An Investigation of the Relationship between Resistance and Thickness of Deposited Nickel Thin Film Resistors

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Abstract: The main purpose of this study is finding the relationship between resistance and thickness of deposited Nickel Thin Film Resistors. It was found that the Sheet Resistance, R_s, is inversely proportional to the thickness of the film on the substrate. It was also observed that when the film thickness is greater than 50 nm, films behave like ordinary resistors. In other words in bulk, films obey Ohm's law if other physical quantities remain constant.

Keywords: Energy dispersive, thin film resistors, X-ray fluorescence analysis

INTRODUCTION

Thin films have so many applications in industries. These include video tapes, audio tapes, glass coating (that cuts off infrared radiation which causes heating effect), camera films extra. Recently thin film deposition has been applied in the manufacture of solar cells (Katumba, 1994).

The main objective of the study was to determine the relationship between resistance and thickness of deposited nickel thin film resistors. It also aimed at determining the value of thickness in nm beyond which films should be considered to be in bulk and hence start obeying Ohm's law. Nickel thin film resistors were deposited on subtrates by evaporation technique (Katumba, 1994).

In this investigation, the characteristic X-rays from the sample together with the scattered primary radiation are utilized for estimating film thickness using Fundamental Parameter Techniques (FPT) which employs the basic relationship between measured fluorescent intensities and the elemental concentration (Kinyua, 1982; Gianque and Jaklevic, 2008).

In the following sections quantitative x-rays fluorescent elemental analysis technique of film samples using FPT will be presented. Basic equations will be stated and all necessary steps in the analytical approach will be discussed.
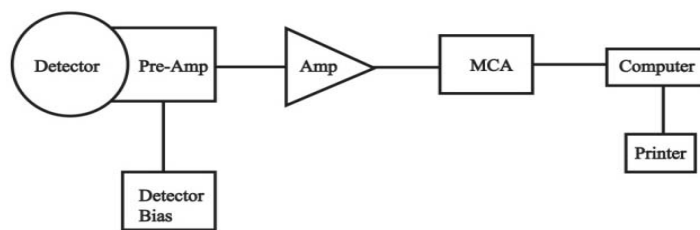
MEASUREMENTS

Instrumentation: A schematic diagram for an Energy Dispersive X-Ray Fluoresce Analysis (EDXRFA) set up is as shown in Fig. 1. It consists of an X-ray source, S and X-ray spectrometer. The spectrometer is made up of:

- Solid state detector Si (Li) Canberra Series 7300
- A Pre-Amplifier Canberra Model 1008
- An Amplifier
- Coolant-Liquid Nitrogen in a Dewar Flask
- ADC Canberra Model 8075
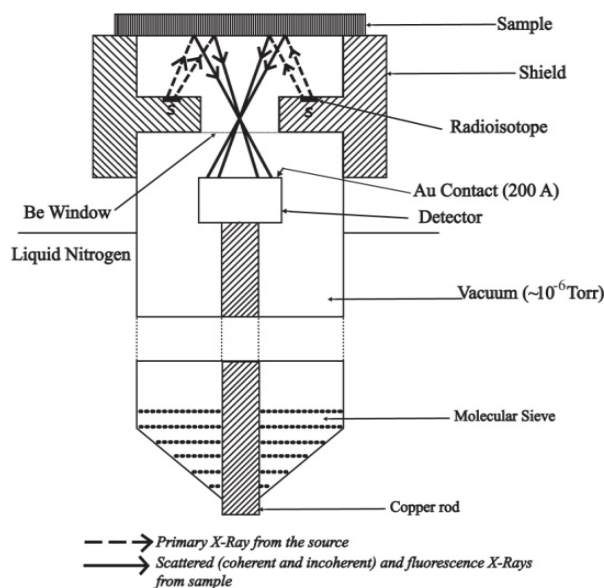- A Canberra S-100 PC based MCA card used for data acquisition

The detector is a diode made of silicon crystal. The diode is reverse biased and electron-hole pairs are produced by x-rays irradiation on the crystal. These electron-hole pairs produced by irradiation are carried away from the crystal by applied electric field. The vacuum under which the crystal is housed is provided by Berrylium (Be) window.

