Use of carbon nanotubes for passive radio-frequency devices

Cometto, Mathieu

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**NANYANG
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Use of Carbon Nanotubes for passive
radio-frequency devices

MATHIEU COMETTO

School of electrical and electronic engineering

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Abstract

As devices get more connected and communication increase in density, new challenges arise. The usual frequency bands used are getting crowded by the number of applications, and the relatively low working frequencies limit the available bandwidth and speed. Applications are now implemented in the GHz, but next generation devices shift towards higher frequencies into the millimetre band (30-300 GHz), seeking larger bandwidths, smaller devices and antenna size and improvement in spatial resolution. (High bitrate communications, Backhaul, radars, imaging, bio-sensors...)

These emerging devices have a need for passive framework for feeding and supporting while keeping the efficiency high, the dimensions short and the bandwidth large. The problematic of this thesis is to devise an innovative way to answer those needs.

Some solutions have of course emerged in the literature, and the plan here is to fork from those pre-existing results. Most solutions revolve around using vertically aligned micro/nano-structures to guide a wave or to reduce wave velocity. The idea is to introduce Vertically-Aligned Carbon Nanotube Forests (VA-CNT) into those designs, for they display inherently conductive anisotropy while improving from the previous solutions in terms of density and equivalent conductivity and ease of fabrication.

As passive devices cover a broad range of devices, the focus will be on some of the simplest devices, to pave the way for this technology. In a first step a simple slow-wave micro-strip transmission line using VA-CNTs is considered, which showcases the possibility to reduce significantly wave velocity propagation in the line, thus reducing the dimensions without the use of high-k dielectrics. Work on the topic demonstrate significant size reduction of devices at a given frequency.

In a second step, another type of transmission line is considered: Substrate integrated waveguides (SIW) and slow-wave SIW (SW-SIW). Waveguides large

dimensions' stop being a collateral at higher frequencies -with dimensions going below the millimetre- while exhibiting relatively better parameters per unit length than other propagation solutions. The feasibility of a SIW using VA-CNT forests is demonstrated through theoretical simulation and fabrication. A solution including a similar setup to the micro-strip line is investigated to assert the effects and possibility of slow-wave in waveguides using only bottom-up processes.

Finally, as an extension of the problematic, some diverse and more complex topologies are considered such as filters, antenna arrays feedings or interposers.

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List of Acronyms

CNT	Carbon NanoTubes
VA-CNT	Vertically Aligned Carbon NanoTubes
VLS	Vapour Liquid Solid
CVD	Chemical Vapour Deposition
LPCVD	Low Pressure Chemical Vapour Deposition
MFC	Mass Flow Controller
PECVD	Plasma Enhanced Chemical Vapour Deposition
TCVD	Thermal Chemical Vapour Deposition
DFT	Density Function Theory
EM	Electro Magnetic
FDTD	Finite Difference Time Domain
TM	Transverse Magnetic
TEM	Transverse Electro Magnetic
TE	Transverse Electric
GSG	Ground Signal Ground
SAC	SnAgCu, Tin Silver Copper.

CHAPTER 1: INTRODUCTION

Context

Figure 1-1: Frequency allocations (Source: US department of commerce, January 2016). Each line is a decade in frequency (0-300 kHz, 300 kHz-3 MHz, 3 MHz-30 MHz, 30 MHz-300 MHz, 300 MHz-3 GHz, 3 GHz-30 GHz and 30 GHz-300 GHz). Some very large bands are fixed for television and radio broadcast in the lower frequencies, while the higher frequencies have many more slots.

With the very steep increase of wireless devices and the volume of communications exchanged for any application, the bandwidth space has become more crowded than ever. Frequency modulation is the most common and used way of broadcasting signal in the air, and allows for many parallel communications. Each signal is modulated around a given frequency allowing for more resilience to noise transmission in comparison to other types of signal modulation: phase modulation and amplitude modulation. This led to a very particular and precise partitioning of the radio spectrum, as shown on Figure 1-1 for example. This way of splitting the radio spectrum is specific for the U.S. but can be generalized throughout the world with slight differences for every country. Miniaturisation and discoveries have allowed communications at higher frequencies but historically communications were done at

lower frequencies. As a matter of fact, it is always easier to transmit at lower frequencies over longer distances. These are the main reasons the lower end of the spectrum tends to be completely crowded. A common example of how detrimental this can be, is the way 2.4 GHz Wi-Fi signal gets disrupted every-time someone uses a micro-wave oven in the vicinity.

Allocated bandwidth also defines the speed of information transmission (Nyquist-Shannon theorem: maximum theoretical information rate is twice the bandwidth symbols per second. The amount of information in each symbol can be dictated with the Shannon theorem when including noise) and radio spectrum allocations are naturally wider at higher frequencies since the frequency band is in the thousand times larger (going from MHz to GHz).

Turning to less crowded (and thus less noisy and expensive) areas in the radio spectrum and seeking higher bitrates are the reasons most applications are now targeted towards higher frequencies. Backhaul, automotive sensors [1], bio-sensors [2], imaging [3], high bit-rate communications [4], next generation Wi-Fi and radar are examples.

Chipset of such devices are already produced industrially. All these new applications working in the 30-300 GHz band need a passive framework to be easily usable. Transmission lines to feed and connect them to the other devices on a chip or board, filters to process the signal and remove noise; passive devices are a requirement to have packaged functioning new applications. All these passive devices are designed for a given working frequency, and usually cannot be pushed out of their range without tremendous losses. Coplanar lines and microstrip lines are the to-go choice for many applications operating at lower frequencies for their ease of fabrication, and their tiny dimensions with acceptable losses. At higher frequencies, the dimensions of the lines tend to become unsuitably short, exhibiting high losses with all the dimensions becoming greater than the wavelength of the signal. For example, a

quarter-wave open stub operating at 150 GHz on a 350 μm thick silicon substrate would measure 150 μm long, 630 μm wide.

