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Master's Degree Thesis

ELECTRICAL SPM CHARACTERIZATION AND ANALYSIS OF IGZO FOR LOGIC AND MEMORY APPLICATIONS

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Summary

InGaZnO (IGZO) is a quaternary, n-type semiconducting oxide under extensive study due to its promising mechanical and electrical properties: initially discovered and studied in crystalline form as Thin Film Transistor (TFT) channel material, more recently its amorphous phase has become a good candidate for a wider spectrum of applications ranging from BEOL logic - due to its low thermal budget - to selectors for emerging memory architectures.

However, this oxide brings of course new kind of challenges together with its good properties: in particular, the main ones are the film quality dependence on both stoichiometry and process parameters, which in turn makes the definition of standard process recipes a very difficult task, and the expensive testing/debugging step, which can only be performed with fully developed devices and not during early stages. In this framework, we explored a particular Electrical Scanning Probe Microscopy technique, the Conductive-Atomic Force Microscopy, as a possible tool able to address and solve these problems: in fact, the capability of this technique to measure electrical properties of insulating thin films with a nanometric lateral resolution is at least in theory the best one to screen, understand and compare different types of IGZO films (and so to correlate with deposition and process parameter) in blanket form (and so without the need of all the expensive steps required from full device development).

This work reports the methodology and the results, obtained during the six month long internship in the R&D centre imec BE, that the proposed solution based on C-AFM allowed to gather from a number of IGZO blanket sample, differing in terms of both phase, deposition technique, process parameters and target application; in particular, it has been demonstrated that this ESPM tool is capable to obtain several figures of merit, useful for any systematic study of this material, and to give results in good agreement with the device testing.

In this way, we are able to propose a procedure for early stage screening of IGZO blankets, which may be used for a wider spectrum of materials in future works.

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Acronyms

ALD

Atomic Layer Deposition

AOS

Amorphous Oxide Semiconductor

\mathbf{BE}

Bottom Electrode

C-AFM

Conductive Atomic Force Microscopy

\mathbf{DFT}

Density Functional Theory

\mathbf{DT}

Direct Tunneling

\mathbf{FNT}

Fowler-Nordheim Tunneling

I-V

Current-Voltage characteristic

\mathbf{MIM}

 $Metal \hbox{-} Insulator \hbox{-} Metal$

PLD

Pulsed Laser Deposition

\mathbf{SPM}

Scanning Probe Microscopy

TAT

Trap Assisted Tunneling

\mathbf{TE}

Top Electrode

TFT

Thin Film Transistor

Chapter 1

InGaZnO, a n-type semiconducting oxide

1.1 The class of Amorphous oxide semiconductors materials

Amorphous Oxides Semiconductors (AOS) are a class of ternary/quaternary oxides of post-transition metals that are attracting large attention because of a number of advantages and improvements that they feature compared with their crystalline counterparts; they have been studied extensively in the past decades especially from a theoretical point of view, a research that lead to more and more understanding of their general properties.

In fact, results showed that these materials possess a lot of interesting properties, both physical and technological (Medvedeva et al., 2017):

- Quite high mobilities with respect to other established amorphous materials, such as a-SI:H;
- High band gap, which makes them natural materials for display applications;
- Tunable electrical properties due to their high sensitivity to process parameters and stoichiometry;
- Low temperature large area deposition makes them good candidates for glass deposition;
- Mechanical flexibility for flexible electronics;

In particular their high variability is what makes this material class both interesting and challenging.

Some understanding have been achieved, especially based on Density Functional Theory calculations: for example the origin of higher mobility with respect to crystalline counterpart is given by the different physical phenomena that generates it, related no more to phonon and impurity scattering but instead to the long-range disorder and metal-oxygen distortion; in addiction, their electrical stability is what makes them an improvement with respect to others amorphous materials, especially when used as thin film transistor channel.

However, the intrinsic complexity of having three or more elements in amorphous phase, the oxygen vacancies and hydrogen infiltration influence, the deposition techniques and parameters adopted makes a systematic study and organization hard to perform; in this sense, while mentioned DFT calculation allow to understand some correlation between composition and performance, it is much harder to actually deposit the material in a controlled way so that the best result can be obtained.

Among others, the quaternary InGaZnO has recently gained a lot of attention: initially discovered for display application, it later turned in a promising in a wider range of application; it however still need a lot of work for the complete understanding of its properties, and this work aimed to explore techniques able to give more insight and methodology in this sense.

1.2 Main properties of IGZO

Belonging to the AOS material class IGZO features most of the previously described feature, such as the high and tunable conductivity - with respect to other amorphous materials like a-Si - flexibility and low thermal budget for deposition.

A first report on its use is given by K. et al., 2004 for thin film transistor, and it was demonstrated to overcome the limitation of others amorphous material:

- higher mobilities (with values easily higher than $10cm^2V^{-1}s^{-1}$): this has been explained later in terms of the different chemical bonding present, which in IGZO is given by the s-type orbitals of the metals and thus the disorder is not very effective (Kamiya et al., 2010);
- the increased electrical stability (which in turn greatly improve durability): this is found because while in a-SI grain boundaries are an issue, in IGZO they are much less present due to the highly increased uniformity;
- it features good electrical behaviour even when mechanical stress is applied,

thus being attractive for flexible electronics;

• its high bandgap ($\sim 3.2eV$) makes it ideal for display and transparent electronic;

There are also other reasons why a quaternary material has been so much considered, and not other known candidates as ITO or GZO: as shown in fig. 1.1b, all its components gives contribution in different ways, from the electrical to mechanical one.



(Kamiya et al., 2010)

(a) Theoretical band diagram of IGZO (b) Each element of IGZO carries a number of properties, and this is both an opportunity and a challenge

1.2.1**Deposition techniques**

For what concerns the deposition techniques, two are currently the main one employed for IGZO film growth (and all the sample characterized in this work are based on these), depending on the film requirements:

- Atomic Layer Deposition (ALD);
- Pulsed Laser Deposition (PVD).

ALD is a process developed to achieve an optimal large area uniformity: this is achieved by means of a series of self-limiting reactions taking place at the interface between the substrate and the precursors in vapor phase; in this way a very fine control of the smoothness, uniformity and composition is achieved.

In the case of IGZO (but also for other AOS), a reported workflow includes the use of tri-methyl Indium, tri-methyl Gallium and diethyl Zinc as metal and water as oxygen precursor (Sheng et al., 2018); the final composition will depends of course on the amount of each precursor, but also on other process parameters, in particular the temperature.

Pulsed Laser Deposition is another important technique used to grow IGZO films (especially on glass substrate, due to the low temperature process): it is based on a high power, pulsed laser beam that is directed in the (vacuum or gas-filled) chamber toward the target material which in turn vaporize and deposits on the substrate.

In the case of IGZO deposition, a common procedure consists firstly on the preparation of the target IGZO, in turn obtained by sintering In_2O_3 , Ga_2O_3 and ZnO powders, and then on the true deposition by laser vaporization (Lee and Dho, 2011). Again, the final composition will strongly depend both on the target composition ratio, the laser power and the process temperature.

In any case, both the technique are established, but the final results (electrical and mechanical properties, film quality and phase) depends on a wide range of process and target parameters, so a systematic analysis of a wide number of different samples can help in understanding these dependencies and to adress future research.

1.2.2 Crystalline and amorphous phases

Depending of deposition process and parameters, target properties and substrate condition, IGZO films can be grown so that both amorphous and crystalline phases can be obtained.

Spinel and CAAC are two different crystalline phases of IGZO, differing from the type of order among all the metallic compounds.

In fig. 1.2 a representation of both crystalline forms is shown: CAAC is characterized by a lack of ordered structure along the (x,y) plane perpendicular to the C axis, that instead show a layered structure on In and GZO planes; Spinel in turn is a more "exotic" polycrystalline phase, which structure is usually cubic close-packed, featuring an intermixing of GZO with In atoms.

They are widely studied in several electronic fields, for example:

- As channel material for TFT, because of the outstandingly low OFF current (Sekine et al., 2011) while maintaining an appreciable carrier mobility;
- More recently as main elements of LCD and OLED display, because of the high band gap and so high transparency in visible spectrum;

- As promising materials for BEOL logic systems, due to the low thermal budget for deposition (Kaneko et al., 2011);
- As new candidates for AI devices, again for the very low power consumption (Yamazaki et al., 2019).



(a) Spinel phase

(b) CAAC phase

Figure 1.2: Crystal configuration of the two main IGZO phases

The amorphous one is the other main IGZO phase, characterized by a complete absence of long range lattice structure (e.g. see fig. 1.3); its electrical properties are not very different from the crystalline phase, for example the electron effective mass and Hall mobilities are similar (Kamiya et al., 2010), however it is important because of the capability to deposit very thin layers (in the following sections, a-IGZO films down to 2 nm are studied) over large areas and maintaining its properties.

1.3 Conduction phenomena in IGZO

The lack of a extended crystal lattice is what makes the conduction of IGZO promising in application where typical silicon technology is not available: in fact, the combination of 3 different metal ions, the oxygen and its vacancies and the phase itself cause the electrons to move quite freely inside the material; on the other hand, depending also on the application the carriers motion can take place both on the surface and in the bulk of the material: in each case, the nature of the electrical behaviour is different. The next sections will focus on these changes.



Figure 1.3: Amorphous IGZO phase representation

1.3.1 Percolative and multiple trap and release conduction

Conduction involving the surface or thick IGZO films has been explained in literature through two different models, the Percolative and Multiple trap and release (MTR) ones.

Percolative motion of electrons is the process that explains a counterintuitive property of IGZO, namely the increase of mobility caused by the increase of free electron density (something that does not happen in typical semiconductors due to increased scattering): in particular, the origin of this behaviour is given by the disordered structure of IGZO, which in turn cause potential barrier distribution to be non uniform (right side of fig. 1.4); in this way, depending on the temperature electrons may or may not be able to overcome local high barriers, and in the second case in particular they will take longer paths across low barriers regions (Kamiya et al., 2010).

