

OBAFEMI AWOLOWO UNIVERSITY, ILE-IFE, NIGERIA.

INAUGURAL LECTURE SERIES 280

**ELECTRONIC DEVICES IN
THE SERVICE OF MAN**

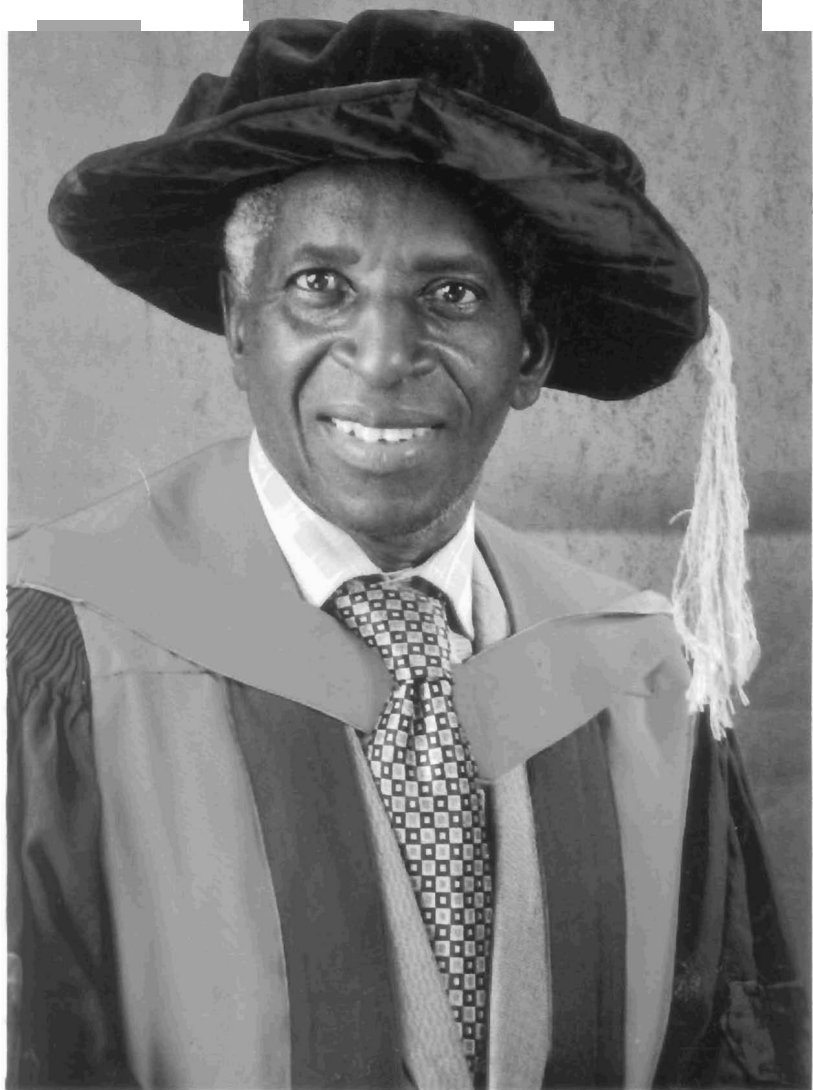
By

OLADIPO OSASONA

Professor of Electronic and Electrical Engineering



OBAFEMI AWOLOWO UNIVERSITY PRESS, ILE-IFE, NIGERIA.



OLADIPO OSASONA

Professor of Electronic and Electrical Engineering

ELECTRONIC DEVICES IN THE SERVICE OF MAN

**An Inaugural Lecture Delivered at Oduduwa Hall,
Obafemi Awolowo University, Ile-Ife, Nigeria
On Tuesday, 24th November, 2015**

By

OLADIPO OSASONA
Professor of Electronic and Electrical Engineering

Inaugural Lecture Series 280

OBAFEMIAWOLOWO UNIVERSITY PRESS, 2015

ISSN 0189-7848



Printed by

**Obafemi Awolowo University Press Limited
Ile-Ife, Nigeria.**

Introduction

Mr. Vice-Chancellor sir, Principal Officers of the University, colleagues, visitors, students, friends, family members, ladies and gentlemen, I thank God for sparing my life to stand before this distinguished audience to deliver my inaugural lecture titled "Electronic Devices in the Service of Man". It is the 6th inaugural lecture in the Department and the first to be given by a holder of a Ph.D. awarded by the Department of Electronic and Electrical Engineering; I have therefore gained so much more than I could possibly give to God and the University and I am incapable of adequately voicing my gratitude.

Electronic Devices in the Service of Man

In the beginning, God gave man dominion over the things He created. Some such creations of God that have been so very well utilized (through technology) are electronic materials. Electronic materials, in turn, gave birth to electronic devices, and electronic devices are the foundation of electronics and power electronics. These technologies have become indispensable in supporting our lifestyle, our health and our safety, social interaction and security. None of today's industries would have been viable without electronic communication, automation and control. Through Electronics, people stay connected, get supported in their professional life; and can enjoy leisure entertainments. Transportation and energy-supply also depend on electronics (Burghartz, 2013). Have we ever stopped to think of how academics have been coping with their research-life in life or anywhere else in Nigeria, before the advent of Internet facilities? Consequently, electronic devices have revolutionized the way we live and think, and our civilization in general.

The world has an insatiable appetite for higher-performing electronic devices which drives the search for new electronic materials and devices. As new challenges emerge, electronic circuit designers and manufacturers of electronic

devices respond to these challenges with novel solutions. For example, as a fallout of the outbreak of the Ebola virus, non-contact infra-red thermometers have been made popular.

The invention of the first successful thermionic valve, known as the “electronic vacuum tube” (i.e a diode, or a Fleming valve) in 1904, by Sir John Ambrose Fleming (often called the Father of Modern Electronics), initiated the era of electric-charge-carrying devices. Fleming's invention was the precursor of all electronic tubes - a development that gave birth to radio communications and the entire electronics industry (Morris, 1993).

Lee De Forest invented the vacuum triode (called “audion”) in 1906. What he discovered was indeed an “invisible empire of the air, intangible, yet solid as granite” (De Forest, 1907). Lee De Forest went ahead to develop the radio (Campbell *et al*, 2000).

The invention and development of vacuum-tube devices for communications and sensing predated the age of transistors. However, it was the advent of solid-state “triode” (later named “transistor”) by John Bardeen, Walter Brattain and William Shockley in 1947 (Shockley, 1984), followed by the monolithic Integrated Circuits – an innovation of Jack S. Kilby in the late 1950s -that laid the conceptual and technical foundation for the entire field of modern Microelectronics. However, in 1965, silicon process technology created the manufacturing conditions necessary for the commercialization of Integrated Circuits (IC) and consequently, the microelectronics industry, as we know it today. The integrated circuit technology has ceaselessly pushed new frontiers in Computers, Communications and many other emerging areas like Mechatronics, in recent decades.

The evolution of quantum mechanics provided the theoretical knowledge necessary to identify semiconductors with moderate band-gap, as the materials of choice to realize the Field Effect Transistor (FET) device.

In today's integrated circuits, Metal Oxide Semiconductor (MOS) transistors are the primary devices. The field effect mechanism on which the MOS device is based, was first investigated in the 1930s . MOS devices offer some advantages such as higher density, lower power operation, and considerable greater design flexibility than Bipolar Junction Transistor (BJT) devices.

The effect of Moore's Law and Robert Dennard's scaling Theory.

The Integrated Circuit technology started from a modest beginning, which permitted the integration of a few semiconductor devices and passive components, on a common substrate. It has grown to become a technology which can integrate tens of millions of components in a square centimeter of silicon. As time went on, more complex circuits were built simply by making the individual components smaller. This down-sizing has allowed more of the components to be integrated in a given area. The overall size of chips has also been made larger. The combination of smaller device sizes and bigger chips has enabled the possibility of building more complex, higher-performance, and more economical integrated systems. Improvements in technology over the past 45 years have resulted in a doubling of the number of transistors on a chip roughly every two years. This "law" has become known as "Moore's law" after Gordon Moore (Plummer *et al*, 2000; Gargini, 2004). How long can the trends of shrinking transistors and increasing chip complexity continue? It will be answered by the effect of quantum tunneling and the line resolution of the lithographic process employed in the fabrication of the devices. Figure 1 illustrates the increasing complexity of integrated circuits over the years.

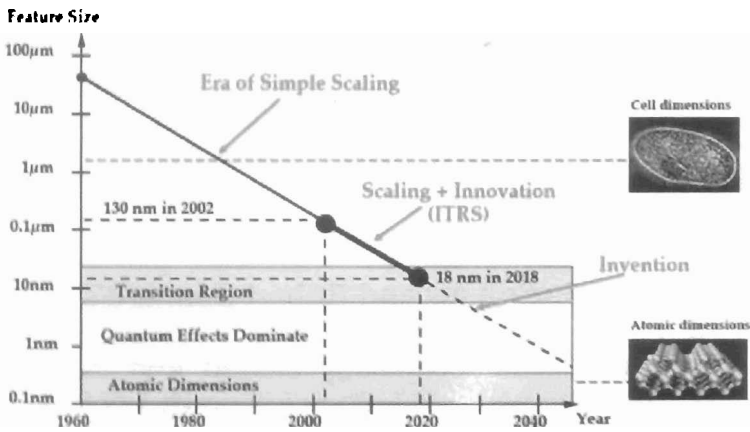


Figure 1: Device Scaling Over Time (Source: Plummer *et al* (2000))

With increasing chip complexities and reduction in sizes and in power supply to run the system, coupled with automated mass-production technologies, the application of electronic devices has therefore, virtually taken over our many facets of life. They now play critical roles in restoring lost function and improving the quality of life. For example, pacemakers have become so common (with implants routinely carried out all over the world including life) that it is easy to forget that **arrhythmias** used to lead to drastic lifestyle changes. Even more exciting is the class of devices termed

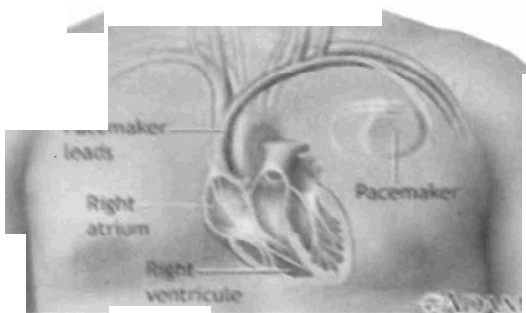


Figure 2: The Pacemaker (Chen, 2014)

Neuroprostheses, which interface with the nervous system to restore sensory and motor function in patients as shown in Figure 3. Over 300,000 people now carry cochlear implants, without which they would have been deaf (National Institute of Health, 2013), while remarkable progress is being made in the restoration of vision using visual prostheses, and there is increasing evidence that serious damages to the central nervous system that now result in paralysis can be repaired or circumvented. This means that the deaf can “hear”, the blind can “see” and the lame can “walk”. In fact, Electronics now allows us to extend the normal range of human motor and sensory capabilities. The output of any arbitrary sensor can now be integrated directly with the brain and central nervous system through brain-machine interfacing, and there are now people out there who can sense magnetic fields and ultrasonic waves (Warwick, 2005), among a growing number of “superpowers”.