Problematic

In the light of this context, the natural question to ask is: how to devise an innovative way to answer the needs of all these new applications in terms of passive circuitry.

State of the Art

Some work has of course been done to address this issue: one of the main issue of the commonly used waveguiding devices is the lengths and the losses that become respectively too short and too high. For high power applications (and thus low losses) metallic waveguides were often chosen but the very large dimensions of those in comparison to the lines makes them unusable at lower frequency. At higher frequencies, their size reduces to a point where they become acceptable candidates for integrated systems (from more than 5 cm at 1 GHz to less than 1mm above 150 GHz) but inversely become very difficult to fabricate and incorporate in devices. Hence a good candidate for transmission of signal at these frequencies are the Substrate Integrated Waveguides (SIW): waveguides directly fabricated onto or into a substrate with dimensions dictated by typical 2D fabrication processes instead of 3D micro-machining. Their first apparition in scientific papers was in 2005 [5].

The concept evolved, as for example alumina sintered filters with top excitation [6] or slow-wave SIW [7]. Both these examples use top down approaches fabrication based on alumina substrates typically, either drilling/etching into an alumina substrate, metallizing the holes to shape a waveguide out of discrete metallic pillars. Top down approaches are most commonly used in mainstream processes for they are easier to scale, and cheaper. [8] shows results from a SU-8 photo-resist bottom up fabricated SIW air cavities. Other topologies for waveguiding were studied: Ferromagnetic slow-

wave micro-strip [9], metallic nanowires for slow-wave micro-strip [10]. These last two are focusing on slow-wave propagation in micro-strip transmission lines in the beginning of the millimetre band.

Methodology

Considering what has already been done in the field, the goal is to find another answer to the problematic which can solve some of the problems raised by the existing solutions: the resolution of the pillars for waveguiding and slow-wave effects are restricted by the aspect ratio of the vertically aligned nanostructures we fabricate. Here we are going to prove the concept of fabricating waveguiding devices using carbon nanotubes (CNT): Vertically aligned CNT (VA-CNT) forests are several orders of magnitude denser than the current state of the art of metallic pillar technology, and each CNT in the forest can reach extremely high aspect ratio. The methodology behind the work done here is to fork from the current literature: the topologies of SIW and slow-wave and to cross them with the state of the art in terms of carbon nanotubes fabrication.

Firstly, an introduction on the fabrication and properties of carbon nanotubes will be done. The novel material has been studied quite thoroughly never to be found an actual use in electrical and electronic fields: extensive research revealed extremely interesting properties and fascinating proof of concept have been published, yet hard constraints on the quality and position of the CNTs have made these ideas extremely hard to scale. The goal is to rely on seasoned fabrication method, while not relying strongly on the properties of the carbon nanotubes themselves, to ensure ease to transition from research to industry. In chapter 1, we will discuss about the different methods to produce CNTs, their characterisation and their modelling to use to answer our problematic.

Then, using the CNTs we can fabricate and model, chapter 2 will describe SIW solutions using CNTs as are fabricated and described through chapter 1. Several

rounds of fabrication and simulation are described to reach a conclusion on the possibility to use these mass-produced CNTs for waveguiding.

In chapter 3, we make use of the anisotropy feature of the VA-CNT, the idea is to demonstrate the possibility to enhance the results from chapter 2, to create slow-wave SIW (SW-SIW). Simulations on the matter prove the concept sound, but fabrication of devices is more problematic than for simple SIW. In addition, the CNTs were also used in slow-wave transmission line as was done previously in the literature using metallic pillars or thick nanowires. If simulations prove the concept to be sound, preliminary measurements raise question regarding the fabrication due to process issues. Other topologies are introduced to circumvent these.

Finally, in chapter 4 some other topologies are introduced and looked into to further complete the tools useable for emerging devices in the millimetre band, such as smart interposers, nano-antennas and filters.

Contribution to the domain of the research

Proof of concept of waveguiding using CNTs: design, simulation, fabrication and characterisation of SIW made using VA-CNTs.

Simulation work, design of novel devices using CNTs, especially Slow-Wave devices.

Design of excitations and optimization of transitions between different transmission media: mostly transition between common existing transmission lines to the newly designed CNT waveguides and lines.

CHAPTER 2: FROM CARBON NANOTUBES TO WAVEGUIDES

Carbon nanotubes

Carbon nanotubes were first reported in 1952 by soviet researchers [11], but the term nanotubes and their recognized fabrication and high resolution observation dates from 1991 using arc discharge and transmission electron microscopy by Iijima[12]. From 1991 onwards, research around carbon nanotubes flourished driven by the remarkable properties of the materials with potential outcomes in various fields of science and technology alike. Since then, CNT properties have been extensively studied, hinting to applications ranging from solar energy harvesting to lab-on-chip drug delivery. Their electrical and electronic properties make them a sought-after candidate for “more-than-Moore” transistors and devices. CNTs are also widely believed to be the material whose outstanding mechanical properties will allow construction of space elevators.

Structure and properties of CNTs can be better understood through graphene - another carbon material. Graphene is a single graphite layer, a 2D sp^2 bonded carbon atoms in a honeycomb lattice structure. CNTs can be seen as rolled up graphene sheets with typical diameters ranging 0.4-40 nm while conserving the hexagonal lattice which can thus be uniquely described by the chirality vector. This vector is defined by its two coordinates in the lattice space (as seen on Figure 2-2 A) namely (n,m) .

Figure 2-1: Left: graphene, Right: Carbon nanotube. Each ball is a Carbon atom, each link represents a sp^2 bond.