Multiple Trap and Release model instead is based on the presence of tail states below the conduction band extended ones(left side of fig. 1.4), that in turn do not contribute on the current (Kamiya et al., 2010); in this way, it turns out that any (gate) voltage-induced amount of charge will feature a portion that is trapped in these states, and only the other portion will end in conduction band.

Recent works (Bhoolokam et al., 2016) show that these models taken separately does not explain completely the TFT characteristics in different conditions, while their combination succeeds in this task; however, despite the fundamental origin of conduction is now known, less information are available for what concerns the dependence on film composition and deposition of the physical quantities that



Figure 1.4: Schematics on the origin of MTR and Percolative models

determine these electrical properties.

1.3.2 Leakage in thin films

In case of thin films, the conduction processes are quite different even though still related, of course, to band concepts; in general, when physical dimensions becomes very small, a number of new effects begin to be important and influence the electrical behaviour of a device. For what concerns this work, two main categories of leakage processes have been considered:

- Field emissions
- Tunnel effects

The most relevant field- related leakage is given by the Thermo-ionic emission (described by Richardson in a paper from 1901): it describes the emission of electron from the material potential barrier (the workfunction ϕ_M), as a process that is temperature dependent.

The origin of this phenomenon is found in the statistical distribution of velocities in the "sea of electrons" inside a metal, that in turn may cause an electron to have enough energy to overcome the workfunction barrier; however, Richardson discovered that the current emitted from a metal featured actually an exponential dependence on the temperature, i.e. the current from a heated material was higher. In particular, he proposed as equation:

$$J = A_G T^2 \exp\left(-\frac{\phi_M}{k_B T}\right) \tag{1.1}$$

with K_B the Boltzmann constant, T the (absolute) temperature and A_G the Richardson constant (Zeghbroeck, 2013):

$$A_G = \frac{4\pi q m_{ox} k_B^2}{h^3} (IGZO :\sim 4.0859e5)$$
(1.2)

This expression is in turn the starting point to obtain a similar expression, the Schottky emission law, fundamental when dealing with material junctions such as Metal-Insulator: it arises when considering the barrier lowering caused by an external electric field and the fact that the barrier becomes a Schottky barrier, given by $\phi_{barr} = \phi_M - \chi_{semicond}$ (Schottky-Mott rule). The latter expresses the increase in emitted current caused by a reduction of the potential due to the band bending:

$$J = A_G T^2 \exp\left(\frac{\phi_{barr} - \sqrt{\frac{q^3 F}{4\pi\epsilon_0}}}{k_B T}\right)$$
(1.3)

where F is the electric field; the effect of a bias is a barrier lowering of the factor under the square root.

A second field-related mechanism is given by the Frenkel-Poole effect, which in turn can be explained as a result of a trap-assisted electron motion inside a insulator. In fact electrons are usually trapped in single localized states, and only through a random thermal energy contribution they may "jump" to the conduction band; then they move for a limited amount of time, ending up relaxing in another state and so on. This slow process is however fastened by the contribution given by a strong electric field (easily found in thin films), that reduces the required energy for the CB "jump" and so strongly increases the current. The current equation for Frenkel-Poole mechanism is given by (Sze, 2012):

$$I = A_{eff} V \exp\left(\frac{q}{k_B T} \left(2\sqrt{\frac{qV}{4\pi\epsilon_r t_{ox}}} - \phi_{barr}\right)\right)$$
(1.4)

The other main class of leakage phenomena takes place when the spatial dimension of a semiconductive material is reduced to the nm range: in this regime, tunneling processes begin to play an (exponentially) increasing role.

One of the most outstanding prediction of quantum mechanics, the tunnel effect is the result of wave nature of matter. Classically, a body with a certain energy can't overcome a potential barrier which is bigger than the body energy; in quantum mechanics, however, a particle (for example an electron) is described as a wave function ψ that satisfy the (time-independent) Schrödinger equation

$$\hat{H}\psi(\vec{r}) = \left[-\frac{\hbar^2}{2m_e}\nabla^2 + V(\vec{r})\right]\psi(\vec{r}) = E\psi(\vec{r})$$
(1.5)

where \hat{H} is the Hamiltonian operator, $V(\vec{r})$ is the space-dependent potential energy and E is the energy; a particular class of solutions is found when the potential has the form (assuming 1 dimensional system, with a<b)

$$V(\vec{r}) = V(x) = \begin{cases} V_0 & a \le x \le b\\ 0 & elsewhere \end{cases}$$
(1.6)

which recalls a metal-semiconductor/oxide-metal system potential profile, because of the barrier given by the factor $E_C - E_{fermi}$ coming from semiconductor. In this case eq. (1.5) takes the form

$$-\frac{\hbar^2}{2m_e}\nabla^2\psi(\vec{r}) = (E-V)\psi(\vec{r})$$
(1.7)

and the solution (a wave incident to the barrier) can be written as

$$\psi = A \exp\left(-ikx\right) \tag{1.8}$$

with $k = \sqrt{2m(E - V)}$. In the case of V>E (inside the barrier), the k becomes imaginary, the final wavefunction exponentially goes to zero, and so does the probability density $|\psi|^2$; however it does not becomes exactly zero, both inside and after the barrier, and this is exactly the prediction of the tunneling effect, i.e. there is a non zero probability to find a propagating wave (so a real particle) after the barrier itself.

The constant potential barrier is the simplest case of tunneling, and in real systems it plays a significant role only at very low spatial ranges (due to the fast exponential decrease): in particular, it takes the name of Direct Tunneling.

If the quantum particle is an electron inside a Metal Insulator Metal (MIM) device (such as a selector), the charges tunnelling process give rise to a current (direct tunneling current in case of constant barrier height) proportional to the amplitude of the electron wave travelling away from the barrier, and so to the thickness of the potential barrier; whenever the systems is biased, one direction is privileged, the barrier becomes *trapezoidal* (right side of fig. 1.5a) and a net current actually flow, given by the expression (Schuegraf and H, 1994)

$$I = A_{eff} V_{bias}^2 \exp\left(-B_{dir} \frac{1 - \left(1 - \frac{qV_{bias}}{\phi_{barr}}\right)^{\frac{3}{2}}}{V_{bias}}\right)$$
(1.9)

where A_{eff} is the effective electrical contact area and B_{dir} has the form :

$$B_{dir} = \frac{8\pi\sqrt{2m_{ox}\phi_{barr}^3 t_{ox}}}{3qh} \tag{1.10}$$

with t_{ox} and m_{ox} the oxide/barrier thickness and electron mass.

A similar result is obtained when the bias voltage applied is higher than the barrier itself (left side of fig. 1.5a): in this case, two differences are present: firstly the distance that must be travelled inside the barrier is actually smaller than the real thickness because of the bending of the bad diagram; secondly, the barrier is no more constant, but can be modeled as a triangular one.

This condition lead to the so called Fowler-Nordheim Tunneling (Schuegraf and H, 1994), whose current expression becomes :

$$I = A_{eff} V_{bias}^2 \exp\left(-\frac{B_{dir}}{V_{bias}}\right)$$
(1.11)





(a) Generic band showing the field-induced (b) Example of tunneling current simulated neling processes depending on the barrier height Φ_b (Schuegraf and H, 1994)

barrier lowering, which leads to two tun- in MATLAB, considering a Pt/IGZO/Pt (plus a series resistance) system; the higher slope is what characterize FN tunneling with respect to DT

1.4 Current challenges

IGZO is still at an early stage in terms of complete understanding of its electrical behaviour and its dependence on all the parameters involved both in material deposition, stoichiometry and full device processes; in fact, only reports of particular devices characteristics are available, even if extensive and helpful to have insights on the origin of particular material features, but there is still a range of question that remain partially or completely unanswered, for example:

• The exact influence of entrapped hydrogen atoms on the electrical characteristics of TFT: for example, it has been shown that H helps the passivation of trap states, causing an inprovement in transistor figures of merit (see Tsao et al., 2010);

- The stoichiometry influence is quite understood, but its relation with deposition parameters and material phase still need systematic study;
- The effect of oxygen annealing (and subsequent trap filling in deep states) on the device performance.

In this framework, our goal was to explore and demonstrate that C-AFM can be a useful and reliable tool in view of a systematic characterization scenario, exploiting its capability of gathering electrical information together with the surface morphological ones; the next chapter describes in detail this technique and its properties.

Chapter 2

Conductive AFM technique for nanotechnology

2.1 Fundamentals of AFM technique

The evolution of technology in the last decades has been mainly pushed toward by the continuous dimensional scaling of electronic devices, which allows to increase the density of logical circuits (and so their computational power), the number of functions integrated on the same chip, the power efficiency of the system, and many others. On the other hand however, the continuous reduction of geometrical dimensions leads at a certain point to several new challenges, brought by the emergence of new phenomena typical of the nanoscale.

The progressively increasing impact of these factors must in any case be controlled as much as possible, so in order to understand these processes and eventually exploit them in new devices and components it is of crucial importance to be able to *measure*, to quantify them at the same, or even lower, physical scale at which they take place.

In 1986, Binning et al., 1986 proposed a novel technique capable of investigating surfaces of insulators on an atomic scale, the Atomic Force Microscopy, that radically changed the approach to study the properties of matter thanks to its capability of detect features with nanometric lateral resolution, without damaging the surface itself. Their solution is based on the detection of elastic deformation of a micro-cantilever beam, equipped with a sharp diamond tip at its end, scanning over the sample: any interaction between the tip and the surface generates forces (e.g. Van der Waals, electrostatic etc.) that are kept constant by adjusting the tip distance through a feedback loop; at the end, the recordedd deflection can in turn be used to obtain 3D images of the sample morphology and/or surface properties.

