., 2025). For example, Yoon et al. (2019) developed a flexible electrochemical enzyme (glucose) biosensor based on a gold nanofilm deposited by sputtering onto a flexible polymer electrode (commercial Kapton polyimide film), evidencing the feasibility of flexible sensing platforms for wearable biosensing systems. Huang et al. (2015) demonstrated the possibility of depositing Au nanofilms ranging from 14 to 180 nm onto an HCl-doped polyaniline substrate. The authors evidence the dependence of the Au nanofilms' electrical behavior on their thickness and temperature. A novel spectroscopy approach called surface-enhanced Raman scattering (SERS) has been used to deeply analyze the chemical and biological properties derived from the material's structural information. For such a technique, Au nanofilms deposited onto polymer substrates are ideal candidates to work with, as can be evidenced elsewhere (Kayaci et al., 2023; Yan et al., 2019). The use of Au thin films on flexible substrates (e.g., polymers) is currently being explored and shows great promise for several applications, necessitating deeper investigations into the effective properties of such systems.

To measure the mechanical properties of metallic thin films, a substrate is required as a supporting element. Glass is one of the most common substrates used for evaluating the properties of metallic thin films due to its high optical transparency, good stability, and amorphous structure. However, fragility, heavy weight, and lack of flexibility are important drawbacks for further implementation and usage. The trends and technological developments in electronic devices require flexible substrates as supporting elements for active films, offering versatility, weight reduction, and increased adaptability. The use of specific polymers as substrates offers various advantages, including flexibility, optical transparency, low weight, easy availability, and high chemical stability, among others. One of the most important conditions required on such substrates is to affect the nanofilms' intrinsic properties minimally, which becomes very important when such properties are investigated and measured. When this condition is neglected, film properties might be masked and/or wrongly estimated by the undesired influence of the substrate.

The use of polymeric substrates is increasing for different technological applications. For example, organic semiconductor materials for electronic and optoelectronic applications require flexible substrates during their implementation (Gao et al., 2020; Promsawat et al., 2019; Kia et al., 2019). Al-doped zinc oxide was deposited on some polyimides treated with plasma for obtaining a large area flexible transparent thermoelectric device with adequate electrical resistance stability (Marques et al., 2019). For studying cell adhesion, a charged polymeric substrate is required to modify the surface (Hoshiba et al., 2018) or to support a bacterial surface to facilitate metal organic growth (Zhang et al., 2018), among other examples. A new triboelectric nanogenerator was proposed for producing current through a contact between a polyimide (Kapton) layer and an Al surface (Cheedarala & Song, 2020). Semiconductor films can be deposited, for instance, by the chemical bath technique onto polymeric substrates for flexible device applications with high transparency (Moreno et al., 2016). This method takes advantage of the low temperature required, which does not affect the polymeric substrate. The surface characteristics of polymeric substrates, such as roughness, surface energy, hydrophobicity, and surface charge, can influence the adhesion or interaction with other layers deposited onto them (Yuan et al., 2017). The mechanical properties of flexible substrates play an important role in different applications, e.g., semiconductors are deposited for flexible electronics applications as supporting elements for high-conductivity layers for printing devices (An et al., 2020; Bhuiyan et al., 2019) and for depositing carbon nanotubes onto a polymer for increasing the electrical conductivity (Parel et al., 2018).

Acetate is a transparent polyethylene terephthalate (PET) polymer commonly used, for example, as a printing material due to the high melting point (260°C), durability, low cost, high chemical inertness, light weight, high mechanical strength, and as waste material for concrete production (Şimşek et al., 2019; Mohammed et al., 2017). The nano-roughness and hydrophilicity of the PET surface produced by carbon nanotubes improve the adhesion strength (Theogene et al., 2020) for obtaining electronic devices and for biomedical applications (Liao et al., 2019). Another popular polymer for several applications in engineering is Kapton, a commercial polyimide, which can operate at temperatures up to 400°C before exhibiting relevant structural changes (Kapton, 2025; Khomiakova et al., 2020). Kapton is commonly used for industrial applications where extremes of heat and vibrations exist, given its very convenient mechanical properties.