Figure 2-2: Chirality of CNTs. A: Schematics of chirality, B: scanning tunnelling microscopy (STM) images of different CNT chiralities [13], c d and e: Front view modelling of respectively (10,0) zig-zag, (10,3)chiral and (7,7)armchair CNTs

Carbon nanotubes can be either conducting or semiconducting depending on their chirality [14]: CNTs can be metallic (no-bandgap) or semi-conducting (and their bandgap is then dependant on their diameter from 0 to about 2eV). There are technically three types of CNTs according to their chirality: metallic CNTs are the armchair $n=m$ (named as such for the shape the lattice takes on a cross section of the nanotube). The other type of CNTs are either semi-conducting or semi-metallic if the band-gap is small enough for the tube to show a conducting behaviour at room temperature. The behaviour of those CNTs can be predicted with their chirality with a simple 3-congruence on $n-m$: if $n-m$ is multiple of 3 then the CNT is semi-metallic. Since semi-metallic are usually considered to be simply metallic and armchair CNTs have $n=m$, we can deduce that 1/3 of CNTs are metallic, while the rest are semi-conducting.

Typical production method of CNTs cannot impose a set chirality thus leaving on average 1/3 of the CNTs produced metallic.

Carbon nanotubes can also be nested, and are then referred to as multi-walled carbon nanotubes (MWCNTs) or sometimes just MCNTs for short, as opposite to their single counterpart single-walled carbon nanotube (SWCNT) or sometimes just SCNT for short. MWCNTs usually have larger diameters than SWCNTs and if only one of the nested CNTs is metallic, the MWCNT is too. This implies that the probability for a MWCNT to be metallic increases with its number of walls if we consider the chirality of each nanotube random. The probability for a MWCNT to be metallic can be found as $1 - (2/3)^n$, where n is the number of walls in the MWCNT. Some fabrication methods can yield a specific type of MWCNT (like 2WCNT), but the precise control over this parameter is hard and usually fabrication of MWCNT yields random number of walled CNTs hence MWCNT are treated as metallic.

CNTs have intrinsic high length to diameter ratio, with diameters ranging from 1 nm to the tens of nanometres and lengths up to half a meter [15]. Typical production method will yield CNTs with length ranging from few micrometres to several hundreds of micrometres.

Carbon nanotubes for RF applications

Since the declaration of Moore's law, a very intense search for smaller devices has driven the research on the use of carbon nanotubes for transistors [16]. Transistors made of CNTs were proven to have superior properties than silicon-based ones [17] and the advances in fabrication now permits the integration of specific quality CNTs with enough yield to make these technologies viable [18].

Quite recently CNT were used to fabricate an on-chip random number generator [19], which is useful to many cryptography and communication processes. Another anecdotic demonstrations of the use of CNT for RF was done in [20]–[22], using the

CNTs for demodulation and narrow-band amplifiers, used in practical conditions. CNTs were also used in antennas as loads [23] or conductors as CNTs can be metallic [24][25][26]. Other applications relying on the ink-jet printing of CNT-loaded conductive ink were done in [27], [28] to fabricate diverse RF circuits. Some more work was done on CNT antennas at higher frequencies, making use of the quantum effects of the CNTs [29] to design millimetre wave resonant antennas.

Fabrication of CNTs

The first observation of CNTs by Iijima [12] was on CNT produced using arc discharge: an electrical arc is discharged from a graphite electrode which produces diverse carbon products, including CNTs, fullerenes (self-terminated sp² carbon balls) and graphene. Arc discharge tends to yield high quality carbon nanotubes with few defects, but it also yields by-products and the CNTs are disordered and thus it requires further filtering to increase the yield. A very similar way of fabricating CNT is using laser ablation of graphite target [30]. The yield of this method is superior to the historical arc discharge, although more expensive. It is also possible to fabricate CNTs using chemical vapour deposition (CVD) with metallic catalyst, usually Fe/Ni/Co [31], and a carbon gas. Research shows that it can grow without external catalyst in some cases [32].

Chemical vapour deposition

CVD processes use vapour-liquid-solid (VLS) mechanism. The first ever reported growth of material with VLS (Silicon nanowires) dates from 1964 [33] in which Wagner and Ellis report the growth of silicon nano-whiskers using gold nanoparticles seeds. It is not before 1993 [34] that CNT were reportedly grown using CVD process with Iron particles seeds. CNT thus produced displayed radii from 50 to 200 Å depending on the seed size, and lengths up to 50 µm.

Since then resources and time invested in VLS are incredibly high, and processes evolved and stemmed a lot from this. Recent CNT growth using CVD had reported lengths exceeding 50 cm [15] in 2013. If VLS mechanism is still partly poorly or not known yet, the decades of work helped better understand and optimize the mechanism and the processes using it ([35] features a live TEM video of the growth process). To roughly summarize the VLS mechanism: a liquid metallic seed is produced by heating a metallic layer. A gas containing the wanted material is introduced in the chamber (Carboned gases, such as C_2H_2 or CH_4 for carbon nanotubes). The gas is adsorbed on the liquid seed, and the atoms of interest are dissolved in it creating a binary (or ternary) compound, until reaching precipitation point and start nucleation of a crystal, either at the bottom or the top of the seed.

Vapour-Liquid-Solid mechanism

The process of VLS formation will be discussed, which will provide insight on the growth of CNTs and the parameters of interest to control the growth of the CNT forests that will be used for the devices later. As opposed to some VLS-based processes, the CVD used for CNT growth (in between low-pressure CVD [LPCVD] and cold-wall CVD) uses only cold gaseous precursors (instead of solid precursors) which improves repeatability and parametrisation of the growth. The VLS mechanism will be focused on carbon nanotubes fabrication.