This setup, and the following improvements, has been demonstrated to be able to detect features with 1Å vertical and 30Å lateral resolution in air, thus proving to be a promising candidate for a variety of new application.

2.1.1 The AFM setup

An AFM tool is composed by a number of subsystems, being the following the most important ones (see 2.1):

- The cantilever structure holding the probing *tip*, the central element of any scanning probe setup since it is the part that truly interact with the sample;
- A Photodiode (PD) array, which is used to detect the position of a laser beam reflected by the backside of the cantilever: this allows to measure changes in cantilever deflection during the experiment that results from the interactions between sample and tip;
- Piezoelectric-based motion system, which goal is to keep the force (or distance, depending on the configuration adopted) between the tip and the sample constant: this is achieved through a feedback loop detecting any change in laser deflection in PD.



Figure 2.1: General contact-mode AFM setup, with visible cantilever group, laser and PD, piezoelectric motor.

2.1.2 AFM modes

By changing the interaction conditions between the tip and the sample, there are mainly 3 ways, or modes, to operate with an AFM tool:

- Contact mode: in this configuration, the tip is put in direct contact with the sample surface with a force, function of the spring constant of the cantilever, kept constant by a feedback loop. The latter controls the piezoelectric motor depending on the changes of laser beam position on the PD array caused by changes in cantilever deflection during the scanning process;
- Non-contact (NC) mode: in this mode the cantilever is made oscillating at its resonance frequency and brought close to the sample. The feedback loop allow to keep the oscillation amplitude and frequency constants: this time, the changes are caused the tip-sample interactions taking places at small distances, e.g. Van der Waals forces;
- Tapping mode: this mode is similar to NC-AFM mode, but it is used to prevent accidental sticking phenomena, caused by surface meniscus forces, that could immobilize the tip. To avoid this, the oscillation amplitude is bigger, so the probe is periodically in contact with the sample but it is prevented to be immobilized.

Each of these allows to perform different types of analysis and to gather a wide range of information about the sample under study, so it is crucial to choose properly the mode and the relevant parameters in order to get the best results.

2.1.3 Tip influence on morphology

It has been mentioned that the AFM technique allows to achieve very high lateral resolution, but what is the final limit of a surface morphology measurement?

Figure 2.2a shows a common situation: the AFM works in contact mode, with the tip scanning the sample surface touching it; the tip is not an ideal, point-like needle, so it will cause a sort of information loss if the surface below it has features with smaller lateral dimensions, such has hillocks or material islands. What happens is that the surface reconstructed by the elaboration of the laser deflection signal will not be exactly equal to the real one, but it will be limited by the geometrical convolution of the latter with the tip shape.

What one obtains from the measurement is something similar to fig. 2.2b: all the small-sized features are lost, and only a "softened" version of the original profile is measured; this is why any AFM tip has a very high aspect ratio, in this way it is possible to minimize this convolution loss.





(a) Schematic of typical AFM scenario in (b) Height profile actually recorded from over it; it is possible that the sample mor- tip shape and the sample profile phology presents features with dimensions smaller than the tip apex profile

scanning mode: the tip (blue shape) is in the laser deflection signal during the scan: contact with the sample surface and scans it is the convolution of the non point-like

Collecting voltage/current information: the 2.2**C-AFM**

The AFM provides an optimal solution to gather information from sample surface with nanometric lateral resolution, so it is a crucial tool for the analysis of materials and components in which 2-dimensional superficial features play a dominant role in their behavior (for example surface uniformity in thin films); however, the morphological information is (almost) the only information that can be gathered from a sample: for example, no clue is given about the electrical properties of the sample, which in turn are very important in today electronic circuits where nanoscale phenomena become more and more dominant and size-dependent (ballistic conduction, tunneling processes, compositional non-uniformity).

In this framework so it would be very useful to integrate in the same tool a system able to detect electrical currents across and/or over the sample itself.

To this end, in 1992 Murrell et al. demonstrated for the first time that it was possible to investigate insulating films in terms of their electrical properties, in their case the dielectric strength of SiO_2 12-nm layers, by using a modified AFM featuring two new elements:

• a conducting cantilever-tip system, in this way by applying a bias between the tip and the sample it is possible for electrical current to flow (see fig. 2.3a);

• a current pre-amplifier, able to convert the analogical I(V) signal into voltage signal readable by the computer (see fig. 2.3b).



on Si cantilever, notice the ~ 10 nm apex setup: in particular, the right part reprediameter ()Celano, Hantschel, et al., 2015

(a) Example of doped-diamond tip mounted (b) Electrical schematic of the C-AFM sents the current amplifier

In particular, they employed two different methods to study the conduction properties of the material. The first one consisted in positioning the probing tip on the oxide surface and measuring the current flowing while a bias voltage was ramped over a certain range: in the end, the I-V characteristic was collected and important consideration on the tunneling regimes were made. In turn, the second one consisted in scanning the surface in a array of pixel and collecting, for each step, the current in the same way as before: in this way, important consideration on breakdown uniformity over a certain surface were obtained.

To summarize, it was demonstrated the capability of this new approach to study from the electrical point of view insulating thin film samples at the nanoscale, opening in this way a large amount of new possibilities and applications in this field. This is actually what has been exploited in this work, being this technique a natural framework to study thin insulating oxides films in terms of conduction properties.

2.2.1The conductive tip

As said before, the solution to obtain C-AFM from the traditional AFM is basically to exploit a conductive cantilever, so that a current can flow through it and the sample itself when a bias is applied.

Several types of C-AFM tip are available, and the choice is again heavily influenced by the type of information desired and the way this technique is used: without taking into account the fact that a C-AFM tool can be used also for techniques like Scanning Spreading Resistance Microscopy and Scanning Capacitance Microscopy (which are performed with particular types of tips), the two main families of tips are:

- Doped diamond tips: this type is based on a heavily doped (usually B-doped) diamond pyramid mounted on a Si cantilever; these usually keep for a long time their shape and electrical properties without deteriorating, at the cost of being very stiff and so increasing the risk of sample damage;
- Si tips varnished with thin metallic films: in this case, the reduced stiffness (and so softer and reliable contact with the sample) is compensated by a reduced lifetime, caused by the high-current thermal melting, oxidation and mass loss during the scan.

For this work, PtIr-coated silicon tips have been used; the reasons of the choice are the very high resolution they offer (radius of curvature ≤ 25 nm), the low stiffness (~ 7.5N/m), the high conductivity of the metal cover and, at least for the a-IGZO samples, the fact that platinum is also the material used as electrodes of the full device.

2.2.2 The current amplifiers

The second element that converts AFM in C-AFM is the current amplifier, which role is to collect the current flowing through the tip-sample contact and to convert it in a voltage readable by the computer; in practise, it is a box containing the electronic circuit that receives as input the wire arriving from the tip holder, over which the cantilever is attached, and gives as output the voltage signal that is recorded for the current map generation.

For this work, the tool was equipped with two different amplifiers:

- C-AFM sensor: this module is used to sense currents in a range that spreads from ${\sim}100 {\rm pA}$ to 500nA
- TUNA sensor: this module is used to sense currents in a range that spreads from ${\sim}1\mathrm{pA}$ to 500pA

The proper sensor must be chosen depending on the current that is expected to flow through the sample, or on the information that is looked for; in our case, we used C-AFM only for the first part of the work, while TUNA was also used in the second part.

2.3 C-AFM measurements preparation

Performing a C-AFM measurements requires a certain number of steps, which can be divided in two main groups:

- Tool and sample preparation;
- Choice and adaptation of measurements parameters;

While the first is a set of predefined steps that have to be performed in a fixed order and way, the second includes much more tricky and sample-dependent ones.

2.3.1 Tool and Sample preparation

When a C-AFM analysis has to be performed it is necessary to prepare the tool (see figs. 2.4a and 2.4b for reference) and the sample, following these general, main steps:

- Mount the tip on the tip holder, ensuring a good electrical contact with the clamping element that will be connected to the current amplifier;
- Mount the tip holder on the tool head and connect the wire to the sensor, attached to the head as well;
- Focus the laser dot, reflected from the top of the cantilever to the photodiode array, by adjusting mechanical screw: in this way one achieve a good S/N ratio on the laser deflection;
- Put the sample under the tip at a proper distance.

These steps are standard and do not change so much also with different instrumentation; what is more difficult is the next part, the scanning parameters.

2.3.2 Engaging and scanning parameters

Once set up the tool, the next step is to perform the tip approaching toward sample surface. This requires a very delicate and tool/sample dependent parameter determination: in fact, the user must choose the best configuration that depends on these main parameters:

• Deflection setpoint: one of the most important value because it is the (voltage) value at which the feedback circuit is set for the cantilever deflection adaptation,



(a) Picture of the tool used for this work, a Dimen- (b) The tip holder with a clamped sion 3100, with the main elements highlighted.

AFM tip; the cantilever is not visible due to its μm dimensions

determining in this way the force that will be applied to the tip; it should be chose very carefully in order to achieve a good contact while reducing the tip damage, but it is not the only factor defining the deflection: the latter in fact depends also on the spring constant of the cantilever which is far from being exactly known.

- Scan rate: is the frequency at which the scanning tip will perform a full trace/retrace line loop before passing to the next scanning line; this is important because it will determine in particular the lateral resolution of current maps, being the latter "limited" by the limited amplifier bandwidth (in general, the lower is the scan rate, the higher is the resolution, however also the electrical stress in each pixel may be stronger);
- Sample voltage: it is the voltage that will be applied to between sample and tip (that is virtually grounded in the amplifier), and will determine the current regime as well as, to a lesser extent, an increased force between the tip and the sample.

Once these parameters have been set, the piezo motor moves slowly the head/tip toward the surface and, once the laser deflection reaches the voltage set point, the scanning process starts accordingly: it is clear that each value decided will probably not be the optimal one on the first try, and adjustments must be applied before being sure the image will be good enough.