In this work, structural properties of acetate (PET, Hewlett Packard) and Kapton® 500HN (polyimide) were examined to determine the most appropriate substrate for thermally deposited Au nanofilms. The mechanical properties of such nanofilms were estimated by subtracting the influence of the substrate during an axial tensile test. The elastic moduli of Au nanofilms were measured and compared with other reported values.

2. Materials and methods

Commercial transparent PET (acetate) and Kapton 500HN (DuPont) films were used as polymeric substrates with lateral dimensions of 8 mm x 40 mm. The thicknesses of the PET and Kapton sheets were 0.100 mm and 0.127 mm, respectively. Axial tensile tests were carried out within the elastic regime on the polymeric substrates with a universal testing machine specially designed and fabricated for thin films testing (Huerta et al., 2010). The force was measured with a commercial load cell of 200 N, while the elongation was measured with a digital micrometer. For the structural characterization of the substrates, X-ray diffraction analysis was done in a Bruker D8 Advance diffractometer with a Cu source of 0.15408 nm, 2θ range of 20° to 60° with steps of 0.02°. Prior to the deposition of the Au nanofilms, polymeric specimens were cleaned with soap, isopropyl alcohol, distilled water, and dried with air. Four 60 nm-thick Au films were deposited into a vacuum chamber by thermal evaporation on the PET and Kapton substrates using a deposition rate of 0.03 nm/s. Au film thickness was measured and controlled with a quartz crystal balance and an Inficom controller. To determine the elastic modulus of the Au films, the force-strain (F-ε) curve of the Au/substrate system was measured. Subsequently, the F-ε curve of the polymer alone (previously registered) was subtracted to obtain the final mechanical response of the gold nanofilm. The stress-strain (σ-ε) curve was obtained from the F-ε curve by incorporating the cross-sectional area A of the nanofilm, and the elastic modulus E of the nanofilm was estimated using the slope ($E=\sigma/\varepsilon$) of the linear section of the σ-ε plot. Such an approach for the estimation of metallic thin film elastic modulus has been previously reported for other material systems (Faupel et al., 1989; Ho & Faupel, 1988; Macionczyk & Brückner, 1999).

3. Results and discussion

3.1. Crystalline structure of the polymeric substrates

The X-ray diffraction technique was used to analyze the crystalline structure of the polymeric substrates, and crystallinity was estimated. The crystallinity is defined as the fractional amount (mass or volume) of polymer that is crystalline. It is an important parameter that determines some mechanical properties. Figure 1 shows the diffractograms of the cleaned sheets of PET (Fig. 1a) and Kapton (Fig. 1b) used as substrates, highlighting important differences between them.

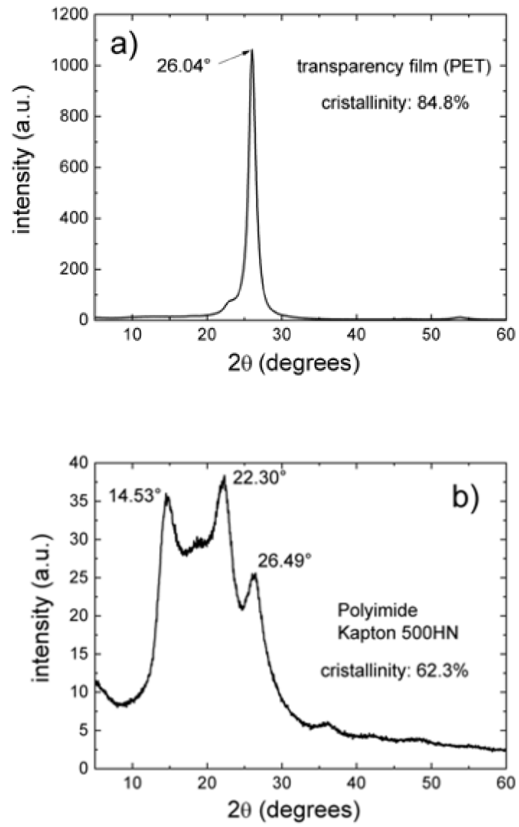

Figure 1. X-ray diffractograms for: a) PET, and b) Kapton 500HN substrates.