The three main steps of VLS growth are: induction, nucleation and growth. These steps start after the catalyst is liquefied under H_2 atmosphere and stabilized in the form of droplets that will act as the seeds. Afterwards, the carbon source gas is introduced (typically C_2H_2 or C_2H_4) with the correct conditions for the eutectic point of the seed, the source decomposes releasing H_2 in the atmosphere while leaving C atoms dissolved in the seed. Nucleation is the point where the carbon atoms reach their super-saturation point, and start precipitation into solid carbon. This precipitation will determine the nature of the product, which can be single walled crystalline CNT, multi-

walled CNT with varying degrees of graphitisation, or purely graphitic nanofiber. Then the growth is the decomposition of the precursor continuing to fuel the precipitation of carbon, arranging itself on the existing nanostructure.

The size of the resulting nanostructure is related to the size of the metallic seed size. The usual way to generate the metallic seed is to deposit a flat thin layer of catalyst (between 0.4 nm to 20 nm) and to heat it past the melting point. The metal will melt and arrange itself in droplets of rather homogeneous radii and disposition on the substrate, depending on the material, the thickness of the layer and the vapour phase pressure of the catalyst and feed gas [36]. Typically, the resulting nanostructure will have a radius shorter than that of the metallic seed. The difference can be estimated by looking at the interfaces tension (liquid solid, liquid vapour and solid vapour) [37], [38]:

$$R = r \sqrt{\frac{1}{1 - \left(\frac{\sigma_{ls}}{\sigma_{lv}}\right)^2}} \quad \text{Equation 2-1}$$

where σ_{ls} and σ_{lv} are respectively the liquid-solid and liquid-vapour interface energies, R the radius of the metallic seed and r the radius of the resulting nanostructure.

The growth speed can be inferred from the flux of atoms adsorbed at the surface of the seed, minus the flux of atoms desorbed from the surface and the flux of atoms diffused at the surface of the seed. The flux of atoms adsorbed can be described by Hertz-Knudsen:

$$\Phi_{ads} = \alpha * \frac{P_g}{\sqrt{2\pi m_g k_B T_{vap}}} \quad \text{Equation 2-2}$$

where Φ_{ads} is the flux of atoms adsorbed at the interface of the seed, α the effective adsorption coefficient, P_g the partial pressure of the precursor gas, T_{vap} the temperature of the vapour phase, m_g the mass of the precursor gas molecule and k_B the Boltzmann constant. For a seed at thermodynamic equilibrium, the desorption and

adsorption rates are equal. For the growth to happen, a supersaturation is needed so that the adsorption is predominant over the desorption. The pressure of the gas can be given by the Gibbs-Thomson equation:

$$P(r) = P_{\infty} \exp\left(\frac{2\Omega_l \sigma_{lv}}{rk_B T_G}\right) \quad \text{Equation 2-3}$$

where Ω_l is the volume of the seed, T_G the temperature of the gas and P_{∞} the vapour tension for an infinite diameter seed and other parameters the same as before. From this pressure, we can infer in the same way with the Hertz-Knudsen equation the desorbed flux of atoms:

$$\Phi_{des} = \frac{P(r)}{\sqrt{2\pi m_g k_B T_G}} \quad \text{Equation 2-4}$$

and thus, by equalling $\Phi_{ads} = \Phi_{des}$ a critical radius for growth is extracted:

$$r_c = \frac{2\Omega\sigma}{k_B T \ln(P/P_{\infty})} \quad \text{Equation 2-5}$$

In this case, the atoms diffused from the surface are predominant for the growth speed of the VLS mechanism. This also implies that the nucleation phase and growths are faster the larger the size of the particles, which is experimentally verified for CNT growth in [35].

These results mostly show that the parameters that we can tweak for the growth mostly are the pressure of the gases during the growth, the radii of the catalyst droplets (the metallic seeds), the nature of the metal used as catalyst, the partial pressure of the precursor gas and the temperature of the seed and of the gases. As a matter of fact, the CVD machine on which the growth work was done has a localized heater: it's not exactly a cold-wall CVD since the walls do not have a set temperature, but their temperature (and by approximation that of the gas) is much lower than that of the substrate. Hence, it is not possible to control that temperature and even difficult to

estimate it. As for the catalyst type, extensive work was done in [31] to compare different catalyst types and came up with the widely accepted conclusion that Fe is the best catalyst for CNT growth. As for the radius of the seeds, it is directly linked to the thickness of the catalyst layer deposited which was taken in most -if not all- cases to be 1 nm. Some work was done on the influence of the catalyst seed size versus the quality and the length of the growth [39], showing that smaller radii CNTs growth tended to yield better quality and longer CNT pointing towards the lowering of the thickness of the catalyst layer to about 0.4 nm. Yet in our use of the VA-CNT forests, thicker CNTs have the advantage of being more likely to be multi-walled, which increases the equivalent conductivity of the VA-CNT forests. Thus, the main parameters of interest for the growth here were the growth temperature, the growth pressure and the partial pressure of the precursor gas.

CVD recipes

The CVD machine used for the growth is an AIXTRON Blackmagic system. As stated briefly before, the heating source is directly located below the substrate, with the gases input located above the substrate to ensure a vertical gas flow on the substrate. The system has 4 inputs with mass flow controllers (MFCs), the max temperature of the heater is 1000 °C, and the temperature ramp up can reach a hundred of degrees per second and while there is no dedicated cooling system to ramp down the temperature, the fact that the chamber is not heated during process allow temperature ramp down relatively fast in comparison with usual tube-CVD systems (at most a few minutes). The system is fed with 7 gas lines and 4 MFCs, to leave choice among the gases to use: H₂ or Ammonia for diluting gas, N₂ or Ar for neutral gas, C₂H₂ or C₂H₄ for precursor gas and O₂. The system supports “high-pressure” processes, with pressure up to 800 mbar, and is equipped with a plasma controller for plasma enhanced CVD (PE-CVD).