Whether after a certain time one can be more or less sure of the choices to be made for morphology measurement when dealing with a certain class of materials, different thing is when dealing with electrical maps. In fact, even sample of the same material can be different (as for example will arise in chapter 3) and the electrical contact becomes a crucial concern: in this case, it becomes much more tricky and less general to set parameters like deflection setpoint (for good contact) and applied voltage, resulting in a lot of pre-study time and explorative sessions required; furthermore, even knowing a good set of approaching setting may not be enough because of tip variability not known a priori.

2.3.3 Example of data collected from C-AFM

Once the scan starts, the image of the surface morphology is progressively generated on one "channel" of the controlling software, and if a bias voltage is applied, possibly a current map is generated on another channel, too; As said, all the parameters set prior the tip approach will have influence on both the images in terms of resolution and reliability.

To give an example of a good measurement, fig. 2.5 shows both surface morphology and leakage current as "heat maps" of a IGZO sample.



(a) Surface morphology of $500nm \times 500nm$ (b) Current map obtained together with the area, obtained with 0.3V deflection set point morphology, with 0.5V of applied bias and 0.6Hz scan rate



A number of information can be obtained both considering each images on its own and cross-analysing them: for example, fig. 2.5a clearly show a very smooth morphology with sorts of grain boundaries, while fig. 2.5b clearly show a non uniform conductive behaviour of the sample, with dark (insulating) regions "dotted" with clear leakage paths; considered together, they show sometime a sort of overlapping of leakage spots with morphology peaks, rising questions on these phenomena.

2.4 Measurement methodology and workflow of this work

The fact that a C-AFM tool is in general unique doesn't mean that there is only one way to obtain information from samples. As already said, the inventors themselves exploited their setup in two different ways, namely letting the tip scanning with a fixed bias a part of the surface and by applying voltage ramps in several surface points.

In this sense, we adopted a similar workflow together with some new steps in order to obtain several figures of merit able to describe electrical behaviour of the IGZO blankets. In the following section, more details about the adopted analysis technique are provided.

2.4.1 Scanning mode analysis

The first methodology adopted to characterize IGZO is the scanning mode analysis: this replicable and reliable technique that we developed requires the following three steps, which lead from the measurement session to the final figures of merit:

- Acquisition of the current map: the AFM tip is let scanning a portion of the sample surface (e.g. $1\mu m^2$) with a fixed bias applied, the flowing current is recorded for each point and a 2D image is generated (see as example fig. 2.6a);
- Offline processing of acquired images via software (Gwyddion); starting from the raw image, two different analysis are introduced:
 - This software allows to apply so called "masks" to the image, that means it is able to enhance or isolate part of the latter depending on what the user wants to focus on (an example is given in fig. 2.6b); since we were interested in the dark,conductive spot, we applied a mask that extract only the darkest regions of the map neglecting the white ones - namely the low current parts. Starting from this, it is possible to gather statistical information about this leakage, lately used for samples comparison;
 - Rethink the image as a matrix of numbers corresponding to the current that has been recorded in each map point. In this way, it is possible to visualize the 2D information of the map in a different way, by extracting the so-called current-per-pixel distribution: briefly, we could obtain via software the relative percentage of the image pixels(which become the y axis) that features a certain value of current (which set become the x axis); for example, an insulating material would give a distribution that features high y (so high percentage) at low x (so low current magnitude)
and very low high-current percentage.

At the end, one can perform this analysis on several samples, both with fixed or variable parameters such as the bias voltage, and then to compare the results.



(a) Example of current map obtained with PtIr tip, applied $V_{bias} = 500mV$

(b) Mask of the current map extracted through the software Gwyddion, and used to gather leakage-related information

Figure 2.6: Example of the first two steps of scanning map analysis flow

2.4.2 Spectroscopy mode analysis

The second methodology adopted is based on the capability of a AFM tool to perform a land-and-withdraw motion of the cantilever, in other words to make the tip land locally and then be lifted away without moving in the *xy* plane during the process; the Spectroscopy mode allows to perform this on an arbitrary number of points on the sample.

Exploiting this, our method requires the following steps to gather other type of information:

- Define a grid of points over the surface, where the tip will land and then be withdrawn (see fig. 2.7a). In this way, it is possible to focus on and investigate an arbitrarily large region of the sample surface;
- Apply bias ramps in each grid point (an example of ramp is shown in fig. 2.7b); depending on the desired information it is possible to vary several ramp parameters, such as the voltage range and the frequency of each sweep;
- Gather a number of I-V characteristics for each point that can be then post processed and used to gather other figures of merit; in particular,
 - it is possible to gather overall information on the uniformity of the electrical behaviour of the region stressed by averaging all the curves;

 quantitative evaluation of the displacement/reliability of each I-V characteristics with respect the average one.



(a) Example of 8x8 point grids: in each square, the tip is put in contact with the sample without scanning, undergoes bias ramping, and then it is withdrawn away and moved to another square



(b) Example of bias ramp applied in spectroscopy mode, once in each grid point

Figure 2.7: Main steps of spectroscopy mode analysis

After this analysis is performed, different information are extracted for each sample, allowing both their understanding and comparison.

2.5 Limits of C-AFM

The very high resolution given by C-AFM and in general the critical, nanometric dimension of any measurements are, on one hand, great and important features that allow to obtain very precise results and deep understanding of a sample, but on the other hand they bring also problems and difficulties that have to be spotted and taken into proper account.

The most important factors that have to be considered when approaching C-AFM involves both the scanning tip and the sample surface: in fact, any problem or change that take place at their interface can lead to unreliable or even completely wrong results, so a combination of experience/awareness and criticism is necessary during any analysis. In the following sections, the most critical problems are analyzed.

2.5.1 Tip contact area and degradation during measurements

The tip is of course the most delicate part of the C-AFM, being the element that defines the resolution and accuracy of any measurement; it is also easily understandable that its apex is the part suffering the biggest stress, both mechanical, electrical and thermal.

In fact, whether it's true that the force applied to the cantilever is very low (typical values are $\sim 10^{-10}$ N, the pressure is very high because of the very small physical contact area, which can be estimated to be around $100nm^2$ (even though lower values are easily reached), so it is inevitable that for example the metal coating is damaged after some scanning time (see fig. 2.8a for an example of tip break); also, the high current densities that flows through the apex (the electrical contact area is smaller than the physical one, easily less than $50nm^2$) lead to temperature increase that can easily melt (or oxidize, in case water film is present on the surface, something common in air) part of the tip (see for example fig.

The result is bad morphology and/or current maps, sometimes this can be even noticed in real time during a scan, as shown in fig. 2.8b; the randomness of these phenomena is the main problem for a proper understanding of C-AFM results.



contact measurement session; this, together tion of tip conductivity impacting current with material sticking on the apex, is the map: scanning from bottom to top at fixed most common sources of bad AFM images (Killgore et al., 2010)

(a) SEM image of a tip before and after a (b) Example of real time, progressive reducpositive bias, it is noticeable the difference between the current collected at the beginning and at the end of the scan.

2.5.2Tip-emission and back-contact emission

Another issue that arise when characterizing samples with C-AFM is an effect that can be thought as a "current emission asymmetry"; this phenomenon arise when one compares for examples a set of I-V characteristics of nominally symmetric systems, for example a platinum back electrode - IGZO - platinum AFM tip. In fact, despite the electron injection from the metal to the IGZO should follow the same behaviour from both sides, what turns out is that this does not happen: in particular when the emission takes place from the tip to the IGZO the I-V curves are much less stable and symmetric than the ones obtained when the back electrode emits the electrons.

The origin of this effect can be attributed to a combination of several problems:

- There is a difference in terms of conformality, extension and uniformity of the interface area between the metal and the IGZO: in fact, the bottom electrode features a large and uniform contact with the film which is not detrimental for the electron emission; on the other hand, the tip has a much worse contact with the material, and also its material uniformity is much less than the one of a big electrode;
- Anything present at the tip-sample interface, from water films to degraded metal coating, becomes a further obstacle for tip emission
- The work function of Pt electrode and PtIr tip are different, so the Schottky barrier is not symmetrical (neither uniform for the tip because of random damages adn changes).

Improvements can be obtained by increasing the deflection of the cantilever (higher force) to achieve a better contact, but this can be detrimental especially in scanning mode and in any case it does not solve completely the problem.

In fig. 2.9b the simplified band diagram of a Pt BE/a-IGZO/PtIr tip system is shown, with a bias applied to the back electrode: in this case, the tip is emitting electrons, but water and bad/small contact area makes the injection less effective and less stable across different surface points; on the other hand, fig. 2.9a shows the negative bias situation, in which the emission happens at the BE-IGZO interface, that is much larger and more uniform.

At the end, it must be said that this can be either a minor or irrelevant problem, in the sense that if the final device based on the blanket will be symmetrical (e.g. IGZO-based selector with platinum TE and BE) it is fine to just focus on the negative polarity; if it is not the case, information can still be obtained from the positive polarity, for example averaging a large enough number of I-Vs and consider the result as a lower bound for the real characteristics.



(a) Tip limited emission



Figure 2.9: Example band diagram of Pt/IGZO and tip system in two opposite biases conditions: in the left, negative bias is applied causing the electron emission to happen from the large and uniform area of the platinum back electrode, while on the right the positive bias cause the emission to take place at the tip-sample interface, which instead features a much less uniform contact area with in addition the influence of any compound of film between them.

Chapter 3

Measurements and results on Spinel/CAAC-IGZO films

In this chapter several results and figures of merit obtained from crystalline IGZO blankets are shown.

In particular, for what concerns these samples, the nature of surface-limited conductivity allowed to exploit both the two C-AFM modes: in the end, these resulted to be useful means to gather a diversified amount of information that allowed to make quantitative comparisons.