In order to minimize noise due to thermal excitation, the Si (Li) detector is maintained at Liquid Nitrogen temperature. The pre-amplifier converts the burst of electrons resulting from the absorption of X-rays, into voltage signal. A Field Effect Transistor (FET) coupled to a pre-amplifier reduces or minimizes noise which may degrade the resolution of the spectrum. The matching of the noise to the signal ratio is achieved by the high impedance output of the detector and low impedance of the coaxial cable to the pre-amplifier input resistance.

The pre-amplifier is followed by shaping amplifier whose polarity is set to positive so that it can accept positive pulses from the pre-amplifier. The pulses are thereby shaped and amplified at the same time. The pulses are linearly amplified to a precise pulse-height. The output of these unipolar pulses are fed into ADC whereby they are converted into a number of signals proportional to the pulse amplitude voltages and are stored in the address memory location referred to as channel number. Canberra S-100PC based MCA was used for data acquisition and storage.

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(a) Electronic set-up

(b) Cryostat

Fig. 1: Schematic diagram for an Energy Dispersive X-Ray Fluoresce Analysis (EDXRFA)

Spectrum analysis was done using the software AXIL (Analysis of X-ray spectra by Iterative Least squares fitting) which is written in fortran and based on a non-linear least squares fitting procedure to optimize the parameters of fitting model (Energy and resolution calibration, peak intensity and background parameters) for each spectrum. The Quantitative X-ray Analysis Software (QXAS) package was used to determine the thickness of the film for every element deposited on the glass-surface (Sparks Jr, 2002).

Method and analysis: When X-rays pass through matter, they are both absorbed and scattered. Scattering is small or very small compared to absorption except for elements of low atomic number at shorter wavelengths. The intensity of the emergent beam of X-rays after interaction with matter is given by Kinyua (1982) and Gianque and Jaklevic (2008):

$$I = I_o e^{-\mu x} \tag{1}$$

where, I and I_o, are the emergent and incident monochromatic beams of X-rays as they leave and enter an absorber of thickness, x and linear absorption coefficient, μ. In x-ray Fluorescence spectrometry, the count rate obtained from a particular element when a specimen is irradiated is proportional to the concentration of the element. Quantitative analysis depends on the correlation of the elemental concentration with the observed fluorescent intensities. This correlation is achieved by using FPT, which assumes that:

- A monochromatic primary source is used to excite characteristic X-rays from the sample.
- The sample is homogeneous i.e., the density is constant throughout the sample.
- A fixed geometry of the sample, source and detector orientation are maintained.

The intensity equation is given by Sparks Jr (2002) and Jenkins (2000) as:

Table 1: The thickness contribution in nm of each element to the film on the substrate

Substrate No.	Ca (nm)	Ti (nm)	Mn (nm)	Fe (nm)	Ni (nm)	Cu (nm)	Zn (nm)	Pb (nm)	Sr (nm)	Zr (nm)	Total thickness (nm)
1	1062.4	10.1	2.1	11.3	7.1	0.8	1.0	0.3	19.4	10.2	1124.7
2	924.5	3.3	1.2	8.2	8.3	0.4	0.7	0.4	73.3	12.9	1033.2
3	1075.3	10.6	3.9	11.8	13.1	1.3	2.1	0.9	18.9	10.5	1148.4
4	877.4	12.9	1.2	9.0	14.6	0.6	1.0	0.7	72.9	12.3	1002.3
5	853.8	4.0	2.0	10.2	25.0	1.0	1.3	0.7	29.3	12.9	940.2
6	909.7	6.7	0.6	9.1	27.8	0.9	1.3	0.3	74.4	12.6	1942.9
7	903.2	3.3	2.4	9.7	42.1	1.2	1.4	0.9	74.0	12.3	1050.5
8	1023.7	4.5	5.2	13.3	68.4	1.0	2.6	0.9	20.0	10.5	1150.1
9	924.5	7.3	3.0	8.7	83.8	1.1	1.1	0.5	73.3	12.4	115.7
10	922.6	8.6	3.7	8.8	99.9	1.8	1.6	0.5	71.4	12.6	1131.5
11	1064.5	11.9	5.9	13.6	225.6	1.8	2.4	1.0	19.4	10.3	1356.4

Table 2: The average value of thickness contribution from each element in the five (5) blanks