Figure 2-3: AIXTRON Blackmagic system. a: system. b: Close view on the substrate holder and heater, with the quartz showerhead for gases input above.

These available parameters are used for three main type of recipes that are useable on the machine: PECVD, Supergrowth and thermal CVD (TCVD).

PECVD

PECVD use electrodes to generate plasma out of the precursor gas, using Ammonia as a diluting gas to control the partial pressure of the precursor and 10 μm to 20 μm thick Ni catalyst layers. The plasma usage will yield very straight VA-CNT of very large multi-walled CNTs, of heights up to a few dozens of microns. The very low density and height of the CNT yielded with this method are not useable for the purpose we seek here, but can be used for RF applications e.g. [40] for THz interconnects. Temperatures are usually slightly lower for PECVD processes (around 500 $^{\circ}\text{C}$)

Supergrowth

Figure 2-4: a: Scanning electron microscopy (SEM) imaging of supergrowth CNT on silicon substrate. Length of about 1 mm (the scale on the picture is taken with a slight tilt). b: Raman spectrum of the Supergrowth CNTs. D & G peaks ratio indicate good crystallinity in the CNT.

Supergrowth recipe make use of the high-pressure capability of the system. The growth is typically carried out at 720 mbar, while the partial pressure of precursor is kept relatively low (down to gas ratios: 500:500:3 for H₂:N₂:C₂H₂). Temperatures are usually around 700 °C for these processes which yield -as the name implies- very long CNTs with very fast growth: 1 mm for a growth time of around 120 s. Raman spectra (Figure 2-4) show relatively good quality of carbon nanotubes, mostly single walls, with very small radii, heralding very high density (estimated between 10¹⁵ and 10¹⁶ m⁻²). On the SEM imaging zoomed in Figure 2-5 we can see the CNTs are not very straight, but are generally straight, while being tortuous. Yet on the background we see rather straight bundles of CNTs. It is difficult to judge the density on these pictures alone, as the used SEM's resolution is above the expected diameter of the CNTs. On the right part of Figure 2-5 the top of the CNTs seem to present micro-scale roughness, while being rather smooth, and even slightly shiny to the eye. It is of importance to note on the SEM imaging of Figure 2-4 that the VA-CNT forest delaminated from the Silicon substrate, which can be dangerous for devices, yet the adhesion of the layer is somewhat better on gold (not represented here). We can assume the diameter of the

CNTs are smaller than other recipes due to the higher pressure of the chamber, leading to better quality and denser forests.

Figure 2-5: SEM imaging of supergrowth CNTs. a: Zoom from the side of the grown CNTs. b: Zoom from the top of the grown CNTs.

Figure 2-6: Recipe example for Supergrowth. Parameters evolution in the time. H₂ MFC is faulty, thus requiring a 273 set to deliver 200 sccm. Pressure ramp up is slow for safety reasons, a too fast pressure increase triggers the chamber seal interlock and stops the process.

Typical recipes vary, but an example can be seen on Figure 2-6. Growth time is 1800 s in the process time, yet effective growth takes less than that. Tests showed that the growth stalls after only a few minutes. The ramp-up of the pressure to 720 mbar is long to ensure good isolation and purity of the atmosphere inside the chamber. The temperature of the substrate started quite high due to previous processes and the

absence of a cooler on the heater, but the ramping up is very fast. Some recipes exist with an anneal time at lower temperatures before growth, below melting temperature of the catalyst to reduce the metal-oxide that may have formed on the catalyst layer.

TCVD

The process we call here TCVD differs from what is typically called so in the literature: TCVD is usually for tube CVDs where the whole chamber is heated. In our case TCVD refers to the basic recipes at rather low pressure, without the use of plasma.

As opposed to the two previous growth methods, TCVD uses no plasma, and the pressure is kept below 100 mbar. Growth can take place at temperatures ranging from 500 to 800 °C, depending on the catalyst. Then again, most the growths were done with Fe catalyst of 1 nm thickness on a 10 nm buffer layer of Al₂O₃/Al on silicon or gold on silicon.

Recipes used could yield up to 800 µm in maximum height with relatively large variance on the high across the sample (~100 µm variation). Uniform height of 750 µm was obtained on 1 cm x 0.5 cm test samples. Uniformity on larger substrates was somewhat difficult to reproduce over time: on 2 x 2 cm samples, the growth would be very good on the first samples, then the uniformity would degrade, probably due to the heating of the chamber.

Measurements were made on the density and size of these nanotubes, which found them larger and less dense than that of supergrowth grown CNTs (estimated between 10^{13} to 10^{14} m⁻²). Raman spectrum Figure 2-7 also show the quality (crystallinity) of the CNTs dropping with this method. According to [35] and [39] this is due to the diameter of the CNTs influencing on the mechanical constraints on the CNTs during the growth, introducing defects. Since we are here more interested on the equivalent conductivity of the bulk of the forest, this effect is compensated by the multi-

walled aspect of the CNTs (virtually removing the $1/3$ factor to the conductivity coming from the chirality of the CNTs).

Figure 2-7: Example of TCVD grown CNTs. a: SEM imaging, low zoom, tilted 4 ° from side. Measured height: 680 µm. b: Raman spectrum on top of the VA-CNT forest. Signature is typical of multiwalled CNTs with some defects. c: Zoom in view of side of the sample, where a part of the bulk CNT was peeled-off with a tweezer (scale 20nm). d: Zoom in on the surface of the CNT as grown. The surface seems to have more macroscopic unevenness than Supergrowth CNTs.