3.1 Schematic of blanket samples analyzed in this work

First of all is important to understand how the samples were prepared for the measurements: in fig. 3.1 a summary and sketch of the system under study is shown.

The main idea was to be able to characterize these crystalline blankets in a proper way considering their application as transistor channel: to this end, the samples were given to us after the IGZO deposition process, in order to have the film exposed (central subfigure). In this way, it was possible to directly contact the IGZO on one side of the blanket to the "gate" by means of conductive silicon paste: the goal of this step was to mimic a drain contact.



Figure 3.1: Sketch of crystalline, blanket samples: on the left, the schematic of a generic IGZO TFT; in the center the real sample studied, i.e. the device development is stopped at channel layer deposition step, is shown; on the right, schematic of the tip, cantilever, bias voltage and side-contacted sample.

At this point, the samples were ready to be scanned by the tip, that can be thought as a nanometrical, movable source contact of a generic transistor, and this can lead in the end to achieve useful information.

3.2 Overview of samples under study

During our work, a number of samples of IGZO in blanket form were studied, each one different from the others depending on several properties and deposition parameters, namely:

- Deposition techniques: depending on the film deposition, two main families of samples were studied: ALD-deposited and PVD-deposited;
- IGZO phases: depending on the resulting material phases, the two main families were the Spinel and the CAAC,
- Deposition thermal budget, interesting in terms of possible implementation in BEOL;
- GZO interlayer thickness: this thin film is deposited prior the IGZO deposition in order to smooth the transition between the lattice constant of SiO_2 and the IGZO one; without this buffer layer the final quality would be quite low.

In particular, in table 3.1 the complete list of samples and relative properties is shown.

The reason behind such a wide range of differences is easily understood in terms of the necessity to study the largest pool of material variability, in order to gather as much as possible an overall understanding of the interdependence among all the samples variables.

The characterization of these samples has been carried out by performing both the main C-AFM modes introduced in section 2.4.1: for each one, several figures of

Sample	SiO2	GZO	IGZO	T_{dep} °C	Deposition	Phase
D05	10 nm	3.39 nm	24 nm	200	PVD	Spinel
D07	10 nm	2.17 nm	24 nm	200	PVD	Spinel
D08	10 nm	1.40 nm	24 nm	200	PVD	Spinel
D09	10 nm	1.29 nm	24 nm	200	PVD	CAAC
D10	10 nm	3.40 nm	24 nm	25	PVD	Spinel
D20	10 nm		24 nm		ALD	Spinel
D21	10 nm		24 nm		ALD	Spinel

 Table 3.1: List of analyzed samples with corresponding properties and process parameters.

merit have been gathered and used to define a valid and reproducible methodology to characterize IGZO samples; in addiction, it has been shown the capability of this workflow to verify particular correlations between its results and process parameters.

3.3 Scanning mode analysis

The first C-AFM approach to the samples characterization is the scanning mode. This method relies on the acquisition of several current maps of each sample while applying (a number of) fixed bias voltage between the tip and the sample; from these 2D images, two types of post processing are carried out allowing to obtain quantitative information capable to give a "fingerprint" of each sample.

In figs. 3.2a to 3.2g one example of current map for each sample under study is shown; although differences are noticeable even by direct images comparison, our elaborations is meant to give more quantitative insight of these discrepancies.

In the following sections, the "off line" elaboration processes are described and the resulting figures of merit are discussed.

3.3.1 Leakage spot densities

In first place, we noticed as first possible characterizing factor the different amount of current leakage spots (dark regions in current maps) in each sample: to this end, we exploited the software Gwyddion for the elaboration.

Following the workflow described previously, we applied masks (with the same "current threshold" criteria) to current maps and extracted a number of quantitative values related to the different leakage spot distributions.



In fig. 3.3a a plot of spot densities for all the PVD samples is shown: in particular, data from current maps measured at the two mirror biases $\pm 500mV$ are displayed.

From this graph a number of information can be obtained. First of all, it is possible to verify that among the gathered data the same trend is present, namely positive bias lead to a reduced amount of spots with respect to negative one. This observation may be caused by two factors, namely an intrinsic material property or the influence of tip-emission issue mentioned previously; in general, also taking into account results from I-V characterization (which show an appreciable symmetry in this bias range) may suggest that the influence of the tip emission is particularly important in scanning mode, where together with the low contact force may lead



Figure 3.3: Plots of leakage spot densities of PVD samples.

to a reduce current collection. Also, the same trend between the two plotted lines may enforce the fact that the material is conductive no matter the bias polarity applied.

Speaking about the more important samples comparison, a first important difference, already noticeable by simply inspecting PVD images, is present: in general PVD Spinel samples feature a spot density which range is one order of magniture or more bigger than the PVD CAAC sample (for both polarities). In this sense, a possible screening method may involve the evaluation of the spot densities and checking wheter large differences are present.

This can also give some insight in view of a possible implementation as transistor channel, in fact being conductive even at low applied voltages may not be a good property, and in this sense the CAAC would be a good candidate for further study.

It is possible to extend the information obtainable from this analysis, for example by comparing spot densities acquired at different biases: in fig. 3.3b an example with 4 different biases is shown.

In this case, what is interesting is to check the presence of particular trends of spot densities with respect the electrical stress: in fact one could be concerned about the origin of the leakage, for instance if these spots increase in number with increasing voltages or simply more current flows through a constant density of them.

We report as preliminary result the fact that, at least for low biases, in general PVD Spinel samples feature a spot density that seems to be almost independent on the applied bias magnitude, both in positive and negative polarities; PVD CAAC spot density, instead, seems to be bias dependent being it increasing with higher electrical stress. Again, this may be another discriminant factor capable to detect different phases among unknown samples.

3.3.2 Current-per-pixel distribution

The second way to extract information from C-AFM current images, as mentioned, is to re-think the current map as a matrix of numbers, each corresponding to the current measured in a particular spot of the sample.

In this way, it turns out to be a good method to extract in a well defined way what we called "current-per-pixel" distribution: in other words, the number of pixels featuring the same value of current are counted together, and this is performed for the whole current range present in the image.



Figure 3.4: Current-per-pixel distributions plot of all the characterized samples: there are clear differences among the curves of samples differing in phase and/or deposition technique

Once this method is performed for a number of different samples, it is possible to plot these distribution.

Figure 3.4 show the distribution of all the analyzed samples: in particular, curves of PVD samples were extracted from maps gathered with 0.5V bias, ALD ones with 2V. Again, the careful inspection of the plot allows to draw conclusions about each blanket: in particular, there is a strong difference among different families of them, demonstrating the capability of this "matrix-conversion" process for the characterization.

In fact, it is noticeable the separation among three main groups of curves:

• PVD Spinel samples (highlighted in fig. 3.5a): they feature curves with

constant distribution up to a certain current magnitude, after which an abrupt reduction around 50nA is observed;

- PVD CAAC sample (shown in fig. 3.5b): differently from other PVD, its curve is monotonically decreasing with constant slope but reaches higher current magnitudes; this is coherent with the fact that it is much more insulating, which translate in a higher density of low current pixels;
- ALD samples: in this case, their distributions feature a sort of plateau for intermediate currents, with a strong reduction of the distribution after a certain value; it is noticeable the order of magnitude of current (pA) and the voltage applied (2V), showing the highly insulating behavior.



ALD current (pA) Current distribution (a.u.) 10 100 0.01 D 20 D 21 02 0.001 D20 009 1E-4 1E-5 100 10 PVD current (nA)

tions: it is noticeable the common behaviour current distributions: it is noticeable the of all four samples, with an almost constant difference between CAAC and the Spinel, curve slope up to a certain current value the first showing a constant negative slope and subsequent abrupt decrease.

(a) Insight of Spinel PVD current distribu- (b) Insight of CAAC PVD and Spinel ALD curve that however reaches higher current magnitudes; ALD ones instead feature a sort of plateau with subsequent decrease

To summarize, in this section we showed another way to extract information from C-AFM current maps: the current-per-pixel can be a good method to gather quantitative information about the samples because of the different curve that arise from different phases or deposition techniques, being in this way another candidate for automatic blanket screening.

Spectroscopy mode analysis 3.4

The second way the C-AFM can be exploited is the so called "spectroscopy mode": as a reminder, in this configuration the tip does not scan the surface, instead a

grid of points is defined so that the tip lands in each one and perform a local I-V characterization.

Examples of what is found after an exploratory spectroscopy session is shown in fig. 3.6, divided in the main group of samples; in particular, or what concerns these samples we adopted as standard ramping procedure two bias swings, the first from negative to positive voltages and the second the opposite.



Figure 3.6: Plots of experimental I-Vs over grid

Once a significant number of I-Vs has been obtained, they can be post-processed to gather quantitative information; in the following sections, the two method described in section 2.4.2 are reported in details together with the conclusions they carry.

3.4.1 I-V characteristics averages

The first post processing performed is an averaging of the I-V curves obtained from the grid points: this allows in theory to have insight of the general behaviour of a certain sample, together with the capability to find important differences among different blankets,

In fig. 3.7 all the averages are plotted in the sample graph, in order to ease a direct comparison.

In general, as already seen from the scanning mode results, spectroscopy mode allows to divide the samples in three main group depending on their characteristics:

- PVD Spinel blankets: represented by continuous curves, they show an bipolar conducting behaviour, with overall higher currents measured in the positive biases with respect to negative one;
- PVD CAAC blanket: represented by the dashed line, it is quite different with respect to the previous curves because current has been found to flow only for negative biases and, in addiction, it reaches higher values from biases higher than $\sim -1.3V$;

• ALD Spinel blankets: these are completely different from the others, being conductive only in positive biases and in general achieving very low current magnitudes even at quite high voltages.