There are many aspects of medical practice that have been revolutionized by the application of electronic devices but will be too numerous to cover in this lecture. However, the aspect of Telemedicine, where medical practice is made possible by the use of the Internet, is important. This form of medical practice has helped to extend the coverage of doctors to areas where it would have been humanly impossible to cover.

Automobile Industry

The automobile industry has also been revolutionized by developments in microelectronics. Luxury vehicles have more than a hundred and fifty sensors built into them. Many diagnostic tools and tracking devices are included in modern vehicles. Therefore with these tracking devices, the speed of a car can be monitored and regulated. Also, its location can be monitored and, in extreme situations, the car can be remotely stopped. If it has been stolen, an arrest can be made. Recently, some luxury cars were stolen from the

United Kingdom and moved to Uganda. . They were tracked and recovered in September, 2015 (Yahoo News UK, 2015).

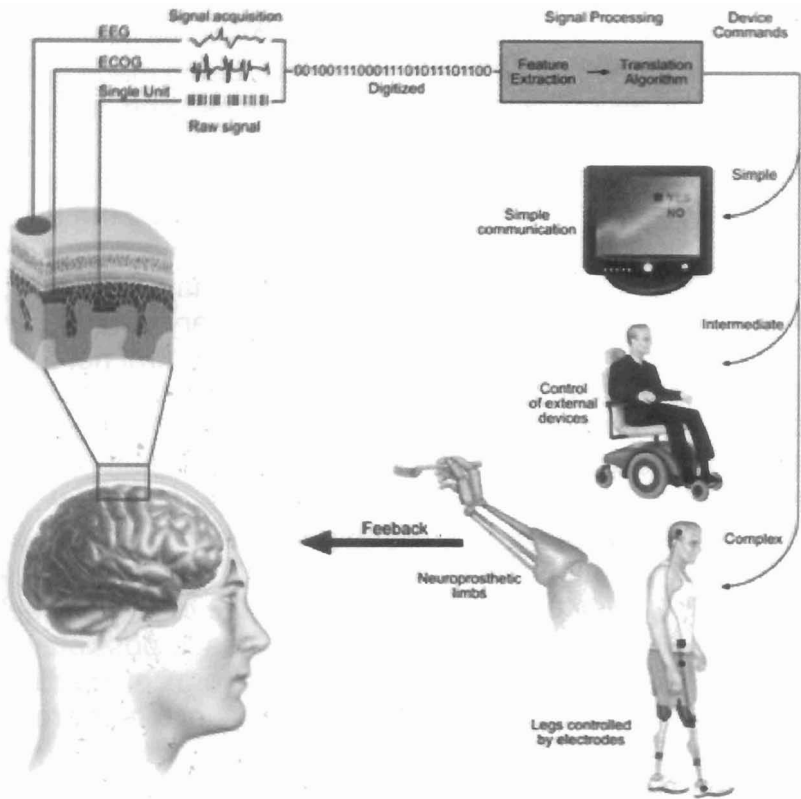


Figure 3: Showing Electronic Implant Technology to Extend Human Sensory Capabilities (Source: <https://en.wikipedia.org/wiki/Neuroprosthetics> Wikipedia 8th October, 2015).

Car-manufacturing plants are almost completely taken over by robots. Generally, repetitive jobs are assigned to machines so human labour force is reduced in size and hence can be put into more quality use. The new dimension is to use autonomous vehicles that are without human drivers!

Animal tracking

Animal tracking (to study their migration) also relies on electronic devices. The fruit bats in Obafemi Awolowo University were being tracked by one of my M.Sc. students in conjunction with the late Professor E.E. Okon of Biological Sciences. A wearable device was designed to help track their movements over the seasons (Adesina *et al.*, 2015). We are aware of the fact that they migrate to other places and are almost completely absent from Obafemi Awolowo University campus during a part of the year.

Cameras

Mo Ti, a Chinese philosopher, was the first to exploit the phenomenon that a pinhole can form an inverted and focused image, when light passes through the hole and into a dark area. He was the first recorded person to utilize this phenomenon to trace the inverted image to create a picture, in the fifth century B.C. From history, the forerunner of the photographic camera was the camera obscura. (Batchen, 1997) A **camera obscura** from Latin, meaning "dark chamber", is an optical device that led to photography and the photographic camera.

As a result of the fact that the camera works with light from the visible spectrum and with other portions of the electromagnetic spectrum, many other versions of the camera have been invented and made use of. From this principle of the camera came the cinematography, video and surveillance cameras, with night and day vision capabilities. It is needless to go into the various developmental stages of the camera, however, it is necessary to note that cinematography and video cameras provide entertainments for man and have helped to aid cultural exchanges through television broadcasts. Also very useful to man are the surveillance cameras. As crimes become more complex and crime rates increase, curbing crime or reducing it has become a challenge to the human capabilities of security operatives. With the introduction of surveillance cameras,

life can be made more secured as security operatives can remotely monitor larger areas using the Internet. Finally, with the introduction of in-built cameras to cellphones, crimes can be reported immediately as everybody has become a photographer and a security officer!

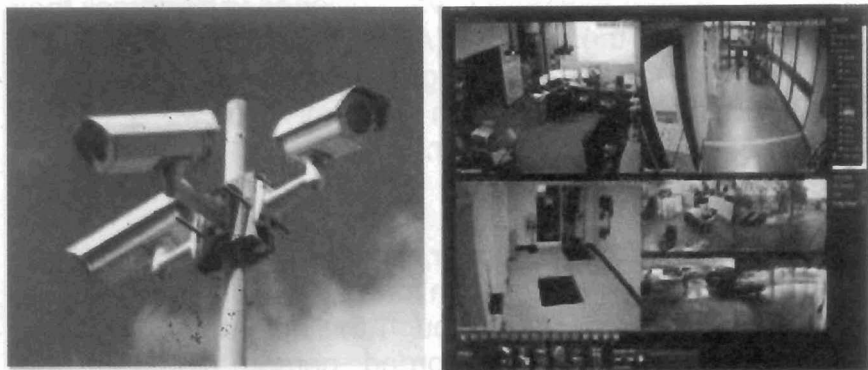


Figure 4: Showing Surveillance Cameras and Monitors

Cell phones

Some years ago, the Nigerian Telecommunications Limited (NITEL) provided only two digital lines for the whole of Ile-Ife Telephone Exchange, it later increased it to six, all other lines were analogue. This implied that to make a call to a country outside Nigeria was limited to the services that can be provided by those few lines. These attendant problems are now history. Then the GSM was introduced to affect our lives, positively and negatively. The advantages far outweigh the disadvantages in advanced country with the reverse being the case in Nigeria. For example, the cell phones made communications easier, bailed us out of the captivity of NITEL and also reduced the number of trips made. However, crime rates increased. Our future is being mortgaged as our future generation is grossly affected. It improved examination malpractices, reduced concentration of our students in class, as they could be watching football matches or films during classes, or even listening to loud music using their headphones.

We are consequently breeding a future generation of deaf people (from Noise Induced Hearing Loss (NIHL)). Auto accidents have increased from lack of concentration on the part of drivers, or people crossing the road while sending text messages on the phones. There is need to correct the side-effects of this useful invention.

Banking

With the introduction of Internet Banking and Automatic Teller Machines, ATM (employing cards containing embedded electronic devices), our economic lifestyles have taken a new turn. In the comfort of one's home, one can transact business with one's customers, moving monies from one point to another. One no longer has to carry cash around – or queue in front a Cashier at the bank - to transact business.

Man has become mentally lazy because of the introduction of calculators. We can hardly multiply 2 by 2 without resorting to the use of a calculator! We now remotely control our dishwashers, washing machines, microwave ovens and even remotely switch on and off appliances in the home. These are a few examples of how electronic devices have modified our lifestyles.

My Academic Journey

I would not have seen the four walls of a university but for the sacrificial sponsorship of my brother Dr. Olagbemi Osasona. To the glory of God, I graduated from the Department of Electronic and Electrical Engineering, University of Ife (now Obafemi Awolowo University) in June, 1975 with a Bachelor of Science in Electronic and Electrical Engineering with a First Class (Honours) degree. After my Youth Service with NEPA, I joined the Department of Electronic and Electrical Engineering as a Graduate Assistant (since no Public Service sector would employ anybody with a First Class degree at that time, knowing fully well that they would go back for further studies).

As a matter of fact, the External Examiner (who normally came either from the University of Manchester or the University of Sussex) already gave those of us who made a First Class, admission letters for further studies in their University in the UK. My inability to take up the admission offered me to Manchester University was technical. I was advised by well-meaning senior colleagues to utilize my time of delay for meaningful academic work. I, therefore, registered for an M.Phil. degree with the Solid State Electronic Research group. It was the highest degree offered in the Department at that time. Through the years - and employing what is now known as multi-site postgraduate work - I became the first to be awarded a Ph.D. degree in the Department of Electronic and Electrical Engineering. I spent part of my research time at the Imperial College, London in 1980, and the University of Sussex, Falmer, Brighton, supported by the Sir Adam Thompson (British Caledonian Airways) Scholarship, in 1984 and 1985.