Yet again, it is difficult to judge from Figure 2-7 bottom left the actual diameter of the CNTs from this SEM imaging, for this is the resolution limit of this SEM, but it shows well in accordance with [35] and [39] that the CNTs have many buckles. For RF applications, the scale of these imperfections is much lower than that of the wavelength (in the millimetre range), thus will not introduce more than possible slight losses.

Of course, growth tests were mainly done on silicon substrates, but growths on gold proved that they are not changed by the surface material the growth takes place on, the buffer layer apparently is sufficient to have the growth unaffected by the surface material. Yet, the surface condition, mostly roughness, had great impact on the overall quality of the CNTs. Large unevenness of the surface lead to likewise large unevenness of the top surface of the CNTs. Furthermore, the roughness at a finer level tend to yield shorter CNTs, and less repeatable recipes, with yields dropping from ~80% of successful growths to less than 50%. This is believed to be due to the catalyst droplets failing to create a homogeneous repartition of same-size seeds, generating growth patterns looking like flowers as seen Figure 2-8 with a hollow centre and petal-like growth on its side, probably due to the catalyst forming a large droplet in the middle, and the growth thus happening only on the sides, where the surface tension allows adsorption, locally having a supersaturated catalyst. If not useful, these patterns are at least interesting to look at.

Figure 2-8: SEM imaging of typical defective growth due to surface roughness of the growth substrate

Figure 2-9: Recipe example for TCVD growth of VA-CNT forest. N₂ only used for cooling down, actual setpoint at 2000 sscm. As for Figure 2-7 , H₂ MFC faulty, set at 804 to reach a 700 sscm flow. Pressure set at 80 mbar for whole process. The actual pressure oscillates a bit during the ramp up but stays stable during the growth.

Typical TCVD growth process is short. Figure 2-9 shows the parameters evolution for process yielding VA-CNT forests on a 2x2cm wafer of approximately 300 μm tall. Effective growth process starts directly when the precursor gas is introduced. The flows are relatively high; thus, we can assume the changes in gas in the chamber are immediate.

It is possible to witness the start of the growth directly after introducing the precursor C₂H₂ in the chamber as picture in Figure 2-10. The catalyst is patterned on a gold metallized surface, and changes colour when the C₂H₂ is released in the chamber and the change is almost instant across the sample. This happens around 654 °C, and is clearly visible in the chamber (slightly less on silicon substrate without gold, because these are slightly more glowing than those with gold). 654 °C is also the temperature at which the C₂H₂ is released in the chamber, which suggests the C₂H₂ is responsible for the change in colour of the catalyst layer.

Figure 2-10: Change on the catalyst right after the introduction of C_2H_2 in the chamber (happening around 654 °C). The catalyst was patterned on a 1 μm thick gold metallization. The catalyst immediately changes to the colour of the carbon heater.

Figure 2-11: Height of CNT from TCVD growth versus growth time

Results from [39] & [31] seem to indicate that the length of CNTs during CVD growth follow an asymptotic law, not starting directly after introduction of precursor gas, slowing down over time until reaching a stop point. According to these models, we tried fitting CNT growth from BlackMagic system as in Figure 2-11. Experimentally the height seems indeed capped, but the growths over 5 minutes had quite a large disparity of height from sample to sample, and on the samples themselves. The height of the forest would vary by 200 μm from one side to the other of the sample. The growth is very fast

for the first few hundred of microns and more repeatable. As stated before, the extended use of the machine tends to heat up the quartz chamber, changing the conditions of the growths.

Figure 2-12: 600 s growth example. a: Side view, tilted. b: Zoom on the ridge of the wall (19000x). CNT height 750 μm.

Simulation bulk equivalent model

A large portion of the work done for this thesis required simulation work. Most of the simulations were done using finite element modelling via ANSYS HFSS. HFSS is a widely used RF design and simulation software for devices working up to the THz. The simulation setup requires one or multiple ports, which are in practical terms the input/outputs of the RF devices. HFSS uses Finite Element Method (FEM), Method of Moments (MoM) in addition to their patented Domain Decomposition Method (DDM). The resolution principle is to decompose the entire simulated area into small sub-domains in which the Maxwell equations are simplified. The size of these elements will dictate the precision of the simulation at the cost of computation power needed. The advantage of this software lies in the fact that it handle the meshing of the entire simulation with tetrahedrons with great variance of size, and uses different method of solving depending on the size of the tetrahedrons. It is possible to impose conditions

on the shape, and density of the meshing with some finer tuning, but the general method works this way: a rough meshing is produced in all the different parts of the simulation space, an S-matrix (the transmission parameters between the different ports) is computed, then, the meshing is refined and the process starts over until the variation of the S-matrix reaches a threshold defined by the user. The actual refinement of the meshing is localized in the different regions of the simulation space, to focus the computation power on the spots of interest, and this is handled by HFSS algorithms. Typically, RF devices have a very large discrepancy in sizes, with elements having several orders of magnitudes between their different dimensions (e.g. a transmission line can be centimetre long, a few hundreds of micrometres wide, and a few micrometres thick) which leads to very large discrepancy in mesh size.

Yet it is computationally impossible to design and simulate each CNT individually for three reasons: the very large number of CNTs, the very large aspect ratio of the CNTs and the very large difference in scales in RF devices. All three reasons leading to insolvably large number of elements in the meshing. This is furthermore exacerbated by the unique electrical properties of the CNTs: quantum capacitance and kinetic inductance, effectively giving CNTs very interesting RF properties. An equivalent bulk model was done in [41] including the quantum effects in the carbon nanotubes.