Figure 3.7: I-V characteristics averages of the samples; it is possible to see differences not only between PVD and ALD blankets, but also among different PVD ones

From this preliminary analysis, spectroscopy mode seems to be in agreement with the scanning mode results, because again three main groups arise featuring coherent information. It may be noticed that the CAAC sample (D09) actually showed conduction also in positive bias in current maps: this asymmetry however can be explained considering a combination of the relative low amount of conducting regions (fact emerging from the spot density) and the tip emission issue, that results in a low probability to land in a leaking region with a good tip-sample contact.

However, the spectroscopy results suggest another, deeper insight for what concerns PVD Spinel samples that was not found in spot density plot: in fact, two subgroup arise among D05, D07, D08 and D10.

In particular, D05 and D10 are found to be more conducting than D07 and D08, behaviour visible by the clear current curve splitting on both positive an negative polarities: this is a very promising result, because it seems to confirm the expected dependence of conductivity on the GZO buffer layer thickness.

In fact, the IGZO deposition can't take place directly on the substrate due to lattice constant mismatch, which would lead to a bad film homogeneity and quality properties; to this end, a interlayer of GZO is deposited prior the real IGZO to ease this transition; however, the final result is dependent on this layer thickness, in general the thicker it is, the better will be the final IGZO film.

Keeping this in mind, we can say that is a good proof of validity the fact that the mentioned division among PVD Spinel samples exactly reflects the GZO layer thickness influence on electrical properties: in fact, the higher conductive samples, D05 and D10, are those which feature the thickest GZO, namely around 3.4nm, while the lower ones, D07 and D08, have sensibly thinner GZO (below 2.4nm).

To summarize, we can say that averaging post process in spectroscopy mode support and complete the scanning mode results, and can even give more information in some scenario where more refine analysis is needed.

3.4.2 Current spread at fixed bias among grid points

Together with the average behaviour, it may be interesting also to check the variability of all the I-Vs obtained from the virtual grid in order to evaluate whether the behaviour is overall stable or the spread among curves is significant: in this sense, another post processing analysis we developed concerning the spectroscopy data is represented by the analysis of the distribution of currents at particular bias values.

To this end, a custom python script has been developed: it receives as input two main information:

- a list of *.txt* files containing the I-V raw data;
- the voltage at which perform the extraction: in other words, a reference bias is given, and from all the I-V the current magnitude measured at that bias is extracted;

In this way, the user can choose in which bias region he wants to focus in order to have a more precise understanding of the material stability in that stress condition.

In fig. 3.8 the current spread at both positive and negative biases is shown. Thanks to this plots a number of information can be obtained: first of all, especially by inspecting the negative polarity plot, we can again clearly notice the presence of the three main groups of samples:

- PVD Spinel blankets D05 and D10 (connected by the blue horizontal line) showing higher currents ($\sim 50nA$) at -2V;
- PVD Spinel blankets D07 and D8 (connected by the red horizontal line) showing lower currents ($\sim 20nA$) at -2V;
- PVD CAAC blanket, showing a very wide spread of currents among all the I-Vs, in any case reaching easily current magnitudes around 200nA.

These results support the findings of the average I-Vs, this time however is possible also to gather information about the stability of these characteristics: in fact there is a clear differences among the spread of Spinel and CAAC blankets characteristics, being the latter much more "unstable" with respect to the first ones. This one one hand important to have insights of what are the mechanism that cause the surface conduction, on the other hand again confirms the current map results, that showed the CAAC D09 to be a very insulating layer, by ensuring the presence of some highly conducting regions and of very insulating ones.



(a) Current spread of PVD blankets, refer(b) Current spread of PVD blankets, reference bias -2V ence bias +2V



Other information can be gathered in turn considering the positive polarity current spread. First of all, it is clearly visible a general increase of curves spread with respect to the negative bias ones, something that we can explain with the mentioned tip emission problem, but it still show the trend among Spinel samples; secondly, despite the instability it is clear that with a positive bias the Spinel IGZO is able to reach higher currents, sometimes around 100nA in the case of the high conductive one.

There is still another curious phenomena clearly happening: it is noticeable a sort of drift in all the samples depending on the ramp considered and the extraction bias: in fact, whenever the voltage polarity belongs to the first ramp applied (e.g. -2V and ramp from -2V to +2V, or viceversa, red boxes), the current measured is always higher than the corresponding value measured when the ramp is reverted

(e.g current at -2V and ramp from +2V to -2V, blue boxes): this can be an sign of (temporary) charging effect that must be taken into account for example when comparing simulations and experimental data, since it may be the signature of trapping sites in the material.

Chapter 4

Measurements and results on a-IGZO films

4.1 Overview of samples under study

For what concerns the selector application of IGZO, the amorphous phase is the one investigated for its high NL factor and the tolerable current density: to this end it is important, given the tunneling origin of its conducting properties, to study the importance of the active layer thickness and its uniformity/stability.

Sample	Phase	IGZO thickness	BE material	Anneal
D11	Amorphous	6 nm	Pt	No
D11-A	Amorphous	6 nm	Pt	Yes
D12	Amorphous	4 nm	Pt	No
D13	Amorphous	2 nm	Pt	No
D14	Amorphous	6 nm	Ru	No

Table 4.1: List of analyzed a-IGZO samples with corresponding thicknesses andBack Electrode materials.

In 4.1 the full list of analyzed sample is shown, together with the corresponding crucial parameters involved: as previously mentioned, IGZO-based selectors are influenced by the active material thickness because of their tunneling conduction behavior, and this motivated the choice of analyzed layers variability. The goal

of our measurements was to check the capability of C-AFM to detect electrical properties found in full devices, in order to align with those results and explore in this way its feasibility as screening tool also for this kind of samples.

4.2 Schematic of blanket samples analyzed in this work

As before, it is important to clarify the preparation of the samples in view of the characterization: in fig. 4.1 a summary and sketch of the system under study is shown.



Figure 4.1: Sketch of the a-IGZO blanket samples: on the left, the generic bipolar selector structure is depicted; in the middle, the real blanket sample structure is shown, obtained by stopping the development after film deposition; on the right, C-AFM tip schematic and bottom-electrode contacted sample is shown.

The idea now was to perform measurements in order to emulate as much as possible a real selector device: in this sense, the samples were provided again right after the IGZO deposition process in order to have the film exposed. In this way, it was possible to scratch the blanket at one end and to contact the platinum/ruthenium back electrode to the tool sample holder.

At the end, the samples were ready to be investigated by the tip, again thought as a nanometrical, movable platinum top electrode of a real device.

4.2.1 Challenges of scanning mode analysis

It must be mentioned that the analysis of a-IGZO blanket could only be performed by exploiting the spectroscopy mode: the high rectifying behaviour and the reduced thickness of the samples caused problems in any attempt using scanning mode.

In fact any measurement, even at very low biases, turned out to be without current at all or with a damage on the surface of the sample. In fig. 4.2a the morphology of Pt/IGZO 6nm (D11) is shown when the scan was performed without applied bias, while in fig. 4.2b the final morphology of the same area after a biased scan is shown:

it is clear that extensive damage took place over some regions, thus impacting any collected current information and rendering it unreliable for any post processing (taking also into account tip-related damages).

The explanation we give to this phenomenon is that a combination of high rectification and lack of control of abrupt current densities during the scanning procedure cause the an extensive damage to the sample in the form of material disruption and oxidation.



Figure 4.2: Example of morphology images of the same region from sample D11: on the left, the smooth morphology has been collected without applied bias between the tip and the sample; on the right, the surface of the region after a scan with applied bias shows high "pillars" of material, probably oxidized and in a different phase.

mode

4.3 I-V characteristics in spectroscopy mode

Given the impossibility to rely on scanning mode, we extensively explored the spectroscopy one in order to compensate this problem and obtain as many information as possible; on the other hand, being the application of IGZO based selector is mainly given by their I-V characteristics, we believe this method is the natural one to exploit for the characterization.

Furthermore, the interest in I-Vs led us to rely not only on the typical C-AFM current amplifier, as done for crystalline IGZO, but also to take advantage of the TUNA one, in order to investigate the characteristics of each sample over a much larger current range than before. The grid-based methodology and post

processing remains the same, what changes is that now we could get information about different conduction regimes. In fig. 4.3 two examples of I-Vs are shown: on



Figure 4.3: Examples of a-IGZO I-Vs captured in spectroscopy mode, divided by sensor used for the detection.

the left, C-AFM current sensor as been used as it can be seen from the current range, and the complete set of I-Vs (dotted background lines) is plotted together with the blue curve corresponding to the total average; on the right, a similar set is plotted, this time it was collected by the TUNA sensor.

Before proceeding, it must be mentioned that given the very small thickness of the blankets the voltage swings were performed starting from 0V and ending at \pm V, in order to avoid abrupt electrical stress across the film with subsequent sample damage.

4.3.1 I-V characteristics averaging

This lead us to verify the capability of this tool to sense coherently both the high and low field regions, and the corresponding currents without noise-limited resolution, for all the samples: in the end, we could extract as first figure of merit for each blanket the average electrical characteristics and plot them (see fig. 4.4a).

From the I-Vs it is in thoery possible to understand which samples are probably the best in terms of characteristics non linearity, i.e. the slope of the curve: while 6nm and 4nm samples behave more or less in the same way, the 2nm (D13) one show a degraded characteristics. This is even more explicit if the current densityelectric field relation is plotted: in fact, as shown in fig. 4.4b, this property is even more explicit because the slope of these curves represent the (average) electrical



sor.

(a) Comparison of the 4 non annealed sam- (b) Comparison of J-E characteristics for ples average I-V characteristics, C-AFM sen- better understanding of the electrical behaviour and conductivity.

Figure 4.4: C-AFM characteristics of the four non-annealed samples. The Pt/IGZO 2nm is shown to feature the worst electrical behaviour among all the others

conductivity.

