EXPERIMENTAL EXPLORATION OF GOLD SPUTTERED LAYER BEHAVIOUR AT MICROWAVE FREQUENCY

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Abstract *The electrical characteristics of an ultra thin gold layer deposited by sputtering on polyethylenterephtalate foil have been examined experimentally at microwave frequencies. Dramatic changes with sputtering time were observed. It turned out that the highest power absorption requirement and the optimal sheet resistance for heating do not coincide, so a balanced compromise needs to be reached. Exploration of ultra thin films by microwaves is an efficient and inexpensive diagnostic method.*

INTRODUCTION

Applications of thin/thick metal layers span from industrial exploitation through mass production of electronic devices to scientific experiments in conjunction with nanotechnology. In dependence on the application, metallic continuous/discontinuous layers of various thicknesses and various measures of homogeneity are needed. An attempt to characterize thin gold layers on polyethyleneterephtalate from these points of view [1] form the background of this work. The need to prepare conductive filaments in polymeric film and to produce active food packaging with effective heat production resulted in a series of measurements of metallic layer electric parameters at microwave frequency. In this paper, we focus on experimental exploration of the behaviour of a gold layer observed at microwave frequency. It turns out that the quality of the layer changes dramatically within a narrow time interval of sputtering. This critical point must be considered when producing a layer with a specific surface resistance.

EXPERIMENTAL BACKGROUND

Gold layers were produced by sputtering of gold on a polyethyleneterephtalate (PET) substrate 50 μm in thickness [1]. The electric parameters of the layer were determined by its reflection and transmission coefficients, and the impedance measured in the waveguide. The measuring set up is shown in Fig. 1. The frequency was calculated from a series of nodes of the standing wave produced by a short in the reference plane (RP). The power meter, replacing the short in the RP, provides the power incident on the sample under test. When the PET foil with the sputtered gold layer was placed in the RP, the transmitted power was recorded by a power meter located on the rear side of the sample totally filling the cross-section of the waveguide. The input voltage standing wave ratio (VSWR) was measured by the calibrated attenuator when the matched load placed behind the sample terminated the waveguide. The impedance of the sample was determined by the VSWR and by the shift of the standing wave node from the RP read at the measuring line [2]. Subsequently the power absorbed in the layer and the surface resistance were calculated. The patterns of the $(\mathrm{VSWR})^{-1}$ and input reflection coefficient S_{11} at 8.2 GHz are plotted in Fig. 2. Their dependence on the sputtering time t is

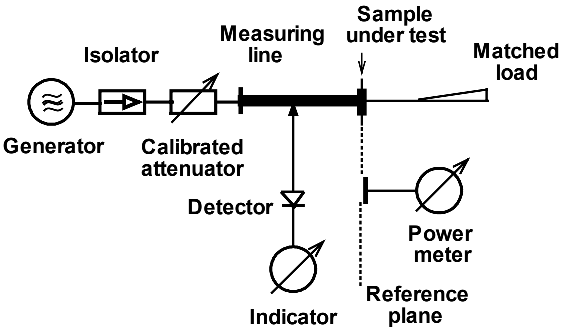

Fig. 1. Measuring set up.

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$$
\begin{aligned}
(\mathrm{VSWR})^{-1}(t) = {} & \frac{0.875}{1+\left(\frac{t}{35}\right)^{35}} - 0.069\,\mathrm{atan}\left(\frac{t+40}{170}\right) + 0.014\,e^{-0.001|t-28.01|^{2.1}} + 0.04\,e^{-2.9\cdot 10^{-3}|t-50.5|^{2.3}} \\
& + 0.48\,e^{-\left(5.377\cdot 10^{-4}|t-11.73|^{2.42} + 1.18\cdot 10^{-4}|t-43.06|^{5.1}\right)} + 0.008\,e^{-1.705\cdot 10^{-3}|t-49.68|^{5.998}} + 0.1084
\end{aligned}
\tag{1}
$$

$$
\begin{aligned}
S_{11}(t) = {} & \frac{0.8499}{1+\left(\frac{35.67}{t}\right)^{35}} + 0.069\,\mathrm{atan}\left(\frac{t-38.01}{193}\right) - 0.013\,e^{-0.001|t-28.01|^{2.1}} - 0.081\,e^{-1.8\cdot 10^{-3}|t-49|^{2.5}} \\
& - 0.9\,e^{-\left(5.377\cdot 10^{-4}|t-11.73|^{2.42} + 1.18\cdot 10^{-4}|t-43.06|^{5.1}\right)} - 0.012\,e^{-1.705\cdot 10^{-3}|t-49.68|^{5.998}} + 0.033
\end{aligned}
\tag{2}
$$