PET shows a main diffraction peak at 2Θ=26.04° with high intensity and 84.8% crystallinity, while Kapton exhibits three wider diffraction peaks with reduced intensity and 62.3% crystallinity. The measured crystalline structure of PET is similar to the results reported by Gupta et al. (2013) and Fawzy et al. (2018). The authors reported that the main peak is correlated with the extrusion process used during the polymer fabrication.

Differences in the crystalline structure of the substrates are expected to play a key role in the mechanical properties of the films deposited onto them, as will be discussed later. Some authors have reported that the mechanical strength and crystalline structure of PET improve after heating and tensile stress processes, which increase the stretch ratio (Onyishi & Oluah, 2020). Also, coatings of SiO_x with wrinkles at the nanometric size are used on Kapton substrates to obtain surfaces resistant to moisture and temperature, reducing stress and cracking of the coatings (Zhang et al., 2021).

3.2. Mechanical properties of the polymeric substrates

3.2.1. Elastic modulus of PET and Kapton

Figure 2 shows representative plots of the stress vs strain curves (σ-ε) obtained for the PET and Kapton polymers for axial strain up to 3%. A linear behavior of the σ-ε plot is evident for both substrates up to ε=1% and non-linear behavior (permanent deformation) is observed thereafter. Thus, ε=1% will be considered as the maximum strain for the Au/polymer system, which is within the polymer elastic zone under mechanical deformation. The selected specimen elongation rate (0.03 mm/s) and the length/width ratio (≈3-10) are in agreement with the requirements indicated in the ASTM D882 standard for plastic films (ASTM, 2018). The elastic moduli (E) of both polymeric substrates were determined by means of the slope of the σ-ε plots. The average and standard deviation (SD) E values were E_{PET} = 4.74 ± 0.06 GPa and E_{Kapton}= 2.34 ± 0.01 GPa. These values are in accordance with previous reported values (Callister, 1994; Dharmadasa et al., 2020).

3.2.2. Effect of the crystalline orientation of the polymer

In order to evaluate if there is any effect of the semi-crystalline structure of the PET and Kapton specimens on their E value, mechanical tensile tests were carried out on substrates cut at 0° and 90° respect to the vertical axis of the commercial original sheets (see Figure 3).

The σ-ε plots for these specific directions are shown in Figure 4. Important variations of E can be seen for PET (main diffraction peak, see Figure 2a) when the tensile direction (i.e., specimen length direction) is changed (Figure 4a). In contrast, very similar E values are observed for Kapton (Figure 4b), evidencing no dependence on the tensile direction, which agrees with its polycrystalline structure (see Figure 1b). Thus, it is observed that the crystalline structure of polymers influences their mechanical properties, with anisotropic conditions observed for PET and nearly isotropic conditions for Kapton.