The Research Group

The Solid State Electronic Research group, like any modern Materials Research Group saddled with a great complexity of tasks, is a multi-disciplinary one, comprising electronic engineers, theoretical physicists, mathematicians, chemists and many others. The research group aimed at providing the needed manpower for the fabrication of electronic devices, for the technological advancement of Nigeria, by setting up a prototype fabrication laboratory. Eventually, the laboratory became too expensive to run and consequently became extinct. Some of the reasons for its demise included instability in the Nigerian political system (manifesting as lack of continuity and focus on the part of those who rule over us). The final nail in the coffin was the dwindling financial support to the University system, as a result of lack of appreciation for what higher education stands for in the development of any country.

electronic components in Nigeria, to date. I was an active member of the team since my academic survival (at that time) depended on the success of the team. Details of the work in the laboratory are as reported in Osasona (1988), Williams *et al.* (1984), Adegboyega (2000) and Kuku (2006).

Fabrication of Resistors

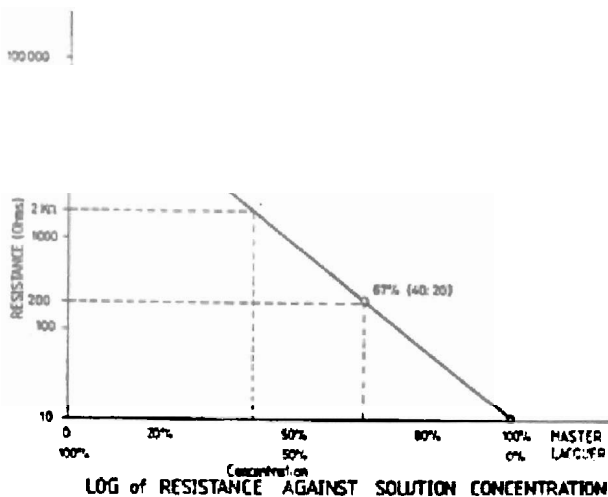
Resistors are the commonest components in any electronic circuit. They can appear in the discrete, embedded or surface mounted form. In most cases, their fabrication can be a simple technology. The resistance value R is proportional to length, l , and inversely proportional to the area, A and resistivity ρ , is the constant of proportionality.

i.e.
$$R = \frac{\rho l}{A} \text{ohms} \quad \text{Equation 1}$$

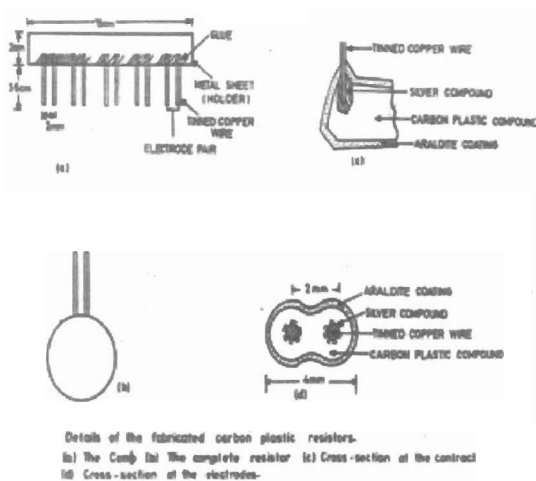
The resistivity ρ is a property of the material element used therefore, depending on the ingenuity of the circuit designer, the length l or the area A can be varied to obtain the desired resistance value.

The research group started with the fabrication of resistors. Three types of resistors were embarked upon:

- (1) **thin film**, produced by the evaporation of Nickel-Chromium (Ni-Cr) or nichrome wires or sputtering, in a vacuum chamber.
- (2) **tin oxide**: produced from the abundance of *cassiterite* from the Nigerian tin mines and
- (3) **carbon-plastic Composition** produced by mixing carbon powder with a resin in a series of predetermined proportions to obtain the "slug type" range of resistors. The mixture is then pressed or moulded into cylindrical shapes with axial wire leads anchored in the moulding operation. A plastic (phenolic resin) coating formed around the moulding insulates and gives the mechanical protection. It is low cost, though "noisy" but preferred in consumer electronic equipment. A variation of this technology was developed in the laboratory (Williams *et al.*, 1984).



(a)



(b)

Figure 5: (a) Log of Resistance Against Solution Concentration and (b) Details of the Fabricated Carbon Plastic Resistors.

My undergraduate project with the group in 1974/75 session, was supervised by Mr. Laszlo Szolgyemy. I worked on the characterization and reliability studies of the resistors

fabricated. The technology developed in the laboratory made use of Lacquer plus 30% carbon soot (particle size between 10-100A° dispersed in the lacquer which served as the solvent. The result was a colloidal suspension of about 10-ohm bulk resistance. To obtain higher values of resistance, the above solution was diluted with another one of composition: 50% *Dialkid ERS* plus 50% *Melamin* thermosetting plastic.

Table 1: Tests carried out on Fabricated Resistors

Test	Resistor Types		
	Carbon Composition	Tin Oxide	--
Damp Heat Test	0.602% to 2.340%	0.111%	1.890%
Temperature Co-efficient of Resistance	560-1996 ppm	402.6 ppm	458 ppm
Mechanical Stability	Stable	Stable	Stable

The resistor technology developed was simple and inexpensive though the process was labour intensive. The products were compared with other commercially available resistor types i.e. tin oxide and carbon “slug type” and the results are shown in Table 1.

Components produced at that time were exhibited in many exhibition centres in Nigeria. They are still exhibited outside Oduduwa Hall today.

Fabrication of Semiconductor Devices

A turn-key project worth three million US dollars (\$3,000,000.00), at the exchange rate of seventy-five Kobo (₦0.75) to a dollar, was offered to the Federal Government of Nigeria in 1973 but was turned down. The project was to make use of the locally available sand to produce transistors as part of a complex producing domestic electronic appliances for the country. A similar project was successfully established in Tunisia, North Africa also in 1973! Never-the-less, the Solid State Research group, University of Ife (now Obafemi Awolowo University) still went ahead on a

“technology transfer” type of project to fabricate electronic devices e.g. diodes and transistors. The Research group believed that it would be expedient to produce the necessary high-level manpower for the take off of Nigeria’s technological development. It believed that the motivation to proceed in acquiring and developing this devices technology far outweighed the disadvantages. In a way, the group came ahead of its time.

The composition of Igbokoda quartz sand, where the “Turn-key” project would have been cited and where Oluwa Glass factory was eventually cited, was analysed in 1987 by Dr. O.O. Adewoye (Prof. Adewole has passed on) and found to contain high-grade silicon for devices fabrication. The composition, in weight percent, was found to be: SiO_2 -97.6; Al_2O_3 - 0.6; Fe_2O_3 – 0.2; CaO – 0.5; and loss on ignition -0.2. Silicon was successfully extracted, by aluminum reduction, from the Igbokoda quartz sand though resources were not available to realize pulling single crystal silicon ingots (Williams *et al*, 1977).

Electron-Probe Testing of Fabricated Silicon Planar Devices

The fact that a silicon planar transistor was fabricated in the Semiconductor Research laboratory at Ife, with the various production steps empirically determined for optimum device yield, and the attendant problems encountered in the fabrication steps, have been widely reported (Osasona, (1988), Adegboyega, (2000), Kuku, (2006). It was the first time a silicon planar transistor was fabricated in Nigeria.

The starting wafer was an n-type Si(111) substrate of resistivity 4 – 5 ohm-cm on which a 10-micron thick n+ epitaxial layer was deposited. The resistivity of the epitaxial layer was 0.8 ohm-cm. My contribution to this work was to examine the structural integrity of the starting silicon wafer using the Reflection High Energy Electron Diffraction (RHEED) system commissioned by me (Osasona 1981).

The chemical and morphological maps of the transistor were recorded in the form of Auger maps, biased-Secondary Electron Images and Absorbed Current Images (as contained in Figure 6) using the University of Sussex modified HB50 Ultrahigh Vacuum Scanning Electron Microscope (UHV- SEM). It towered above others in its time with a two-lens Field Emission Gun (FEG) SEM with a nominal 5 nm resolution. It was also equipped with many novel features like in-situ Molecular Beam Sources (e.g. Knudsen Cell), Quadruple Mass Spectrometer, Auger Electron Spectroscopy (AES) and Scanning Auger Microscopy (SAM) of 30 nm resolution from the attached Cylindrical Mirror Analyser (CMA). Detectors for Reflection High Energy Electron Diffraction (RHEED); Electron Backscattering Patterns (EBSP) at 10 nm lateral resolution, and in-situ specimen cleaning facilities. The entire unit was interfaced to a Digital Equipment Corporation (DEC) PDP-11/32 On-line computer for data acquisition and storage. The details of this system are as contained in (Osasona, 1988; Venables, 1976; Venables *et al.*, 1976; Venables and bin-Jaya, 1977; Venables and Janssen, 1978; Venables and Archer, 1980; Venables *et al.*, 1983a & b).

Energy-selected Auger aluminum (1396 eV) map (Figure 6 (ii) (c)) was acquired using a dynamic background subtraction technique in Figure 6(i) Spectrum **b** from the equation:

$$\frac{A - B}{A + B} \quad \text{Equation 2}$$

Where A is 1396 eV and B is 1500 eV selected from Figure 6(i) (b).

Energy-selected Auger Oxygen (487 eV) map (Figure 6(ii) b) also obtained using the same method. These chemical-specific scans showed areas covered by aluminum metallization layer, for contact, and silicon oxide (from where Oxygen was picked are clearly visible. Biassed Secondary Electron Image of a section of the transistor showed only

topographic features. The circular steps resulted from windows opened for diffusion and drive-in processes. The fabricated transistor was found to be similar in shape and characteristics to BFY33, a general purpose, medium power type (Osasona, 2002; Osasona and Adegboyega, 2003). The problem of device degradation arising from improper alloying of contacts (to the device) was also studied by me with a view to finding new metallurgical schemes for the device metallization. Consequently, the formation of Palladium silicide on (111) and (511) silicon substrates was studied. With Auger depth profiling technique, a large collection of Si on Pd₂Si surface, confirming non-stoichiometry at the surface (63.1% Si to 14.4% Pd by weight) was discovered. This phenomenon is the basis for Solid Phase Epitaxial (SPE) growth of silicon (Rubloff *et al* (1981), Poate *et al* (1978), Freeouf *et al* (1979); Freeouf *et al*, (1980); Oustry *et al*, (1982)).