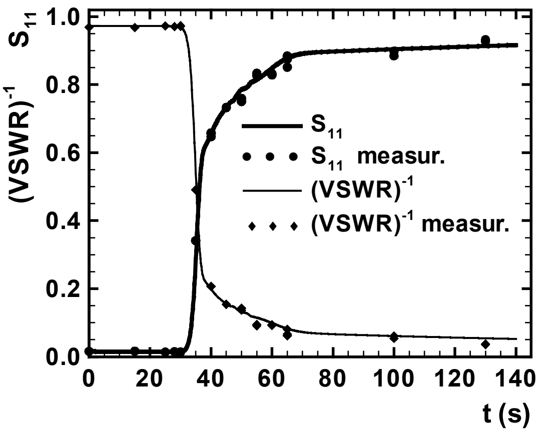

Fig. 2 Dependence of $(\mathrm{VSWR})^{-1}$ and the input reflection coefficient S_{11} on the sputtering time at 8.2 GHz, measured and curve fitted.

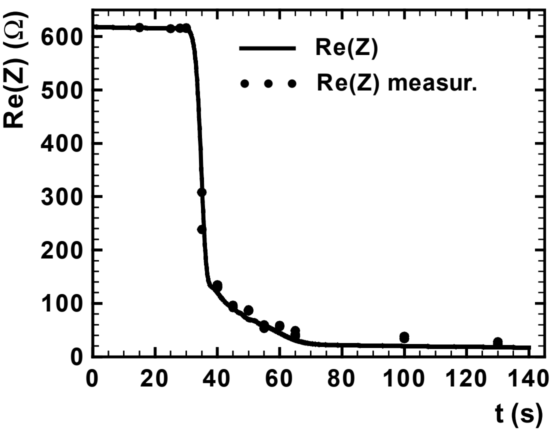

Fig. 3 The measured and curve fitted dependence of the real component of the layer impedance ReZ on the sputtering time at 8.2 GHz.

$(\mathrm{VSWR})^{-1}$ and S_{11} are practically the same at 8.2 and 12.6 GHz and clearly indicate continuity/discontinuity of the gold layer. When the sputtering time is less than 30 sec the layer is discontinuous and the metal particles are isolated from each other. They get closer and closer and create an almost continuous layer when the deposition time is between 30 and 40 sec. Above this interval the layer slowly and fluently becomes homogeneous with increasing thickness. Over 130 sec, a perfect conductive gold layer is formed.

The measured surface impedance Z of the layer has a small capacity component, not exceeding 4 % of the real part of the impedance when the layer is discontinuous, which vanishes when the depositions are longer than 35 sec. Thereafter the impedance is real. The measured real component of the surface impedance ReZ at 8.2 GHz is shown in Fig. 3. Its time dependence is described by (3) and is almost the same also for 12. GHz

$$
\begin{aligned}
\mathrm{Re}\,Z(t) = {} & \frac{588}{1+\left(\frac{t}{35}\right)^{35}} - 22\,\mathrm{atan}\left(\frac{t-40}{170}\right) + 0.014\,e^{-0.001|t-28.01|^{2.1}} + 40\,e^{-0.0029|t-49.5|^{2.3}} \\
& + 390\,e^{-\left(5.377\cdot 10^{-4}|t-11.73|^{2.42} + 1.18\cdot 10^{-4}|t-43.06|^{5.1}\right)} + 4.5\,e^{-1.705\cdot 10^{-3}|t-50|^{5.998}} + 35\ .
\end{aligned}
\tag{3}
$$