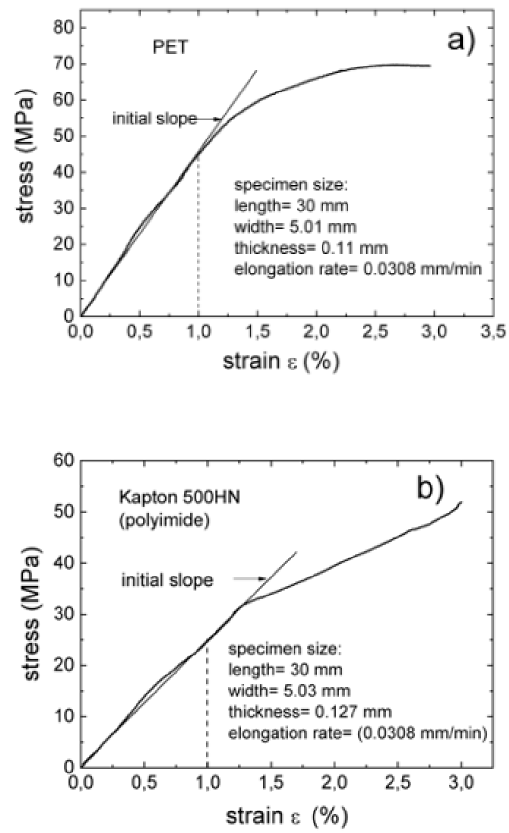

Figure 2. Representative σ-ε plots for a) PET and b) Kapton substrates.

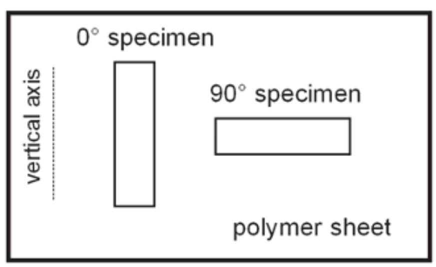

Figure 3. Polymeric specimens cut in different direction from the commercial polymer sheet (PET and Kapton).

3.3. Estimation of the elastic modulus of Au nanofilms

Once a reduced anisotropic mechanical behavior was observed for Kapton, this substrate was selected for the thermal evaporation of four Au nanofilms with a thickness of 60 nm. Each Kapton substrate used for depositing Au films was previously mechanically characterized in order to properly extract the specific properties of the deposited nanofilms when subtracting the substrate influence. After the nanofilm deposition, the Au/Kapton system was mounted on the universal testing machine, and the force-strain (F-ε) plot was obtained. By subtracting the F-ε data of the Kapton substrate from the gold/Kapton system, the elastic modulus of the Au film is obtained. The following relation is used to determine the strength σ (ε) film property,

$$\sigma(\varepsilon) = \left(\frac{1}{h_f w}\right)[F_{tot}(\varepsilon) - F_{sub}(\varepsilon)] \quad (1)$$

where h_f and w are the length and width of the gold film, F_{tot} is the applied force on the Au/Kapton system, and F_{sub} is the force applied on only the Kapton (substrate) test. This methodology has been reported for characterizing different metals with nanometric thickness (Faupel et al., 1989; Ho & Faupel, 1988; Macionczyk & Brückner, 1999; Huerta et al., 2012). It is important to mention that Eq. (1) stands for materials with identical Poisson's ratios and assuming that the influence of the materials' interface is negligible. However, based on the reported materials and the applied deformation ranges, the measured error is around 4%, which is an acceptable tolerance (Macionczyk & Brückner, 1999). Using this approach, Faupel's group (Faupel et al., 1989; Ho & Faupel, 1988) evaluated the adhesion and deformation of Cu/polyimide and Cr/polyimide structures, where electron-beam evaporation was used as metal deposition technique. Also, Macionczyk and Brückner (Macionczyk & Brückner, 1999) reported the estimation of mechanical properties of AlCu thin films deposited by magnetron sputtering by subtracting the substrate influence (polyimide foil) from the stress-strain plot of the AlCu/substrate system.

Figure 5 shows the F-ε plot and the corresponding σ-ε plot, by using Eq. (1), of a 60 nm-thick Au film. Figure 5a shows, in a semi-log F-ε plot the corresponding curves of the Au/Kapton system (higher curve), the Kapton, and the gold film for 60 nm-thick (lower curve). The two upper curves show a slight difference, while the Au film curve was obtained by subtraction according to Eq. (1). Inset in Figure 5a shows a detail of the F-ε plot where the highest curve corresponds to the Au/Kapton system. The curve obtained for only the gold film is represented in Figure 5b through the σ-ε plot derived from data of Figure 5a. In this plot, the E value (slope of the curve) of the analyzed Au nanofilm is estimated as 38.8 GPa. The average E value for the Au thin films was 36 ± 6 GPa, being a lower value than the bulk value reported for Au (74- 80 GPa) (Salvadori et al., 2003; Kracke & Damaschke, 2000; Noel, 2016), but in concordance with previous values reported for Au nanofilms (Salvadori et al., 2003; Kracke & Damaschke, 2000; Noel, 2016; Birleanu et al., 2026; Baek et al., 2005; Gan & Chen, 2009).