It is clear that D13 is not a good candidate for applications, or that at least the 2nm layer needs further investigation to understand if material damages, dishomogeneity or deposition issues are the cause of this degradation; on the other hand, other samples are pretty much similar, with good NL factor and high current densities. In particular, for what concerns the current density it was necessary to give a reasonable value to the electrical contact area: despite it can be as low as tenth of nm^2 , we choose a very conservative value of ~ $100nm^2$, that is a(very) worst case scenario.

4.3.2Merging C-AFM and TUNA I-Vs

The reproducibility and the overall stability of the I-Vs measurements sessions with both the sensors gave rise to the question about the feasibility of a "merging" process of the average characteristics obtained through the C-AFM and TUNA amplifiers.

In fact, usually only one is used for a measurements depending on the desired information; however this particular type of samples are interesting both in the high current range, from a device point of view, and in the low one, for a fundamental material modeling point of view, and that's why we wanted to achieve a "extended range I-V" characterization workflow; in addiction, the capability to achieve such merging would prove C-AFM technique to be even more important and reliable for this kind of analysis.

The result of our procedure is plotted in fig. 4.5: what is shown is a comparison plot featuring both the C-AFM and the TUNA I-Vs averages, with a green inset showing the limiting regions of both sensors. It turns out that is actually possible to merge results from the two amplifiers (through a "manual" selection of curves intervals though) and to obtain a full range characteristics of the blankets, particularly useful for comparison with simulations that model electrical behaviour in these ranges.



Figure 4.5: Extended range, averaged I-V comparison of the four, non-annealed samples

Again, D13 is clearly affected by problems in conduction, now given also by a bad superposition of curves with a combined highlight of bad high field currents and lack of "thresholded" behaviour in low fields, both caused probably by film deposition problems or intrinsic electrical properties (the tunneling processes are mainly Trap-Assisted-Tunneling and Fowler-Nordheim, and with 2nm of thickness they may already be very strong, while at 4nm or more they may be exponentially lower).

On the other hand, D11, D12 and D14 show very good electrical characteristics, in particular the Pt/IGZO 6nm seems to be the best one in terms of non-linearty (curve slope), followed by the Ru/IGZO 6nm and the Pt/IGZO 4nm; also, the low field current region show a sort of threshold in conduction (no cusp around zero), a promising result also because device simulations suggest a change in current phenomena in this "plateau" range.

To summarize, we successfully developed a framework able to obtain reliable results

for a-IGZO samples, capable to give a general insight of the average electrical behaviour over a extended current range, without noise limits given by C-AFM sensor only.

4.3.3 Threshold voltages spread of grid I-Vs

As for the crystalline IGZO samples, the set of I-Vs gathered from spectroscopy mode can again be post processed to extract a figure of merit that gives information about the stability over a surface portion of the characteristics collected.

For a-IGZO samples, the uniformity of I-Vs is also a concern because the smaller becomes the single metal-IGZO-metal selector, the more important becomes the electrical stability and reliability, so to check this property is important. In fig. 4.6 this figure of merit is shown as box chart, one for each device.



Figure 4.6: Box plot showing the spread of threshold voltages in each reliable sample (unannealed); the current chosen for the extraction is 100 pA

In particular, we considered the TUNA current for this elaboration. To this end, another python script has been developed in order to automatize the entire process: it is similar to the one adopted for crystalline IGZO analysis, but this time the threshold current is the crucial parameter, and not the reference voltage. Since we wanted to obtain knowledge of the high current inset we elaborated the TUNA data, setting a threshold current of ~ 100pA.

The box plot shows both the positive and negative voltage values at which each I-V reaches $\pm 100pA$. In particular, the first information is given by the fact that there is an asymmetry between positive and negative polarities, a trend that is also found in full devices testing - although less pronounced (probably caused by a combination of the asymmetry in Pt/IGZO/tip band diagram and tail states). Another property that arise is the fact that D14 (Ru/IGZO 6nm) seems to feature

(on average) higher thresholds, that means wider I-Vs: this is something interesting because it may give information about the different band diagram bending of IGZO at its interface when put in contact with the two metals (for example, the widening could be theoretically explained because of the higher Ru work function with respect to Pt one, after a thermal annealing is performed, Nabatame et al., 2006 and Schaeffer et al., 2004), or on the quality/properties of the interface itself.

It is interesting to mention that in this case the typical spread found in the positive branch of I-Vs is less noticeable than in crystalline IGZO: this may be caused by the different nature of conduction, now involving tunneling (and not percolative) processes that may be less influenced by contact non idealities at these thicknesses.

4.4 Indirect detection and confirmation of deposition issues

By comparing the C-AFM I-Vs of two sample in particular, the Pt/IGZO 6nm and Pt/IGZO 4nm, and considering simulation results from other imec groups, a question arose. As noticeable in fig. 4.7, there is almost no difference between the characteristics of the two blankets, while a higher non-linearity factor and reduced threshold is expected when the film thickness is reduced and tunneling phenomena are the predominant ones: why this is not observed?



Figure 4.7: Comparison of D11 (6nm) and D12 (4nm) averaged I-Vs; they look very similar, almost as they were measured from the same device

By verifying that any possible cause of this counter-intuitive result, for example bad tip-sample contact, anomalous tip resistance, damaged or non uniform film region (it is possible to have deposition problems at this thickness scale), were not involved, the only explanation was that the film was actually thicker than expected. To verify this with our tool, we could exploit a particular way to use AFM: as said in the beginning, doped-diamond tips can be dangerous because their stiffness can lead to sample damage, so in typical measurements this must be avoided; On the other hand, it sometimes may be useful to damage, or scratch, the sample. This is exactly how the *Scalpel AFM* works: by using hard tips and applying high forces to the cantilever it is possible to dig holes in a sample, a interesting way to investigate for example the state of the sample under the surface (a crucial example of this technique is its use to detect filament formation in RRAM, see Celano, Goux, et al., 2014).



(a) C-AFM morphological map of Pt/IGZO 4nm with scalpel mode dug hole

(b) Height profile of the hole, demonstrating a difference between nominal (4nm) and real (~5.5nm)film thickness

Figure 4.8a shows the morphological map of the D12, Pt/IGZO 4nm sample after a hole has been obtained by scratching a small area of IGZO through a hard doped diamond tip; starting from this map, again the software Gwyddion has been exploited because it allows to draw lines over the image and to extract its profile, in this case the height along blue dotted one.

Surprisingly, it turned out that this sample was not as thick as it should have been: in fact, as clearly visible in fig. 4.8b, the hole height profile shows that the real film thickness is ≥ 5.5 nm, quite different with respect the nominal 4nm and more similar to the 6nm of D11 (the noisy "floor" is probably caused by a shared feedback existing between the piezo-feedback circuit and the current sensor, which saturates when the PtIr tip and the Back Electrode are shorted).

To sum up, this last result is important from two different points of view:

• It is a positive feedback on the precision of information that this C-AFM based approach can give for early screening test: it demonstrated to be able to

achieve electrical characterization precision enough to both identify problems or unexpected outcomes and give results that align well with devices testing;

• C-AFM gave an important warning about the deposition process adopted for a particular sample, which is going to be investigated further; however, it proves again to be a very promising screening tool when used in our configuration.

4.5 A last comparison: effect of annealing

In the last days of my internship a new lot of IGZO was received for analysis: in particular, it was coming from the same lot of D11 (Pt/IGZO 6nm), this time with a post annealing process: the goal was to check the presence of differences among the two and so to quantify the effect of that extra step.

To this end we performed the same procedure adopted for the non annealed blankets, namely the spectroscopy mode.

4.5.1 I-V Averages

First of all, we wanted to compare the averaged characteristics obtained from both samples, so we performed the combination of C-AFM and TUNA methods to investigate the electrical behaviour of annealed blanket: in fig. 4.9 the merged average I-V is shown. It was possible again to merge the TUNA and C-AFM



Figure 4.9: Extended range I-V characteristic of annealed Pt/IGZO 6nm (annealed)

average I-Vs, demonstrating the reliability of this methodology; in addiction, this

preliminary analysis already gives some information about the behaviour of this sample.

To better appreciate it, a one to one comparison has been made with the non annealed D11: in fig. 4.10a the full scale I-Vs are plotted together, while in fig. 4.10b a focus on the low field region is shown.





(a) Comparison of annealed and nonannealed extended range I-Vs

(b) Insight of low bias region: it is noticeable the deep difference among the two curves

From these plots it is possible to verify the presence of differences among the samples caused by the annealing. In particular, the low field region seems to be the most affected by that extra step: while in the non annealed IGZO there is a smooth transition from "zero" (actually sub-pA) current to conduction, in the annealed one this is not present, and current starts to flow abruptly as soon as the bias assume a value different from zero.

Away from this region the two films seems to behave quite similarly, as visible in particular by the overlapping of curves in the negative polarity region.

In general, the explanation that can be proposed from these results is that the annealing step may cause a reduction/filling of those deep localized states, that are thought to cause a reduction in the charge actually flowing by means of trapping phenomena; of course, further study is required by means of other techniques able to give insights on the gap and tail states.

4.6 Comparison with simulations

Since the tunneling behaviour can be easily modeled in MATLAB, we wanted to check whether the conduction processes described in section 1.3.2 are enough to provide a satisfactory fit of the I-V characteristics within a certain degree of accuracy.

To this end, we considered the negative polarity only, in order to remove any tip-emission issue and be able to focus on material properties only; then, all the collected I-V were plotted together, and a custom MATLAB code was developed to simulate the system I-V.

In particular, the negative polarity and the symmetry of Pt/IGZO/Pt band diagram lead us to consider a simplified system composed by a semiconducting material in contact with a metal (Schottky barrier), so for all the computation, only the electrical parameters of IGZO were necessary.