The sheet resistance R_{sq} given in Ω/sq of the gold layer can be determined by the measured ReZ and the characteristic impedance Z_0 of the waveguide with the TE_{10} mode as

$$
R_{sq} = \frac{Z_0\,\mathrm{Re}\,Z}{Z_0 - \mathrm{Re}\,Z}\cdot\frac{a}{b}
\tag{4}
$$

where a>b are cross-sectional dimensions of the waveguide. R_{sq} at 8.2 GHz is shown in Fig. 4 and its curve fit is

$$R_{sq}(t)=\frac{81200}{1+\left(\frac{t}{31.81}\right)^{55}}+100\,e^{-0.0015|t-48|^{2.1}}+400\,e^{-0.0029|t-33|^{2.3}}-90\,e^{-0.0049|t-39|^{2.3}}+55\,. \tag{5}$$

The sheet resistance of an active food packaging foil for microwave warming should be 188.5 Ω/sq [3]. Our layers exhibit such a value at deposition time about 50 sec. Reproducibility of sputtering greatly influences the resultant surface impedance and consequently also the sheet resistance.

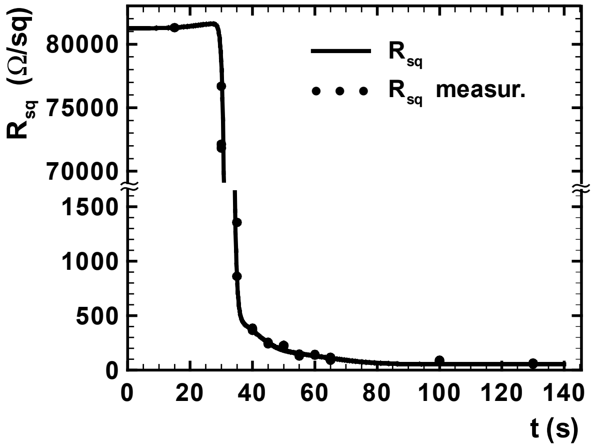

Fig. 4 The sheet resistance determined at 8.2 GHz and its curve fit.

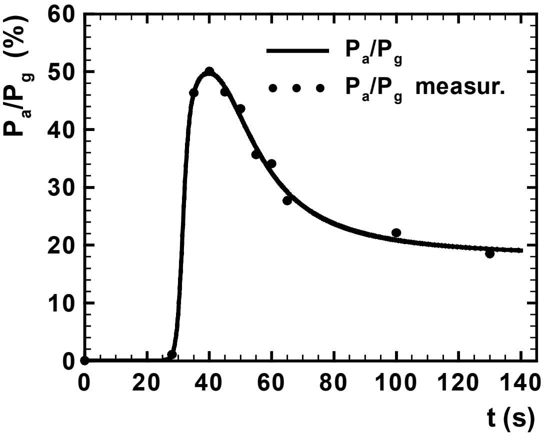

Fig. 5 The time dependence of the measured and curve fitted ratio of P_a/P_g at 12.6 GHz.

It was shown in [4] that thin metallic film may absorb up to 50 % of the incident power even if its thickness h is much less than the skin depth δ. The ratio of the power absorbed in layer P_a and the power incident on it from generator P_g is a symmetrical bell-shaped curve depending on h [4]. The quality of the metallic film was not specified, but it can probably be assumed to be continuous and homogeneous. In our case for deposition time 50 sec the mean layer thickness h=8.4 nm as follows from (7). For the required R_{sq}=188.5 Ω/sq the layer conductivity $\sigma=1/R_{sq}h=6.3166.10^4$ S/m and δ=2.2 μm. Consequently, the above condition is met. From our measurement it follows that the sputtering time dependence of the P_a/P_g is asymmetric due to the morphology of the layer formed in the sputtering process. Fig. 5 confirms the highest absorbance of the incident power in the layer when the deposition time is from 35 to 45 sec. The curve fit of this dependence is

$$P_a\,/\,P_g(t)=\frac{8.914\,t^2-683.7\,t+2.14\cdot 10^4}{t^2-78.4\,t+1869}\cdot\left(\frac{1.994}{1+\left(\frac{31.39}{t}\right)^{31.46}}+0.00068\right)\,. \tag{6}$$

An Atomic Force Microscope (AFM) examination provided additional information about the quality of the layer. First the equivalent or mean thickness of the gold layers was obtained by ultra violet–visible spectroscopy (UV-VIS) and from atomic absorption spectroscopy (AAS) analysis [1]. The results are reproduced in Fig. 6. For practical purpose the following linear function is acceptable

$$h(t)=0.16768\,t \tag{7}$$

where h(t) is in nm and t is in sec.