Element	Ca	Ti	Mn	Fe	Ni	Cu	Zn	Pb	Rb	Sr	Y	Zr
Thickness in nm	1227.1	5.9	2.5	28.5	0.6	1.6	1.3	1.2	9.2	13.4	2.0	17.5

$$I_i (E_i) = G_o K_i \in (E_I) \propto_I \frac{(1-\exp(-a\rho d)}{a\rho d} \qquad (2)$$

Was used;
where,

$I_i (E_i)$: The measured fluorescent intensity of the element

$G_o = I_o \, \Omega_1 \, \Omega_2 \text{ sc cos } \emptyset_1$: The geometrical constant whose units are counts per second

K_i : The relative excitation efficiency for the element i given by Kinyua (1982):

$$K_i = \sigma^{ph}{}_i (E_i)\left\{1 - \frac{1}{j_k^i}\right\} f_k^i \omega_k^i \qquad (3)$$

in which,

$(1 - \frac{1}{j_k^i})$: The relative probability (fraction) for the photoelectric process to occur in the K shell

σ_i^{ph} : The element photoelectric cross-section for primary radiation of energy E_i

ω_k^i : The fluorescence yield of the element i for the K-shell

ρ & d : The density and the thickness of the sample respectively

$\in (E_i)$: The relative efficiency of the detector at energy, E

$\propto_i$: The elemental concentration of i in gcm-2

$f_k^i = \frac{I(a)}{I(a) + I(\beta)}$: The ratio where I ($\propto$) is the intensity of a given K line and I ($\propto$) + I (β) the total intensity of the K and L series

$\propto = \mu_s (E_i)\text{csc } \emptyset_1 + \mu_s (E_i) \text{ csc } \emptyset_2$: The total absorption for primary and fluorescent radiation in the sample

If $a\rho d \ll 1$, we deal with thin films, then $I_i (E_i) = G_o K_1 \in (E_i) \, \rho d$ (Jenkins, 2000).

Energy-Dispersive X-Ray Analysis (EDXRA) employs semi-conductor detectors which are very sensitive with quick response to weak mono-energetic excitation sources such as radioisotopes or filtered radiation. This technique is advantageous because it enables multi-element determination.

In the study, the surface of the substrate containing the film was placed over the source for irradiation. The characteristic X-rays from the sample together with the scattered radiation were utilized for estimating film thickness using FPT. Characteristic X-rays and scattered radiation were detected by Si Li detector.

When these radiations interacted with detector, electrical impulses were generated, were amplified and shaped by both pre-amplifier and main amplifier. After amplification and shaping, the electrical pulses were fed into Analog Digital Converter (ADC) for computer data acquisition.

Software package known as Analysis X-ray spectra by Iterative Least square fitting (AXIL) was used to obtain the concentration of every element in the film deposited on the substrate. The software computed concentration in terms of $\mu gm/cm^{-2}$ for each element. The thickness contribution by each element was calculated using the relationship pd equal to concentration. The results for various thicknesses determined and are shown in Table 1.

The thicknesses contribution of each element in the blanks was determined. Five blanks were used to obtain average thickness contribution of each element in the blank and results are shown in Table 2. Comparison between Table 1 and 2 was made to eliminate elements which did not contribute to film thickness in Table 1. It was found that most of the elements did not contribute to the film thickness in Table 1. It was found that most of the elements did not contribute to the film thickness except Nickel (Ni) and Strontinum (Sr). The results in Table 3 show the film thickness of the samples due to Ni and Sr. To assess the validity of this technique for determining film thicknesses, analysis was done on

Table 3: The thickness of nickel-strontinum films deposited on substrates

Substrate No	Nickel (nm)	Strontinum (nm)	Total thickness in (nm)
1	6.5	6.0	12.50
2	7.7	59.9	67.6
3	12.5	5.5	18.0
4	14.0	59.5	73.5
5	24.4	15.9	40.3
6	27.2	61.0	88.2
7	41.5	60.6	102.1
8	67.8	6.6	74.4
9	83.2	59.9	143.1
10	99.3	58.0	157.3
11	225.0	6.0	231.0

Table 4: XRFA of standard thin film samples (Whatman-Mylar backing)

Element	Theoretical calculated thickness in (nm)	Experimental calculated thickness in (nm)	Error (%)
Gold	24.4	25.0	2.5
Molybdenum	35.3	36.9	4.5
Lead	47.8	52.9	10.7