The simulation code is structured in the following parts:

- Definition of materials parameters, listed in table 4.2;
- Reasonable values for particular quantities, mainly the equivalent series resistance;
- Definition of Current Models: in particular Fowler-Nordheim, Direct tunneling, thermionic Frenkel-Poole emission were included;
- Self-consistent routine based on Newton method, in order to take into account the real voltage drop across IGZO: this is necessary if the more realistic circuit including a series resistance is considered, because the current expression becomes and implicit equation $(I = I(V_{IGZO}(I)))$ due to the ΔV across R;
- Plots comparison for parameters check.

ϕ_W (eV)	χ_e (eV)	${ m m_{eff}}$	$\epsilon_{\mathbf{r}}$	$t_{ox} (nm)$	$A_{\rm eff}~({\rm nm^2})$	$\mathbf{R}_{\mathbf{series}}$ (k Ω)	T (K)
IGZO	PtIr	IGZO				50	50	200
4	4.8	4.16-4.4	$0.34 \ m^0$	10	$5,\!5$	00	50	500

 Table 4.2:
 Simulation parameters

In particular, the self-consistent Newton Method has been employed to solve the implicit equation that arise when dealing with a realistic equivalent circuit of the measurement setup.

As it is well known, the Newton method is based on a iterative loop aiming to determine the root of a non-linear equation : one starts with the implicit version of the equation written in terms of Taylor expansion around an initial guess point x_0 :

$$f(x) \simeq f(x_0) + (x - x_0)f'(x_0) \tag{4.1}$$

letting x_1 so that this expression goes to zero one has

$$f(x_1) = 0 = f(x_0) + (x_1 - x_0)f'(x_0)$$
(4.2)

where the parenthesis term is the first "correction" term of x_0 ; after n steps one has

$$0 = f(x_n) + (x_{n+1} - x_n)f'(x_n)$$
(4.3)

that allows to write the (n+1)-correction (which has the role of convergence parameter, i.e. the loop stopping criteria) and the corrected x_{n+1} as

$$\Delta x_{n+1} = -\frac{f(x_n)}{f'(x_n)}$$

$$x_{n+1} = x_n + \Delta x_{n+1}$$
(4.4)

Our equation was the (implicit) Kirchhoff law of the equivalent circuit, with ΔV (the voltage drop across IGZO) as implicit variable:

$$0 = -V_{bias} + \Delta V + R_{series}I(\Delta V) =$$

= -V_{bias} + \Delta V + R_{series}(I_{DT}(\Delta V) + I_{FNT}(\Delta V) + I_{TE}(\Delta V) + I_{FP}(\Delta V) (4.5)

For what concerns the samples used to tune our model, we considered D12, because of its good characteristics in combination with the precisely extracted film thickness, and D13, to understand the degree of characteristics "badness".

4.6.1 Sample D12

In fig. 4.11 both the simulated and experimental curves of sample D12 are show: in general a good agreement is visible as curves overlaps over the whole experimental current range, which means that the predicted current contribution are in general able to reproduce the real characteristics with a certain degree of accuracy (taking into account the fact that some parameters are assumed, but not exactly known, e.g. the contact area A_{eff}); the crucial choice of a IGZO electron affinity range (reported in the parameters table) give a "window" of curves that in turn allow to check the influence of Schottky barriers inhomogeneity: in fact, our range is centered on the value corresponding to a theoretical, perfect $\phi_{Pt} - \chi_{IGZO}$ barrier. This "widening" is introduced for a number of reason, which may require further study for this (but also other) particular sample:

• IGZO electrical affinity is usually obtained as a weighted average of In, Ga and Zn affinities, with weights depending on their stoichiometry (not known in our case);

- A number of studies show that stoichiometry is not uniform across IGZO, but a sort of segregation can happen at the surface for some species, for example Indium;
- Surface quality can influence the real Schottky barrier by means of trapped charges, dangling bonds, water or adsorbant.

In any case, these influences are not so important to completely change the magnitude of this physical quantity and so the shape of the curves, which in turn translates in a certain accuracy in the resulting I-Vs. At the end, simulations show



Figure 4.11: Simulated I-Vs versus experimental curves of 2 samples

that the main tunneling and field-related conduction mechanism are able to explain the electrical properties of the sample.

4.6.2 Sample D13

Results from simulation are shown in fig. 4.11b: what is possible to see is that there is a complete disagreement between the real characteristics and the simulated curves (based on the same parameters of D12, except the 2nm thickness that in fact should turn into very high non-linearity) both in terms of curves slope and threshold: what was previously understandable by sight, now is quantitatively plotted.

As mentioned before, the reasons of this behaviour could be explained in terms of bad film deposition and chemical uniformity, as well a sort of phases mixture, which in turn may change drastically a number of crucial electrical parameters such as the electron affinity and the effective mass; in any case, at this thicknesses the origins of IGZO conductive properties deriving from its band structure may be quite different, leading in this way to different conductin mechanisms.

Chapter 5

Results summary

5.1 Summary on Spinel/CAAC IGZO

Here we summarise the results obtained from a pool of 7 different Spinel/CAAC IGZO blankets with applications as TFT channel:

- Scanning mode C-AFM gives a number of figures of merit able to identify three main samples classes:
 - 1. PVD Spinel samples feature comparable leakage spot density over the surface; this information is supported by the current-per-pixel distribution curves, which feature the same behaviour over the whole current range (that reaches ~ 50 nA;
 - 2. PVD CAAC sample features a reduced number of surface leakage regions, indicating a more insulating behaviour; again, the current-per-pixel distribution confirms this fact by means of a constant decreasing slope of its curve, which however reaches currents in the range of ~ 200 nA;
 - 3. ALD Spinel samples are much more insulating (~ pA currents at $V_{\text{bias}}=2V$, with almost no leakage spot detected; the non conductive behaviour is confirmed by current distribution, with plateau between 1 and 20 pA;

In addition, PVD Spinel samples leakage density seems almost unaffected by the applied bias, while CAAC show a sort of proportional dependence on electrical stress magnitude;

• Spectroscopy mode C-AFM both confirms scanning results and give more insight for each sample:

- 1. PVD Spinel samples show bipolar conduction, with higher currents for high positive biases applied; in addition, a new information is achieved: D05 and D10 (featuring thicker GZO layer) are more conductive than D07 and D08 (featuring thinner GZO), confirming an expected behavior and proving C-AFM to be precise enough to detect such properties;
- 2. PVD CAAC sample show no current in positive biases, while higher current with respect to Spinel seems to be achieved in negative polarity;
- 3. ALD Spinel samples confirm scanning mode results being completely insulating in negative biases, and featuring very low current only at high positive biases;

Current-spread box-plots also show tip-unrelated instability in CAAC I-Vs for negative polarities; spinel ones are instead very coherent and overlapping;

In general, it can be said that our method of using C-AFM for material screening is able to obtain a range of figures of merit that allow coherent comparison among samples, also confirming expected results and behaviour obtained with full devices and simulations.

5.2 Summary on a-IGZO

For what concerns the 5 a-IGZO sample analyzed in this work, we can summarise the results obtained by spectroscopy mode C-AFM:

- I-V averaging show a general overlapping of I-V characteristics of D11,D12 and D14 (respectively Pt/IGZO 6nm, Pt/IGZO 4nm and Ru/IGZO 6nm), with high non linearity factor and high current densities even considering a worst case scenario electrical contact area;
- D13 (Pt/IGZO 2nm) instead shows poor I-V characteristics, suggesting that further improvements on film quality and deposition parameters are necessary to achieve functional layers at such thickness;
- TUNA and C-AFM sensors were both employed to study a wide range of currents, and we demonstrated the capability to merge coherently these results to obtain extended-current I-Vs of all samples;
- Current distribution box plots suggest a good electrical uniformity on both polarities for D11 and D14, with slightly wider spread in D12;
- Pt/IGZO 4nm was suspicious due to a behaviour very similar to the 6nm sample: the investigation with Scalpel C-AFM revealed a film thickness

 $\geq 5.5nm$; this is a sanity check for the C-AFM precision and capability to gather reliable information;

In addiction, a MATLAB code has been developed in order to simulate I-V characteristics of IGZO blankets taking into account both field-assisted and tunneling processes: results showed good agreement with experimental data for what concerns properly-working samples (D12); D13 simulations in turn completely disagreed with real characteristics, confirming the fact that in this 2nm sample the origin of conducting properties are not the expected one for a thin film (as in the other case) and suggesting intrinsic problems in the deposited film.

5.3 This work as basis for future ones

This work and the results obtained are obviously neither the best ones or the most complete from many point of view; on the other hand, the whole methodology and approach can be easily considered as a launching pad for a large number of future works and improvements.

The C-AFM has been shown to be a powerful tool to investigate and characterize IGZO blankets by extracting a number of figures of merit, thanks to the two ways - scanning mode and spectroscopy mode - we adopted for this task. Despite the limited samples available, we did our best to explore this approach and demonstrate that it is feasible for future implementation.

Because of this, an example of future work may be a wider study considering a large pool of samples, which however may be performed rapidly thanks to the "standardized" procedures that have been described - our fast screening methodology goal: this may include a-IGZO samples with a number of different electrodes, layer thicknesses and deposition process variability, or CAAC/Spinel IGZO deposited on a range of GZO interlayer, with different deposition temperatures and variable stoichiometry.

Another type of exploration may be attempted by considering other classes of insulating thin film materials, that in turn may confirm or even improve the comparison capability of C-AFM: for example, doped IGZO is a possible candidate for this analysis, that may pave the way to further improve composition understanding of AOS.

For what concerns TFT applications, an outstanding improvement may be obtained by developing as sort of back-gate biasing system which may allow to study the conducting properties of IGZO at different gate polarization: in this way, one may actually obtain a real MOS system with movable source electrode, so a even more generalized comprehension of the behaviour of the device could be obtained, together with the capability to compare this "naive" MOS with a fully processed one.

In general, all the methodology, workflow and post processing scripts developed for these tasks have been transferred to the Material and Component Analysis (MCA) group for future use.
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