Other methods exist to simulate accurately CNTs, mostly relying on Density Functional Theory (DFT). The problem with these methods lies in the difficulty to conciliate with the usual large size difference in RF components, reaching in the centimetre scale.

Model

The model here was made taking into account several other models of CNTs. The work in itself can be found in [41]. The model, simply dubbed Bulk equivalent tries to fill a gap left by the others models specifically for RF applications. Typically, DFT

[42]–[46] is one of the most accurate method to simulate CNT's electrical behaviour. It provides quantum treatment of the structure by solving iterations of the electron density function. The main issue with this method is the range for which the computations are still possible to solve, typically solving systems of a few nanometres take in the hours of computation time on personal computation machines.

The Maxwell-Schrödinger approach [47]–[52] is to solve Maxwell and Schrödinger equations while trying to keep results consistent on both models. This allow quantum mechanical treatment of the CNTs while staying consistent with electromagnetic (EM) environment. Technically, this uses an EM framework set by Maxwell equations in which finite difference time domain (FDTD) approximate solutions of the Schrödinger equation are solved. If the scale of this solution is somewhat larger to DFT, it stays under the dozen of CNTs, and requires custom implementation.

Effective surface conductivity [53]–[58] models carbon nanotubes as hollow tube with complex conductivity derived from Boltzmann kinetic equation and tight-binding theory. The model is based on Hanson update to the conductivity of CNTs, having the advantage of nesting the quantum considerations of the CNTs. The problem of this method is that it relies on the special wavenumber which makes the implementation difficult.

Other ways of modelling CNTs is to build a circuit model of CNTs based on Luttinger liquid model [59]. This covers SWCNTs but was extended to MWCNTs in [60]–[62]. Knowing that transmission lines can be described as a series of lumped RLC two-port segments of infinitesimal length [63].

Starting mainly from the effective surface conductivity model, and applying it to the infinitesimal approach, a CNT circuit model was produced. This model encompasses SWCNT and MWCNT. This implementation is already useable in S-parameters analytic software, such as Agilent's ADS. But for radiation and field imaging,

this model is still not useable with finite element modelling software such as HFSS. CNTs are typically bad for RF because of their poor efficiency and high impedance which can be overcome with the use of bundled CNTs.

The model used in this thesis here works as follows: consider an array of CNTs in a defined small area of space (smaller than the considered wavelength). Consider the CNTs as hollow tubes and use the effective surface conductivity model to extract a complex conductivity. Then this conductivity is averaged on the whole bundle. From there it is possible to model a larger bulk using a lumped method as described before.

The model was then retro-fitted using existing experimental results in the literature of RF measurements on CNTs to enhance the precision. The advantage of this model is to have a low computational cost relative to the number of CNTs considered, to convene very well to RF simulations, as it doesn't reduce the lower end of the size scale of the simulations (which is a reason why it has a low computational cost), it is possible to export on any EM solver that has a 3D modeller and custom defined materials. The other interesting point is that the model is valid even when the quantum effects are predominant (low number of CNTs). Yet the model has limitations too: the precision is still way lower than the models relying on Schrödinger equations, it becomes void for nanometre-scale problems and it hasn't been confirmed by specific experimental work (as in, devices specifically made to prove the model).

Conclusion

With the objective of replacing state of the art techniques to make integrated RF passive devices, and mostly transmission devices we intend to use carbon nanotubes. If single CNTs properties are incredible for electronics, mechanics and even optics, they never really broke through as their properties are highly dependent on their physical topology: their radius, length, quality and chirality. The electrical properties of CNTs can be improved by relying on high densities, as the resistivity scales down and

the physical topology averages thus forming a bulk material with sufficiently high conductivity.

Of the different fabrication methods of CNTs, Arc discharge and Laser ablation yield the high quality of CNTs (with the least defects in the lattice), yet the produced CNTs are disordered, carbon by-products are also created and the size of the CNTs is not consistent. CVD methods on the other hand tend to produce CNT with more control over their length and position, yet their quality is usually lower. Among the different methods of CVD, it is possible to grow CNTs horizontally in tube-CVDs, or denser vertically aligned forests in TCVD, PECVD or supergrowth. Among those three, supergrowth yields the longest and thinnest CNTs (and thus densest forests). PECVD yields very straight large CNTs, but short. TCVD CNTs are somewhat in between.

Parametrisation of TCVD and supergrowth CNT growth was done, for the length, density and quality of the VA-CNT forests on different substrates is important for the work done afterwards. The designing of RF devices was done on HFSS using a model for CNT bulk modelling.

CHAPTER 3: SUBSTRATE INTEGRATED WAVEGUIDES

Transmission of information is arguably the most simple and important RF application. Transmission lines can be used to guide the energy and information from generator to load. Waveguides are a specific type of transmission line but are different to typical transmission lines (such as micro-strip lines and coplanar lines) in that they can support several modes of propagations, the transmission doesn't have to be only transverse electro-magnetic (TEM). Furthermore, at the frequencies we are considering (microwave band, 3-300GHz) when transmission lines start becoming inefficient and lossy due to skin effect increasing current densities and dielectric losses increasing, waveguides keep low signal attenuation. Due to geometric constraints, waveguides will act as high-pass filters: below a geometry-dependent cut-off frequency the waveguide will not propagate, as opposed to transmission lines operating from DC to high frequencies. As stated, the cut-off frequency is geometry-dependent but waveguides can never transmit DC frequencies and exhibit dimensions too large for practical use at lower frequencies.