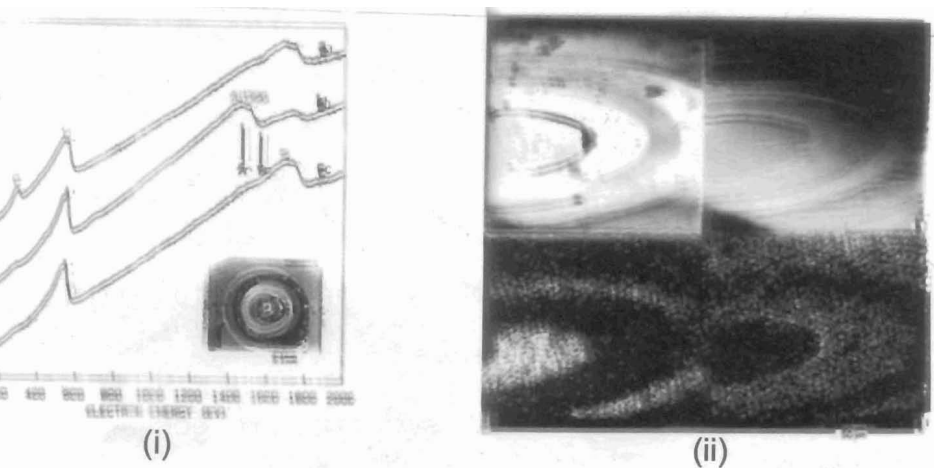


Figure 6: (i) Micro Auger Spectra obtained from the Transistor after the bakeout process.
(ii) Pulse Auger Mapping of the Iife Transistor using 6(i) Spectrum b.

The current-voltage (I-V) curves of the Pd₂Si /Si interface in Figure 7 showed a rectifying characteristics. A higher forward current was observed in the Pd/Si(111) system than in the Pd/Si(511) interface. The intermixing between Pd and Si atoms was observed to be more pronounced in the (111) substrate than in the (511) substrate.

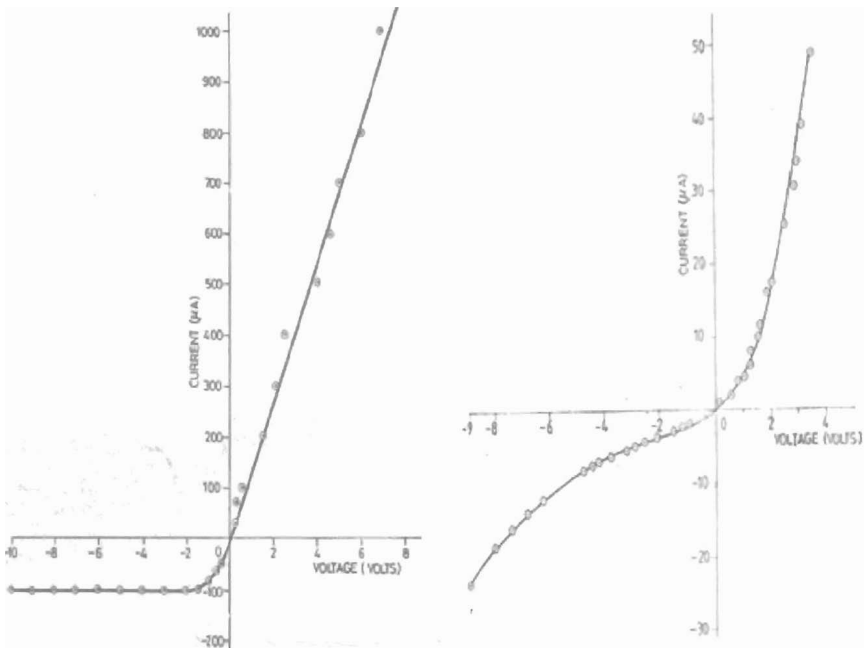


Figure 7: I-V Characteristics of a Thin Film of (a) Pd on Si(111) and (b) Pd on Si(511) Substrate

Studies also carried out on NiCr-Si system using Auger depth-profiling technique showed that considerable intermixing occurred between NiCr and Si even at room temperature. Ni and Cr atoms were detected at about 5 nm depth (Table 2) implying that NiCr was unsuitable for device metallization (Osasona, 1988, Olowolafe *et al*, 1976a and 1976b).

Table 2: Composition of NiCr/Si System Using Auger Depth Profiling

Sample	Depth	Composition in Weight %						
		C	Pd	O	Si	S	Cr	Ni
NiCr on Si(111) Unannealed	Surface	11.7	--	28.9	--	--	37.6	21.8
	5 nm	2.7	--	8.0	66.3	--	11.4	11.6
	20 nm	--	--	--	100	--	--	--

Silver on Silicon(111)

Ordinarily the fact that Ag is a very good electrical conductor would have qualified it to be a good candidate for metallization material for planar silicon semiconductor devices but it was discovered from studies carried out that Ag layers spread on Si. Therefore, the spreading of Ag deposits on S(111) was studied between 350 - 450 °C temperature range. Within this range, no desorption of Ag occurs. After the initial islands formation (Stranski-Krastanov growth mode) Ag begins to spread out of the initial deposit zone (Osasona, 1988 and 1990).

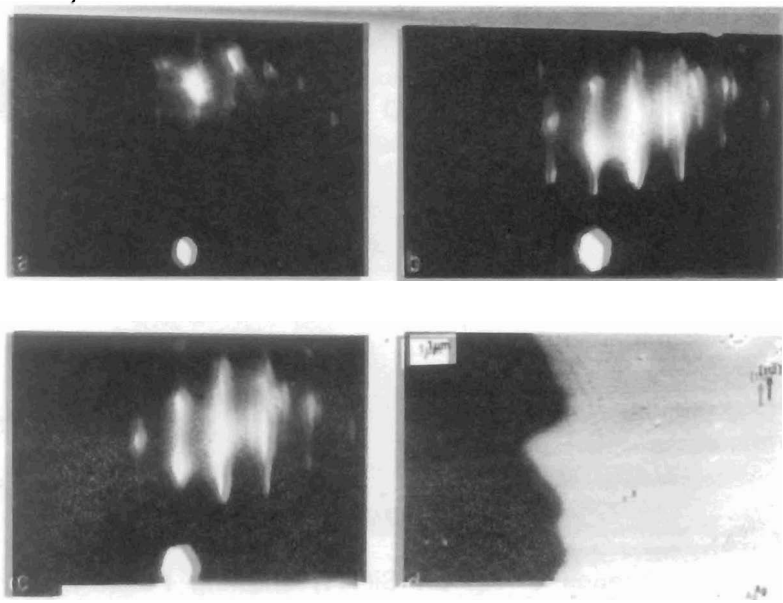


Figure 8: RHEED patterns from Ag/Si(111) system from the $[112]$ azimuth at glancing angle (a) dirty Si(111) surface (b) Si(111) 7×7 patterns from Clean surface (c) $\sqrt{3}$ Ag structure from point "x" in the SEM image in (d).

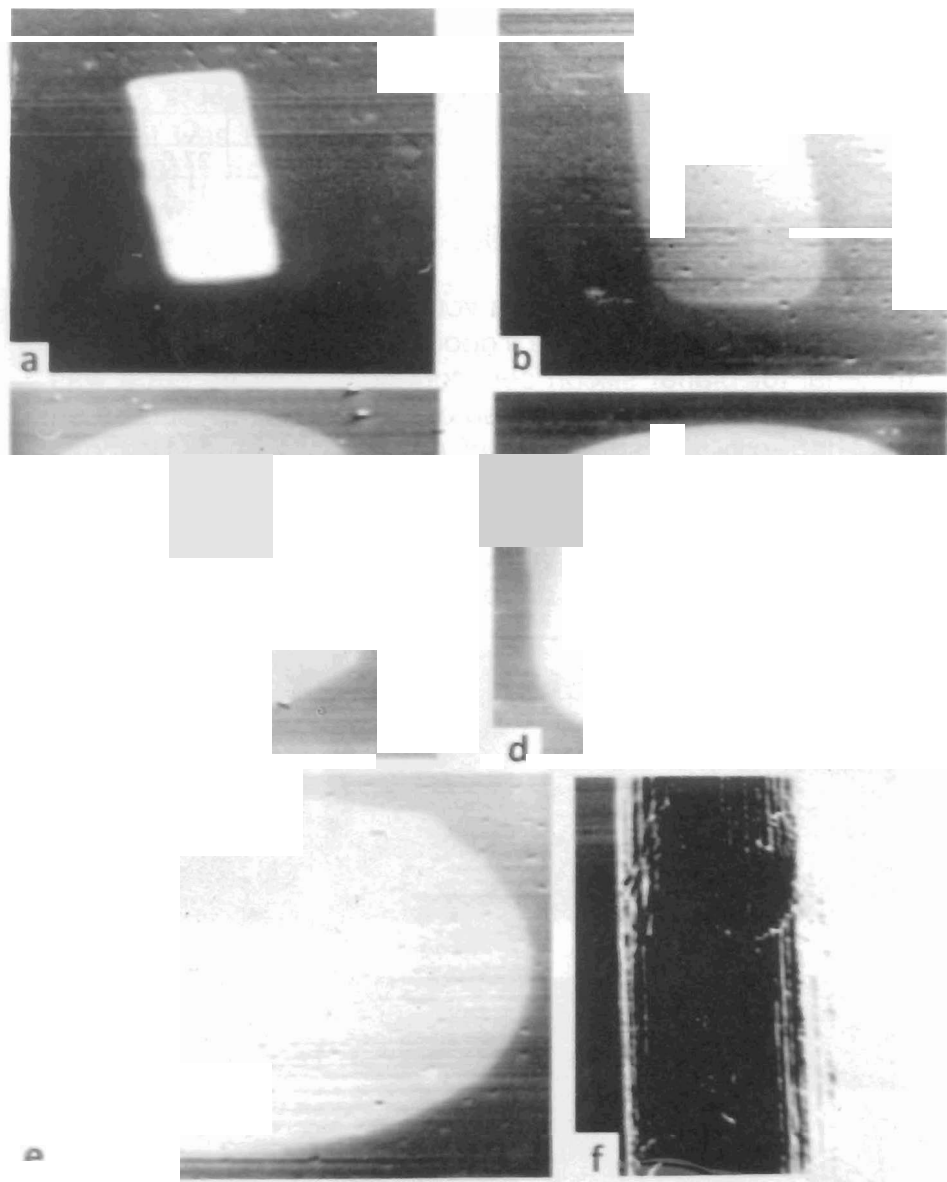


Figure 9: SEM images of Ag on Si(111) showing Patch width broadening as a function of monolayer(ML) coverage (a) 1 ML at Room Temperature through a $20\mu\text{m} \times 100\mu\text{m}$ mask (b) 1 ML at 400°C (c) 3 ML at Room Temperature (d) 4 ML at 400°C (e) 5 ML at 400°C and (f) $50\mu\text{m}$ tungsten wire for internal calibration.