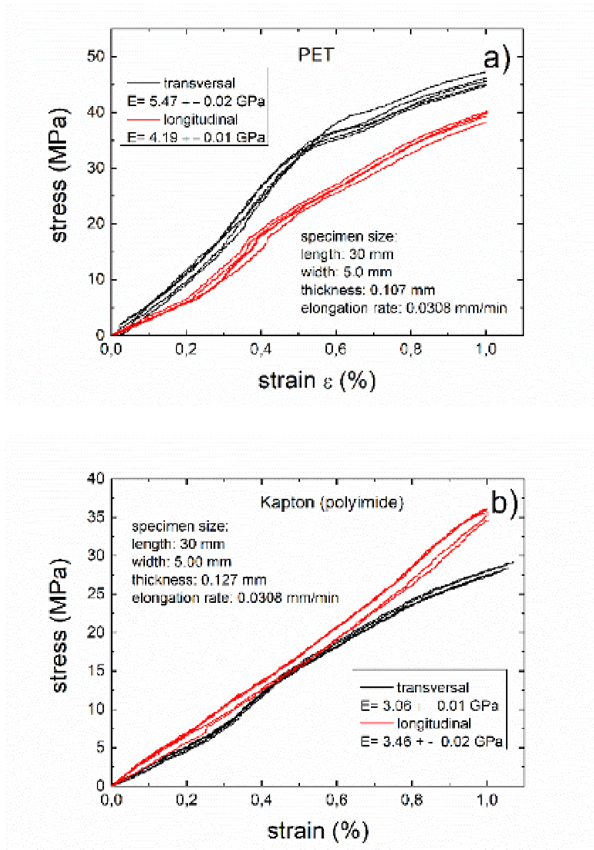

Figure 4. σ-ε plots for determining the E value for: a) PET, and b) Kapton. Specimens were cut at 0° and 90° respect a vertical axis of the commercial sheet.

Birleanu et al. (2016) reported a value of E= 58 GPa for gold films of 100 nm deposited on Si substrates by electron beam evaporation. Baek et al. (2005) reported E values ranging from 26 to 36 GPa for Au thin films deposited onto a silicon substrate of thicknesses between 200 nm and 1000 nm using bending tests. By using a resonance frequency approach, Salvadori et al. (2003) reported an average value of 69.1 GPa for Au films ranging from 19