The gold sputtered surface was scanned by the AFM in a square 1x1 μm. Then the distribution F(h) of measured altitudes at 40000 points was calculated. From F(h) we changed to the probability density f(h)

$$f(h)=\frac{F(h)}{\Delta w\sum_i F(h_i)} \tag{8}$$

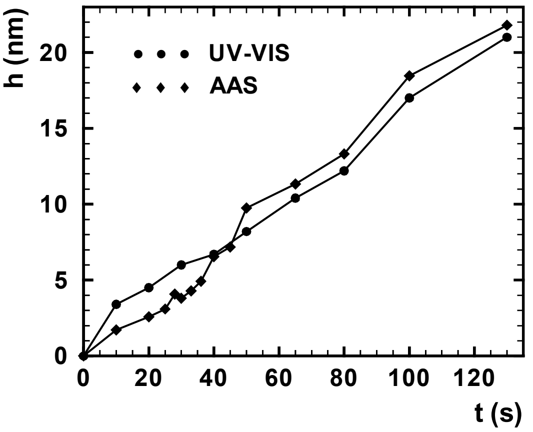

Fig. 6 The dependence of the gold layer thickness determined from UV-VIS spectra and measured by the AAS method [1].

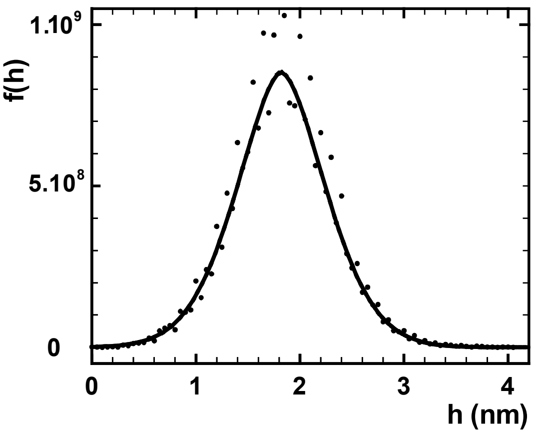

Fig. 7 Probability density and its approximation for t=60 sec resulted from a 1x1 μm square area of the sputtered gold layer.

where Δw is the altitude bandwidth, constant for all discrete altitudes h_i. Finally f(h) is fitted by the error function, e. g., for deposition time t=60 sec

$$f(h) = 0.8527\, e^{-2.3362|h-1.821|^{1.7102}} \quad . \tag{9}$$

If f(h) is shifted to higher h by the difference of the mean value of the layer thickness h_m known from (7) and the mean value of F(h) we can calculate the probability of altitudes of the surface profile. Measurement of copper layers led to similar qualitative behaviour to that of gold, unlike silver, which showed totally different sputtering time patterns. Platinum layers are at present under test.

CONCLUSION

The electrical parameters of ultra thin film have been investigated experimentally. They are strongly influenced by the sputtering time. The transition from a discontinuous to a continuous layer is very fast with direct consequences on the layer characteristics. For the sputtering process carried out in our laboratory [1] the $(VSWR)^{-1}$, input reflection coefficient, sheet resistance and power absorbed in the layer depending on the deposition time have been described by closed form formulae obtained by curve fitting techniques. Our measurement confirmed absorption of maximum 50 % of the incident power predicted in [3,4] but its time dependence is not symmetric due to the real morphology of the layer. The greatest power absorbance does not coincide in time with the required value of the sheet resistance suitable for the optimum production of heat in active food packaging foil. A compromise therefore has to balance these two demands.

Examination of thin metallic layers by microwaves is a suitable and effective method easy performed with equipment that is not expensive. The probability of the layer surface height can be determined by outputs from the AFM scanner. Examples of all discussed quantities have been provided, with emphasis on the strong and rapid change in the real layer characteristics.

ACKNOWLEDGEMENT

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