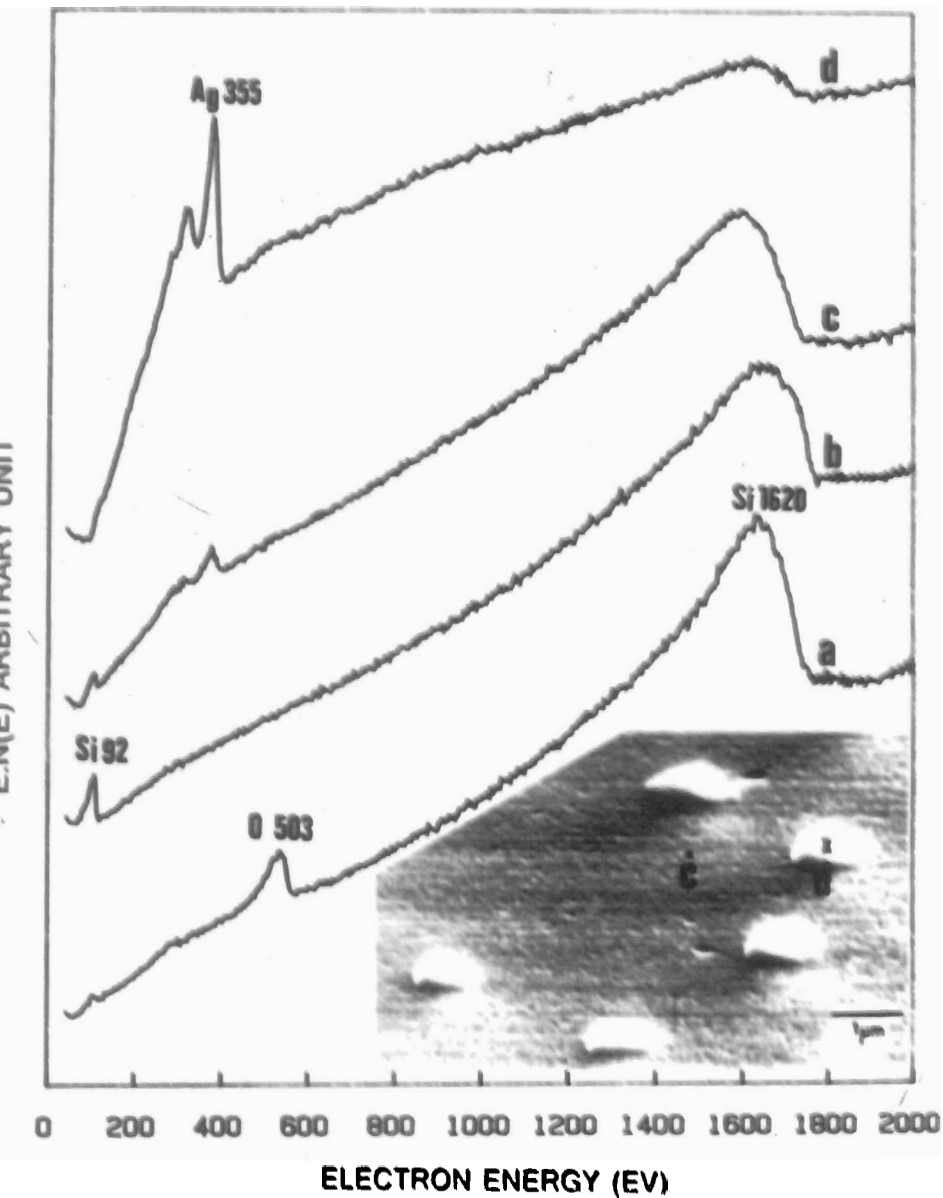


Figure 10: Auger Spectra from Ag/Si(100) showing in (a) carbon contamination (b) clean silicon substrate after cleaning and (d) Ag on Si(100)

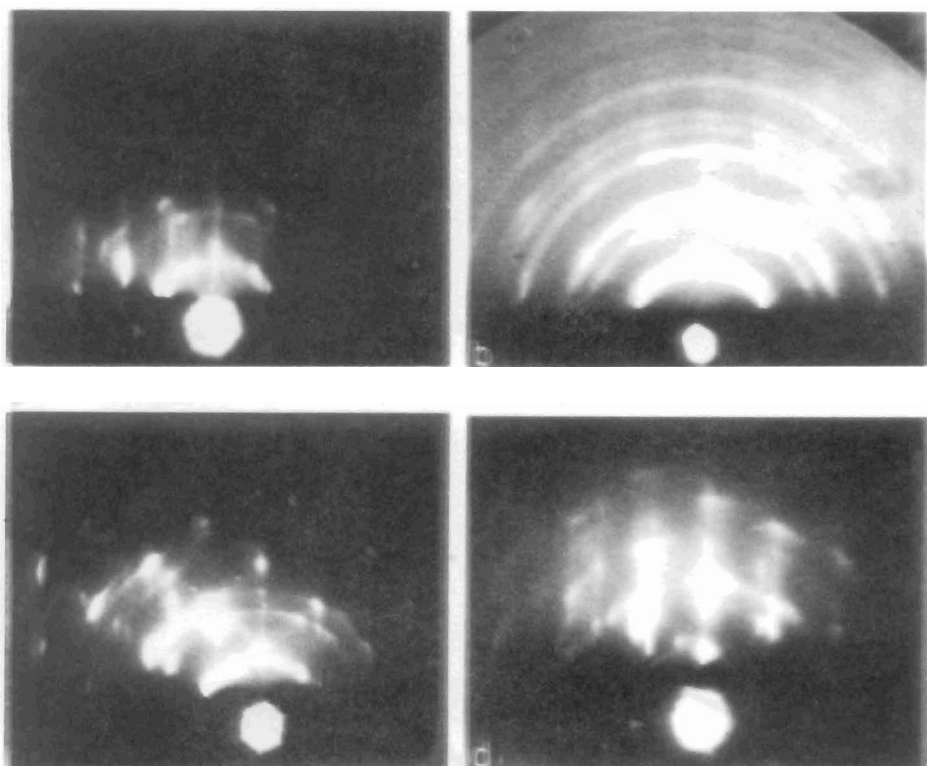


Figure 12. HRTEM patterns from Ag/Si(100) interface (at glancing angle and [011] azimuth): (a) 2x1 patterns from clean Si(100) surface (b) polycrystalline rings of deposits at room temperature (c) deposits at 200°C and (d) annealed room temperature deposition in (b).

Island Orientation

The orientations of the Ag islands formed on the Si substrates as shown in Figure 12 were studied at points marked "x" using the Electron Backscattering Patterns (EBSP) technique. This lecturer modified the Computer programs used in Sussex for the EBSP data analyses from card reader FORTRAN programs. They were made adaptable for use in the analysis of the orientation relationships of deposits and substrates. A 32-page FORTRAN program was therefore written making it possible to identify the orientations of the Ag islands (or any similar islands) formed from the Kikuchi patterns in **Figure 13**.

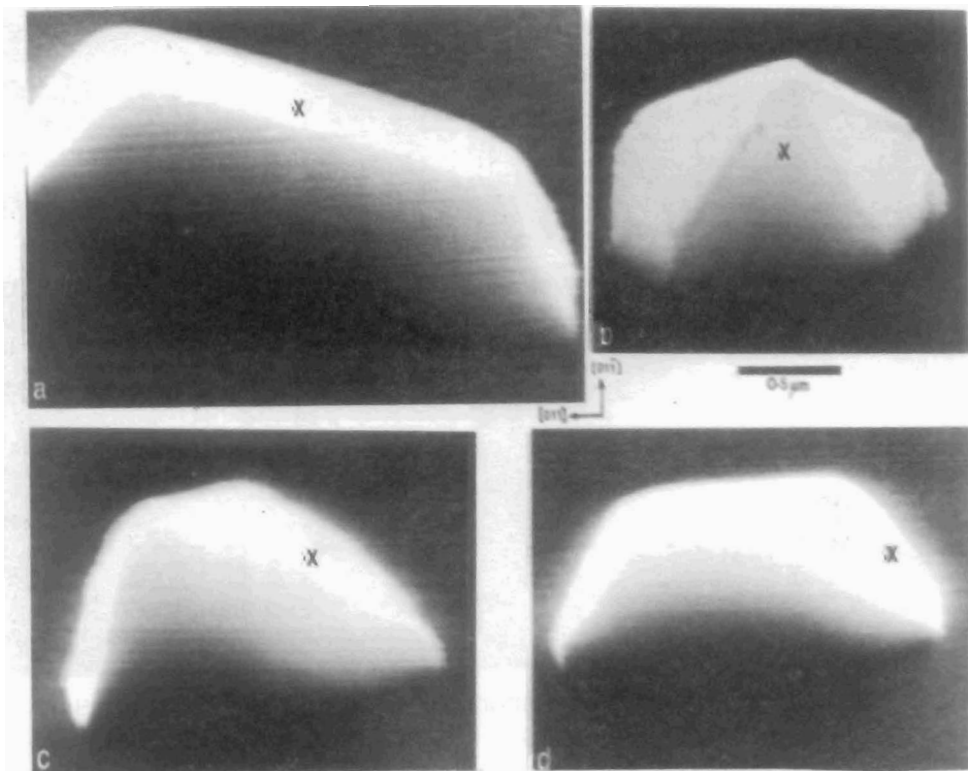


Figure 12: Biased-Secondary Electron Images of different Islands Observed from Ag deposited on Si(100) Substrate and used for orientation identification.