Table 5: Thickness of nikel films and their sheet resistances in ohms, (Ω)

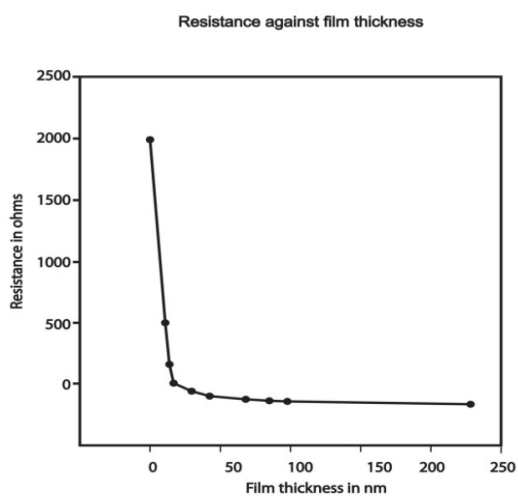| Substrate No	Nickel thickness in (nm)	Slopes, S	Sheet resistance r, in Ω
1	6.5	463.43	2,101.7
2	7.7	116.05	526.3
3	12.5	34.30	155.7
4	14.0	13.38	60.9
5	24.4	6.27	28.4
6	27.2	4.72	21.4
7	41.5	1.24	5.6
8	67.8	1.00	4.6
9	83.2	0.97	4.4
10	99.3	0.48	2.2
11	225.0	0.31	1.4

Fig. 2: Graph of sheet resistance, Rs against thickness in nm

standard films (Mangala and Patel, 1996). Film thicknesses of gold, molybdenum and lead were determined by the same method. The results were as shown in Table 4. In order to measure the sheet resistance, Rs a Four Probe Technique was used. A graph of voltage against current for each sample was plotted and its slope, s, was determined. From the relationship, $S = R_s \frac{1n2}{\pi}$, the sheet resistance for each sample was calculated. The values of nickel thickness in nm and their corresponding sheet resistances were as given in Table 5.

A graph of sheet resistance, Rs against thickness in nm was plotted and the results were as presented in Fig. 2. It is observed from the graph that at a film thickness greater than 50 nm, the sheet resistance, Rs, behaves like a normal resistor i.e., obeys Ohm's Law.

RESULTS, DISCUSSION AND CONCLUSION

Discussion: The thickness of the thin films deposited on the substrates together with the blank elements contributions were as shown in Table 1. Table 2 shows the average value of thickness contribution for each element from the five blanks. After the analysis of Table 2, it was observed that major thickness contribution came from calcium, followed by iron, then zincrorium (Zr), then strontinum and the rest were minor. It was concluded that those elements which had very small contribution when blanks were used and yet had signification contribution when samples were analyzed, were regarded to be the ones that made major contributions to deposited film thickness (Mangala and Patel, 1996).

With that assertion, the average value of the thickness contribution for each element in five samples was substracted from the contribution of the same element in very sample. It was found that the elements that contributed to the film thickness were Nickel and Strontinum. This was arrived at by subtracting resulting of Table 2 from the results of Table 1.

The contributions due to Nickel and Strontinum are shown in Table 3. Quality control procedures test the accuracy of method may be presented earlier. Table 4 shows the results of XRF Analysis of the three standard thin sample-gold, molybdenum and lead (Whatman-Mylar backings). This enabled to estimate the order of accuracy of this method. Table 5 shows the results of the sheet, R_s and the corresponding thickness.

Conclusion: This study has shown that XRF method is useful in determining elemental thickness in films deposited on substrates. Using this technique, one may determine many elemental thicknesses simultaneously within relative errors of 10% or better. The only limitation of this technique is in measurements of light elements such as silicon at short wavelengths. The investigation has shown that film thicknesses of high

atomic number elements used in video tapes, audio tapes and film resistors can be determined by using FPT method. It is observed that the sheet resistance, R_s, is inversely proportional to the thickness of the film on the substrate and this is in agreement with what Katumba found in his investigation of aluminimum thin film resistors. From the graph, one observes that when the film thickness is greater than 50 nm, films behave like ordinary resistors. In other words in bulk, films obey Ohm's law if other physical quantities remain constant. It is therefore suggested that this method could be used in conjunction with inteferomentry technique which Katumba (1994) used.

ACKNOWLEDGMENT

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