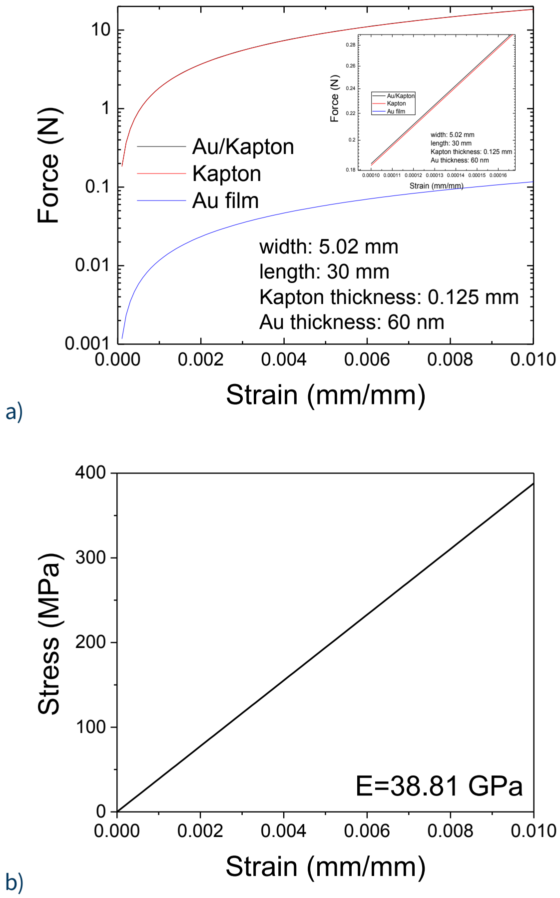

Figure 5. Mechanical characterization of the Au/Kapton system. a) F-strain plot for the Au/Kapton system; b) stress-strain plot of only the Au nanofilm and E estimation.

nm to 61 nm. Kracke & Damaschke (2000) reported an average E modulus of 50 ± 16 GPa for gold islands 50 nm thick, measured using scanning force microscopy under ultrahigh vacuum. In a comprehensive review of Au properties, Noel (2016) reported values ranging from 30 GPa to 80 GPa for Au thin films ranging from several nm to few microns, from different authors and measurement techniques, highlighting the great relevance of developing novel and confident strategies to measure the mechanical properties of metallic thin films properly. Theoretical calculations by using molecular dynamics (Gan & Chen, 2009) refer to E = 40 GPa for a gold thickness of 36 times the lattice parameter a_o (≈15 nm-thick). From the presented reports, it can be observed that the value here estimated for 60 nm thick Au films (36 ± 6 GPa) is in good agreement with most of the studies, providing then an accurate strategy for the metallic thin film E measurement by extracting the mechanical contribution of the substrate.

Understanding the properties of polymers as substrates is crucial as a preliminary step in determining the properties of metallic films. Polymeric substrates may influence the mechanical properties of the grown metallic films (Noel, 2016). The presented results highlight the potential influence of a flexible substrate on the effective properties of metallic thin films and need to be considered for the corresponding applications. Also, the accuracy of the measurement of the elastic modulus of Au nanofilms by subtracting the substrate influence on the mechanical response represents a relevant strategy to easily explore the mechanical properties of such metallic films, representing an important contribution to the technological development of these nanomaterial systems.

Three features are suggested to be taken into consideration before selecting specific polymers as substrates: the crystalline structure, the quality of the surface, and the thermal properties. The crystalline orientation of the polymer is important; it is known that most of the polymers exhibit semi-crystalline structural properties. The tensile or compressive tests to obtain mechanical properties of the nanofilms can vary depending on the orientation of the mechanical deformation and the crystalline structure of the supporting substrate. Surface quality is a key parameter for achieving good adhesion, adequate film roughness, and good chemical compatibility when the surface is treated. Finally, the high thermal properties of the polymer are also of paramount importance for the high melting temperatures of the nanofilms and their environmental and chemical stability. Also, good thermal conditions increase the possibility of using thermal treatments after deposition for improving the films' properties. According to these considerations, Kapton is a convenient substrate due to its isotropic crystalline structure, high surface quality, high melting temperature, and chemical stability.

Conclusions

Crystalline and mechanical properties of flexible substrates, Kapton and PET (acetate), were evaluated to be then used as substrates for thermally evaporated Au films with nano-thickness. Isotropic structural and mechanical properties of the polymer were prioritized for further consideration as the substrate of the Au nanofilms. Kapton results in a more suitable substrate due to its more isotropic properties and higher thermal stability. By subtracting the influence of the polymeric substrate on the Au/Kapton system, the elastic modulus value of Au films of 60 nm-thick was 36 ± 6 GPa, similar to the values reported in the literature for gold nanofilms. The obtained results highlight the importance of previously knowing the properties of the polymeric substrates to eliminate any significant influence they may have on the metallic films' properties.

Conflict of interest

The authors do not have any type of conflict of interest to declare.

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