UNIVERSITY LIB
Obafemi Awolowo Univer
ILE-IFE, OSUNDA

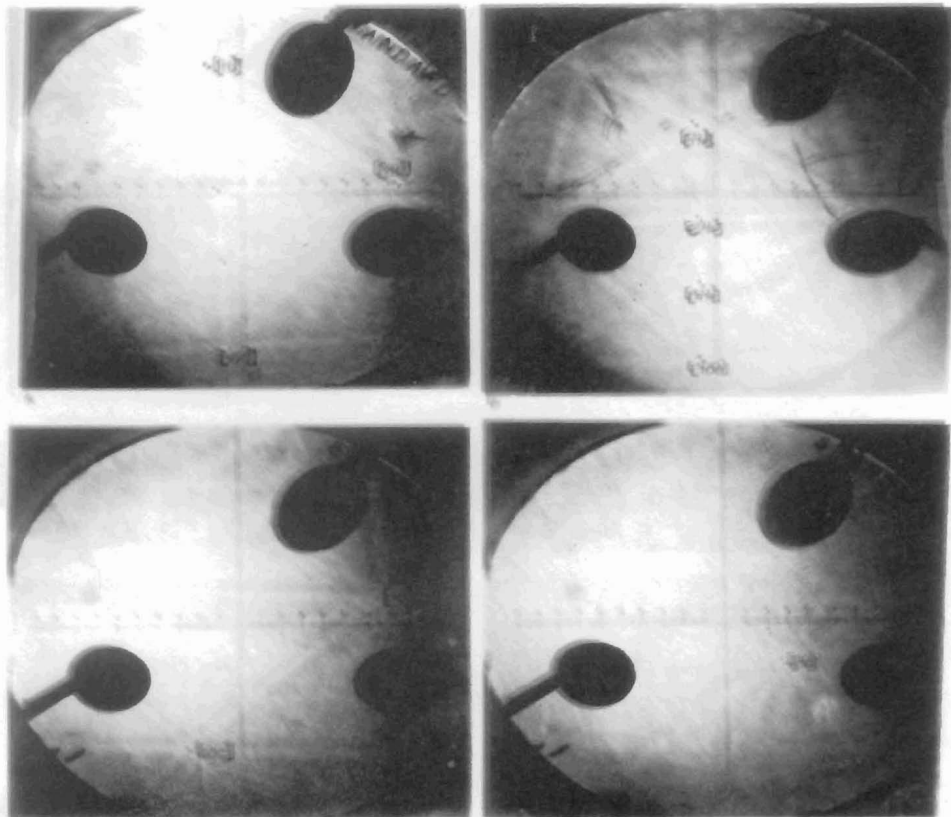


Figure 13: Electron Backscattering Patterns (EBSP) from (a) clean Si(100) surface and (b) – (d) Ag Islands in parallel Epitaxy with Si(100)Substrate.

Keep it Simple

In any complex technology, the simpler it can be designed and installed the better. However, simplicity requires knowledge. The first set of astronauts sent to space by NASA discovered that ballpoint pens would not work in zero gravity. In order to combat the problem, NASA scientists spent a decade and millions of dollars to develop a pen that would write in zero gravity, upside down, under water, on almost any surface, and at temperatures ranging from below freezing to 300 °C. The Russians simply used pencils!

My time in the University of Sussex, Brighton, UK, with the Surface Physics group of Professor John A. Venables,

afforded me the opportunity of using the University of Sussex modified HB50 Ultrahigh Vacuum Scanning Electron Microscope (UHV- SEM). There was no facility to continue the line of research in Nigeria. However, by divine providence, Professor E.O.B. Ajayi pioneered a research group that provided a simple, novel and cost-effective Metal Organic Chemical Vapour Deposition system (MOCVD) for the deposition of many materials of interest to an electronic engineer for the purpose of device fabrication (Ajayi *et al.*, 1981). The typical system is as shown in Figure 14. An improved version has been built with patent pending by Prof. E.O.B. Ajayi, in 2010. The system is a solid source delivery one with nitrogen, oxygen, or compressed air as the carrier gas and the precursor in a powdered form. Depositions were done under inert conditions as opposed to the ultra-high vacuum system I used in Sussex and many high temperature materials were deposited at relatively low

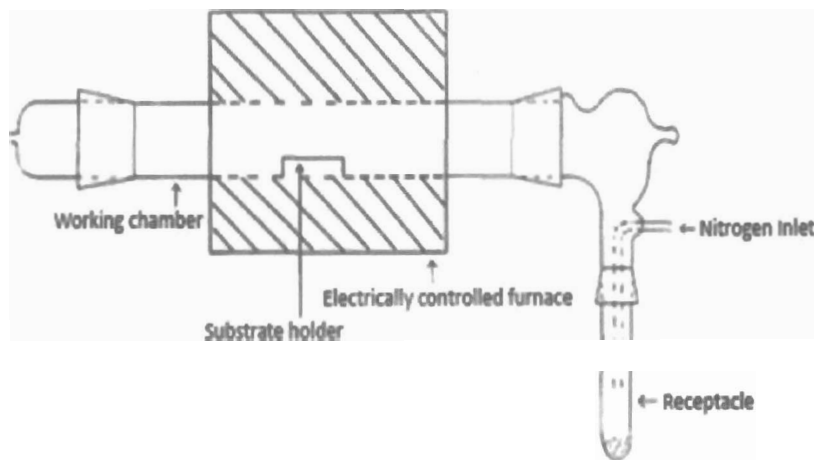


Figure 14: The basic setup of the Metal Organic Chemical Vapour Deposition (MOCVD) System.

The precursors are easily synthesized from metal oxide acetylacetonates (e.g. $\text{MoO}_2(\text{C}_5\text{H}_7\text{O}_2)_2$) and readily decompose at relatively low temperatures. The precursors are kept in a receptacle and nitrogen gas is passed through at a flow rate of $2.5 \text{ dm}^3 \cdot \text{min}^{-1}$. A temperature profile of $320 \text{ }^\circ\text{C}$ to $450 \text{ }^\circ\text{C}$ is typical. The substrate is soda-lime glass and a deposition period of 2 hours is typical.

Many metal oxide thin films have been deposited and characterized using this technique. Thin solid films deposited include but not limited to the followings: CuO , $\text{CuO}/\text{Al}_2\text{O}_3$, In_2O_3 (Ajayi *et al.*, 1986); Cr_2O_3 , Cu_2O (Ajayi *et al.*, 1990); Co-V-O , Cr-V-O (Adedeji *et al.*, 2002); ZnO , $\text{ZnO}:\text{U}$ (Elerujah *et al.*, 2002); LiCoO , LiNiO , NaCoO , NaNiO (Eleruja *et al.*, 1998); Indium Tin Oxide (ITO) (Akinwunmi *et al.*, 1999); V_2O_3 (Ajayi *et al.*, 1981); $\text{Na}_{0.33}\text{V}_2\text{O}_5$ (Ajayi *et al.*, 1983); MoO (Ilori *et al.*, 2005); and Li-Mo-O (Ilori *et al.*, 2005); PbS and PbCdS (Omotosho *et al.*, 2013); ZnS (Osasona *et al.*, 1997); $\text{Zn}_x\text{Cd}_{1-x}\text{S}$ (Eleruja *et al.*, 1998); nickel oxide and lithium nickel oxide (Eleruja *et al.*, 2007); Molybdenum Oxysulphide (Olofijana *et al.*, 2010); and CdO (Adekoya *et al.*, 1998).

Oxides of transition metals such as M_2O_3 (M represents transition metals) and V_2O_5 catalyse the oxidation of hydrocarbons as well as the dehydrogenation of alcohols. These features make metal oxides potentially suitable as gas sensing materials (for example MoO_3 films are used as sensors for gases like H_2 , NH_3 , CO , NO and NO_2) Sputtered MoO_3 thin films have whisker-like structures. High surface to volume ratio is an advantage. It is also known to be highly sensitive to these gases (Feroni, 1997; Di Giulio, 1998 and Imawan, 1999).

This simple system became the major system used for so many of our publications in international journals. Two of them particularly deserve mention. The prevailing slang here in Nigeria is "publish or perish", with the emphasis on the number, rather than the scientific content. If this had not been so, these works would not have been published, and they would have fetched the University some money by way of patent and

intellectual property. One of the papers formed the basis of the Molybdenum Oxide materials being marketed by a company called "LookChem", with branches in the USA, China Russia and India. The first ever reference to their work was this paper. (Ilori *et al.*, 2005). The second paper (Ajayi *et al.*, 1986) was the basis for the Patent EP0526994A1, in 1993, for Ford Motor Company Limited in the United Kingdom.

The major challenge faced by the group is to fabricate devices from these thin films prepared by us from this simple MOCVD equipment, so that we do not end up labouring for others to reap the benefits. The Ph.D. thesis of Mr. O.O. Ilori (Ilori, 2015), just defended yesterday (and jointly supervised by Professors G.A. Adegboyega and E.O.B Ajayi) is a positive step in this direction. His work is on the fabrication and characterization of MOCVD transparent thin film transistor. Another Ph.D. student of mine is working on the use of this MOCVD to fabricate and characterize an intelligent gas sensor. A locally configured MOCVD system has thus gained international recognition.

OAU iLabs

OAU iLabs was born out of the desire to allow our undergraduate students participate in the characterization of semiconductor devices using state-of-the-art equipment that we could not afford because of dwindling financial support to the university system by the government. These experiments are, nevertheless necessary for their training. An alumnus of OAU in the Department of Electrical Engineering and Computer Science, Massachusetts Institute of Technology (MIT) - Professor Tayo Akinwande - was contacted. He in turn worked through Professor Jesus Del Alamo of the same Department and the project was supported by the Carnegie Corporation of New York. In collaboration with MIT, the University of Dar es Salaam, Makerere University, Kampala, Obafemi Awolowo University became the West African hub of the project. It expanded to include sharing commons with other universities during the time of the first Principal Investigator, Professor L.O. Kehinde.

The OAU team made significant contributions to the MIT Batched Architecture in use then, and many journal papers were published, including a Chapter contribution to a book (Ayodele et al, 2008; Jonah et al, 2008; Olowokere et al, 2008; Akinwale et al, 2011a, Akinwale et al, 2011b; Kehinde et al., 2011 and Zubia and Alves. (Eds) ,2011). I succeeded Professor Kehinde (as the second Principal Investigator, since 2008).

One unintended (but, nonetheless, significant) outcome of the iLabs project is the emergence of a vibrant research and development community within the ranks of undergraduate students of the Department. Working out of the now famous “*iLabs Room*”, students of the Department have participated in three World Robotics Olympiads, with the most recent event coming up just two weeks ago. Another group of iLabs students developed a hand-held device that allows visually-challenged individuals to operate mobile phones, and was widely featured on international Cable Channels. Student research on iLabs has, however, not been limited to the undergraduate level, as two graduate students (one Ph.D. and one M.Sc.) supervised by Prof. L.O. Kehinde successfully defended theses on iLabs-related topics in 2012.

This concept of the iLabs was shared with the Physics Department of the University of Sussex, during my seminar in 2010 (Osasona, 2010) and it proffered a solution to the problem of data linkage between their Laboratories in France and Sussex.

Conclusion

Mr. Vice-Chancellor sir, my research work has contributed immensely to the resolution of the problem of device degradation arising from improper alloying of contacts to devices. It has also gone ahead to contribute to the finding of new metallurgical schemes for device metallization.

It has been demonstrated during the course of this lecture, that electronic devices have affected our lives both positively and negatively. However, the positive impacts far outweigh the negative ones. It has equally been demonstrated that meaningful multi-disciplinary research can be carried out locally. Our collective resolve needs to be strengthened by the university authorities in providing an enabling research environment, through encouraging more multi-disciplinary groups to be set up and financed. The number-game for promotion and the single-authorship stance need to be reviewed in order to promote meaningful and useful scientific outputs. The **impact**—not the number-of publications must be brought to the fore.

The dwindling financial support to the universities by the government and the undue interference with the University Research and Development Centres, have not helped the university system – at all. The MOU setting up Centres like the Centre for Energy Research and Development (CERD), and the National Centre for Technology Management (NACETEM), among others, should be returned to, in good faith.

Finally, in the course of the recent review of our curriculum, certain facts emerged. If we are to maintain our leading role in ICT competence (Ogunfunmi, 2009), our undergraduate students must be trained with more fundamental science course contents: more Physics courses for the Electronic and Electrical Engineering students; more acoustic-related courses in both Departments of Architecture and Music. These courses will provide a sound foundation for their continued excellent performance.

I thank you all for listening.

References

- Adedeji A., Egharevba G., Jeynes C. and Ajayi E. (2002): "Preparation and characterization of pyrolytically deposited (Co–V–O and Cr–V–O) thin films," *Thin Solid Films*, vol. 402, no. 1–2, pp. 49-54.
- Adegboyega G. A. (2000): "*Before the Dawn of Molecular Electronics*", Inaugural Lecture Series 142, Obafemi Awolowo University, Ile-Ife, Nigeria.
- Adekoya S. O., Eleruja M. A., Olofinjana B. and Akinwunmi O. O. (2012): "Enhanced Light Absorption in Textured Metal Organic Chemical Vapour Deposited (MOCVD) CdO Thin Films," *MRS Proceedings*, vol. 1432, pp. 12–14.
- Adesina T. O., **Osasona O.**, Ajayi E. O. B. and Okon E. E. (2015): "**Solving the Long Range Wildlife Migration Tracking Challenge: A Low Cost Wearable Satellite Transmitter Approach.**" Submitted for publication.
- Ajayi O., Anani A. and Obabueki. (1981): "Characterization of pyrolytically deposited V_2O_3 thin films," *Thin Solid Films*, vol. 82, no. 2, pp. 151–156.
- Ajayi O., Taiwo A. and E. Ibanga. (1983): "Electrical conductivity of $\beta\text{-Na}_{0.33}\text{V}_2\text{O}_5$ thin films," *Thin solid Films*, vol. 105, no. 2, pp. 97–101.
- Ajayi, O.B., Akanni, M.S., Lambi, J., **Osasona O.**, Podor B., and Burrows H.D.: (1986) "Preparation and Optical Characterization of Pyrolytically deposited thin films of some metal oxides" *Thin Solid Films*, vol. 138, pp 133 – 141.
- Ajayi O., Akanni M., LambiJ., BurrowsH., **Osasona O.** and Podor B.(1986): "Preparation and optical

- characterization of pyrolytically deposited thin films of some metal oxides," *Thin Solid Films*, vol. 138, no. 1, pp. 91–95.
- Ajayi O., Akanni M., LambiJ., Jeynes C. and Watts J. (1990): "Compositional studies of various metal oxide coatings on glass," *Thin Solid Films*, vol. 185, no. 1, pp. 123–136.
- Akinwale, O.B., Ayodele K.P., Jubril A.M., Kehinde, L.O. **Osasona, O.**, Akinwunmi O., Asiimwe, A.T. Mwikirize, C., Musasizi, P.I., Tickodri Togboa, S, Katumba, A. Butime, J, Nombo, J.P., Baraka, M.M., Teyana, S., Mwambela, J.A and Kissaka. M.M. (2011b): "Online Laboratories: Enhancing the Quality of Higher Education in Africa" *Proceedings of Conference of Rectors, Vice Chancellors and Presidents of African Universities, COREVIP 2011*, Stellenbosch, South Africa.
- Akinwale, O.B., Ayodele, K.P., Kehinde, L.O. and **Osasona, O.** (2011a): "Implementing Remote Laboratories with the iLab Architecture: Three Case Studies from Obafemi Awolowo University, Nigeria", *Computers in Education Journal*. ELSEVIER, ISSN: 0360-1315.
- Akinwunmi O., Eleruja M., Olowolafe J., Adegboyega G. and Ajayi E. (1999): "Preparation and characterization of MOCVD thin films of indium tin oxide," *Optical Materials*, vol. 13, no. 2, pp. 255-259
- Ayodele K.P., Kehinde L.O., Jonah O.P., Ilori O.O., **Osasona O.** and Ajayi E.O.B.; (2008) "**Development Of An Operational Amplifier Virtual Laboratory Based On iLab Architecture And Ni Elvis**" *Proc. of the 2008 ASEE Annual Conference & Exposition*, paper

AC2008-1098; June 22 - 25 , - Pittsburgh, PA . USA.
(Nominated for best paper)

Batchen G. (1997): *Burning with Desire: The Conception of Photography*, MIT Press, Cambridge, 1997

Boutaoui N., Roux H., Tholomier M (1990): Ag mass transport on Si(111) in the 350 – 450 °C temperature range; *Surface Science* vol. 239, no. 3, pp213-221.

Chen M. A. (2014): *Heart Pace Maker – Discharge*, U.S. National Library of Medicine, Available Online: <https://www.nlm.nih.gov/medlineplus/ency/patientinstructions/000097.htm>, Accessed on 10th November, 2015.

Campbell R., Christopher R. and Bettina F. (2000): “Sounds and Images, Media and Culture: An Introduction to Mass Communication, Boston, Bedford/St. Martin's;vol. 113, additional Text)

Egbujor, I. I., **Osasona, O.** and Adewunmi, C. O. (2003): A Review of problems of Purchasing, Servicing and Maintenance of Scientific Equipment in the Nigerian Universities. Workshop Report, *International Workshop on Purchasing, Servicing and Maintenance of Scientific Research Equipment in West Africa*, University of Buea, Cameroon, November, 5–8, 2002, pp. 67 - 73.

Eleruja M., Adedeji A., Ojo I., Djebah A., **Osasona O.**, Aladekomo J. and Ajayi E.(1998): “Optical characterization of pyrolytically deposited $Zn_xCd_{1-x}S$ thin films,” *Optical Materials*, vol. 10, no. 4, pp. 257–263.

Eleruja M., Adedeji A., Egharevba G., Lambi J., Akanni M., Jeynes C. and Ajayi E. (2002): "Preparation and characterization of undoped zinc oxide and uranium doped zinc oxide thin films," *Optical Materials*, vol. 20, no. 2, pp. 119-123.

Eleruja M., Egharevba G., Abulude O., Akinwunmi O., Jeynes C. and Ajayi E. (2007): "Preparation and characterization of metalorganic chemical vapor deposited nickel oxide and lithium nickel oxide thin films," *Journal of materials science*, vol. 42, no. 8, pp. 2758–2765.

Freeouf J.F., Rubloff G.W., Ho P.S. and Kuan T.S. (1979): "Microscopic compound Formation at the Pd₂Si-Si(111) Interface". *Physical Review Letters* vol. 43, pp 1836 – 1839.

Freeouf J.F., Rubloff G.W., Ho P.S. and Kuan T.S. (1980): "Relative Schottky barrier formation: The Pd/Si interface". *Journal of Vacuum Science and Technology*, vol. 17, no. 5, pp 916 – 919.

Hanbucken M., Doust T., Osasona O., Le Lay, G. and Venables J.A. (1986): SEM Observations of Ag Surface diffusion at the Si(111) $\sqrt{3}$ -Ag Interface. *Surface Science*, vol. 168, pp 133 – 141.

Ilori O.O. (2015): "*Fabrication and Characterization of Metal Organic Chemical Vapour Deposited Transparent Thin Film Transistor*" Ph.D. Thesis, Obafemi Awolowo University, Ile-Ife, Nigeria, 224 pages.

Ilori O. O., Osasona O., Eleruja M. A., Egharevba G. O., Adegboyega, G. A., Chiodelli, G., Boudreault, G., Jeynes, C. and Ajayi, E.O.B. (2005): Preparation and Characterization of Metallorganic Chemical Vapour

Deposited Molybdenum (II) Oxide (MoO) Thin Films, *Thin Solid Films* vol. 472 , no. 1-2, pp 84 – 89.

Ilori O., **Osasona O.**, Eleruja M., Boudreault G., Jeynes C. and Ajayi E. (2005): "Preparation and characterization of metallorganic chemical vapour deposited LixMoyOz using a single source solid precursor," *ionics*, vol. 11, no. 5–6, pp. 387–392.

Joachim N. B. (2013), *Guide to State-of-the-Art Electron Devices*, First Edition. John Wiley & Sons Ltd, New Jersey.

Jonah O.P., Ayodele K.P., Kehinde L.O. **Osasona O.** and Ajayi E.O.B.(2008): Comparison between a Web-based Laboratory (WBL) and Conventional Laboratory Models. *Journal of Computer Science and Its Application (Nigeria)*. **Vol. 15, no. 1**, pp 124 - 135.

Kehinde L.O., Ayodele K.P., Akinwale O.B. and **Osasona O.**(2011): "Using Remote Labs in Education: The Obafemi Awolowo University Experience", In, J. Gracia Zubia and G.R. Alves. (Eds). *Using Remote Labs in Education: Two Little Ducks in Remote Experimentation* (Chapter 3 pp. 81 – 111). University of Deusto Publishers, ISBN 978-84-9830-398-8.

Kilby J.S. (1976): Invention of the Integrated Circuit. *IEEE Trans. Elec. Dev.* vol. 23, pp. 648.

Kuku T.A. (2006): "*Materials, The Basis of the Beauty of Modern Day Electronic Systems*", Inaugural Lecture Series 192, Obafemi Awolowo University, Ile-Ife, Nigeria, June 13, 2006.

Morris, H. M. (1984). *A history of modern creationism*. Master Book Publishers San Diego, California.

- Morris H.M. (1993): *History of Modern Creationism*: Santee CA: Institute for Creation Research, pp 68-69
- National Institute of Health (2013),"Cochlear Implants", Publication No. 11-4798
- OAU Ilabs, Obafemi Awolowo University, Ile-Ife, Nigeria, March 2011.
- Ogunfunmi Tokunbo (2009): "New Curriculum Development for a top African University"**, American Society for Engineering Education, Paper presented at 2009 Annual Conference & Exposition, Austin, Texas. <https://peer.asee.org/5843>
- Olofinjana B., Egharevba G., Eleruja M., Jeynes C., Adedeji A., Akinwunmi O., Taleatu M., Mordi C. and Ajayi E. (2010): "Synthesis and Some Properties of Metal Organic Chemical Vapour Deposited Molybdenum Oxysulphide Thin Films," *Journal of Materials Science & Technology*, vol. 26, no. 6, pp. 552–557,.
- Olowolafe J.O., Nicolet, M.A. and Mayer J.W. (1976a): "Formation Kinetics of CrSi₂ films on Si substrates with and without interposed Pd₂Si layer". *Journal of Applied Physics*, vol. 47, pp 5182 – 5186.
- Olowolafe J.O., Nicolet, M.A. and Mayer J.W. (1976b): "Influence of the Nature of the Si substrate on Nickel Silicide formed from thin Ni Films", *Thin Solid Films* vol. 38, pp 143 -150.
- Olowokere O., Jonah O.P., Kehinde L.O., Ayodele K.P, **Osasona O.** and Ajayi E.O.B.; (2008): " Human And Infrastructure Issues In The Development of Web-Based Laboratories In Challenged Environments"

Proc. of the 2008 ASEE Annual Conference & Exposition, Paper AC2008-1375; June 22 - 25 , -Pittsburgh, PA.

Omotosho E., Adegboyega G.A, Eleruja M., Olofinjana M., Ilori O., Akinwunmi O., Taleatu B. and Ajayi E.O.B. (2013): "Synthesis and Some Properties of MOCVD Lead Sulphide and Lead Cadmium Sulphide Thin Film," *Journal of Non-Oxide Glasses*, vol. 5, no. 1, pp. 9–19.

Oluwasanmi H.A. (1971): "Address of the Vice-Chancellor, Professor H.A. Oluwasanmi to the University of Ife Convocation; 3rd July, 1971, pp.5.

Osasona O. (1981): "*Characterization of a Reflection High Energy Electron Diffraction (RHEED) System*" M.Phil Thesis, University of Ife, Ile-Ife, Nigeria.

Osasona, O. and Williams, V. A. (1983): "In-situ Observations on the growth of ZnS on Silicon(111) using RHEED", *Nigerian Journal of Science*, vol. 17, pp 133– 142

Osasona O (1988): Development and Stability Studies of Silicon Planar Devices Ph.D. Thesis Obafemi Awolowo University, Nigeria.

Osasona O., Djebah A., Ojo I., Eleruja M., Adedeji A., Jeynes C. and Ajayi E. (1997): "Preparation and characterization of MOCVD thin films of zinc sulphide," *Optical Materials*, vol. 7, no. 3, pp. 109–115.

Osasona O. (2002): "AES and SEM Analyses of a Transistor Developed at Ife". *Nigerian Journal of Engineering Research and Development (NJERD)* vol. 1, no.4, pp 47 – 53.

- Osasona O.** and Adegboyega, G.A. (2003): "Silicon Planar Devices Fabrication at Ife". *Nigerian Journal of Engineering Research and Development (NJERD)* vol. 2, no. 3, pp 39 – 47.
- Osasona O.** (2010): "25 years After Sussex: Some Reflections on Science and Engineering from Obafemi Awolowo University, Ile-Ife, Nigeria" Presented at the Special Seminar organized by the Department of Physics, Pevensey 1, Room 2A3, University of Sussex, Falmer, Brighton, UK, Wednesday, September 1.
- Oustry A., Berty J., Caumont M., David M.J. and Escout A. (1982): "Structural Properties of the Pd-Si interface: An investigation by Reflection High Energy Electron Diffraction". *Thin Solid Films*, vol. 97, pp 295 – 300.
- Poate J.M., Tu K.N., Mayer J.W. (1978): *Thin Films- Interdiffusion and Reactions*, John Wiley and Sons, New York (1978), pp 399-403
- Plummer, J. D. (2000). *Silicon VLSI technology: fundamentals, practice, and modeling*. Pearson Education, India.
- Rubloff G.W., Ho P.S., Freeouf J.F. and Lewis J.W. (1981): "Chemical bonding and reactions at the Pd/Si interface". *Physical Review B* vol. 23, no. 8, pp 4183 – 4196.
- Shockley W. (1984): The path to the conception of the Junction Transistor; *IEEE Trans. Elec. Dev.*, vol. 31, pp. 15-23.

- Warwick K., Gasson M., Hutt B. and Goodhew I. (2005): "An Attempt to Extend Human Sensory Capabilities by Means of Implant Technology" *2005 IEEE International Conference on Systems, Man and Cybernetics*, vol. 2, pp 1663-1668, 10 -12 October, 2005.
- Williams, V.A. (1966): "High Conductivity Transparent Contacts to ZnS" *Journal of the Electrochemical Society*, vol. 113, no. 3, pp 234 -237
- Williams, V.A., Aliu S.O., Osasona, O. and Kuku T. A. (1977): "Preparation of Silicon from Igbokoda Sand", Internal Report, Semiconductor Laboratory, University of Ife, Nigeria.
- Williams, V.A., Szolgyemy, L., Adegboyega, G.A., and **Osasona, O.**: (1984) "Progress in Electronics Technology Transfer at the University of Ife: Resistor Fabricating Technology" *The Nigerian Engineer*, vol. 19, no. 2, pp 41 – 48.
- Venables J.A. (1976): "An UHV-SEM for In-situ Deposition and Surface Studies". *Proc. Of EMAG '75. Institute of Physics Conference Series* no. 68, Academic Press, London, pp. 23 -26.
- Venables J.A., Janssen A.P., Harland C.J. and Joyce B.A. (1976): "Scanning Auger Electron Microscopy at 30 nm resolution". *Philosophical Magazine* vol. 34, no. 3, pp 495 – 500.
- Venables J.A. and bin-Jaya R. (1977): "Accurate Micro crystallography using Electron Backscattering Patterns". *Philosophical Magazine* vol. 35, pp 1317-1332.

Venables J.A. and Archer G.D, (1980): "On the Design of FG-SEM Columns"; *Electron Microscopy I*, vol. 54.

Venables J.A.; Spiller G.D.T, Fathers D.J., Harland C.J. and Hanbucken M. (1983a): "UHV-SEM Studies of Surface Processes": *Recent Progress: Ultramicroscopy* vol. 11, 149 – 156.

Venables J.A., Janssen A.P., Akhter P., Derrien J and Harland C.J. (1983b): "Surface Studies in UHV Field Emission Gun Scanning Electron Microscopy": *Journal of Microscopy*, France, vol.118, pp 351 – 365.

Yahoo News UK (2015): "SUVs Stolen In The UK Are Tracked Down By Police... In Uganda" n.d.. <https://uk.news.yahoo.com/suvs-stolen-uk-tracked-down-110222271.html> [accessed 29th October, 2015].

Patents

Patent EP0526994A1- A method of improving the pyrolytic ...
www.google.com/patents/EP0526994A1?cl=en

Feb 10, 1993 - A pyrolytic deposition process for forming a *metal oxide* on a hot glass ... 1, February 1990, LAUSANNE CH pages 123 - 136 O.B.AJAYI ETAL. Cited on 9th Nov. 2015.

Patent EP0526994A1- A method of improving the pyrolytic ...
www.google.com/patents/EP0526994A1?cl=en

Feb 10, 1993 - A pyrolytic deposition process for forming a *metal oxide* on a hot glass ... 1, February 1990, LAUSANNE CH pages 123 - 136 O.B.AJAYI ETAL. Cited on 9th Nov. 2015.

US Patent 841387 De Forest, Lee, "Device for Amplifying Feeble Electrical Currents", issued 15 January, 1907.