Figure 3-1: Example of metallic waveguides [64]

Topologically, waveguides are hollow metallic conductors filled with a dielectric propagation medium, and although they can be shaped arbitrarily they are usually rectangular or circle shaped [63]. Most of the waveguides studied here are rectangular with dimensions (a,b), in which case it can be found that the cutoff frequency for a $TM_{m,n}$ (transverse magnetic)(Magnetic field orthogonal to propagation) wave follows:

$$f_c = \frac{u'}{2} \sqrt{\left(\frac{m}{a}\right)^2 + \left(\frac{n}{b}\right)^2} \quad \text{Equation 3-1}$$

f_c in Hz. Where u' is the phase velocity of the wave in the propagation medium, (m,n) are the mode numbers of the incident wave which are both in $\mathbb{N}$ but cannot be both zero. Visually, these m and n numbers represent the number of half periods of the standing magnetic wave in the cross-section plane of the waveguide (m along the a-dimension, n along the b-dimension of the waveguide). The cutoff frequency is dependent on the mode of propagation of the wave in the waveguide, yet it is often implied when referring to cutoff frequency of a waveguide to mean the cutoff frequency of the dominant mode of the waveguide, which is the lowest cutoff frequency of all modes: usually for $TM_{1,0}$ or $TM_{0,1}$, depending on the longest dimension between a and b.

From here onwards, unless otherwise stated the medium of propagation is considered to be air ($\epsilon=1$, $\sigma=0$), and thus $u' = c$. In this case, typical widths for waveguides are 30 mm for 5 GHz cutoff, 6 mm for 25 GHz cutoff, 3 mm for 50 GHz cutoff and 1.5 mm for 100 GHz cutoff. As for the height, it will be kept below half the width to ensure no parasitic mode below the dominant mode. It shows that at higher frequencies dimensions of waveguides stop being a collateral.

Introduction on SiW

Ultimately, the goal is to showcase passive devices that can be used for applications, as waveguides are hardly usable as they are bulky being carved from

metal, even though the cavity dimensions can drop below few millimetres at higher frequencies. Integration is difficult with typical metallic waveguide, which is why we will focus here on SIW. As the name implies, the waveguides here are directly integrated in or on top of a substrate with the associated transitions to feed devices (usually micro-strip or coplanar lines to waveguide).

CNTs' use for guiding

In most cases, the main disadvantage of using waveguides and SIW is their large size when compared to transmission lines. Apart from working at higher frequency, it is possible to reduce the size of devices by reducing u' in Equation 3-1, which is done by increasing ϵ of the propagation medium. This does not come without cons, as the propagation media other than air display higher losses. For SIW, the guiding of the wave is done with the use of pillars smaller and closer together than the wavelength of the signal to propagate. Since the SIW is fabricated using discrete pillars, the size of the pillars and their spacing will define the resolution with which the SIW can be fabricated. Current state of the art methods are limited in aspect ratio of said pillars, and on their density. Using CNTs in this technology would allow to reach higher aspect ratios, thus giving more control over the quality factor and working frequency of the devices. In addition, the very small radii of CNTs give increased precision in the patterning of the devices, allowing more complex shapes and more continuous designs (as opposite to having to use pillars of diameters comparable with the device size).

High conductivity, high density

The density of VA-CNT forests has been estimated to reach up to 10^{16} tubes per square meter [65] removing constraints of SIW patterning, as it will be lower than the resolution attainable through the patterning methods of the catalyst (typical lithography processes). Moreover, due to the CNTs of the being mostly vertical, the aspect ratio of the patterns are limited not by the radii of the individual CNTs but by the

size of the patterns produced. It is thus possible to have straight walls made of 1-5 nm wide CNTs 600-800 μm long if the wall at least around 50 to 100 μm wide.

Considering the effective density (Only that of Metallic CNTs, which is at worst 1/3 that of the full forest) the conductivity can reach $3 \times 10^6 \text{ S/m}$ which is approximately one tenth of gold. In more real case scenario, the density will be usually lower depending on the growth method used, but the CNTs will be predominantly multi-walled, which is why density was chosen arbitrarily to vary from 10^{15} tubes per square meter (equivalent to 100 tubes per square micro-metre). The objective was to find the limitations on the density starting from an expected value of the density. For this purpose and using the model from [41] a very simple and perfect waveguide was modelled in ANSYS HFSS.

Density variation in perfect waveguides

Figure 3-2: Schematics of waveguide simulation. a: Schematics front view, b: Schematics isometric view, c: Electric field in the propagation medium, top metallic cover hidden. (Arbitrary logarithmic units)

The simulation setup uses two perfect conductive planes on top and below and two VA-CNT walls thus creating the waveguide in between. Each of the ends of the waveguide is terminated on a waveport, to only assess the losses due to the waveguide. The capping being set to perfect metallic conductors, no field leaks through these surfaces, the only losses are thus the wave leaking through the walls, the attenuation in the walls, and the attenuation in the air. The latter is ignored and the air is set to have a $\tan \delta = 0$. The waveport setting in the simulation is seen in green on Figure 3-2 (top), covering both the propagation medium and the walls. This introduces a problem in the simulation as the Bulk Equivalent Model for the CNTs has some frequency dependent parameters which can mathematically make the material as left handed, and HFSS is designed to not let the setting up of ports in left handed material. To enable simulation with this setup, a buffer right handed material was used (in grey in Figure 3-2 (top right)), set to be anisotropic gold: the conductivity equals that of gold ($4.7 \cdot 10^7 S/m$) only in the z direction and 0 otherwise. To ensure the impact of this buffer layer is minimal, it was kept as small as possible without impacting too much the computational complexity of the simulation (the smaller the elements, the smaller the meshing, and thus the higher the number of mesh elements and the computational weight of the simulation) in addition to preliminary simulations with its size varied to check the impact (variations in thickness from 10 to 100 μm changed the transmission parameter of the waveguide by less than 0.01 dB).

Figure 3-3: Transmission parameter (S21) for perfect waveguide designed for 50 GHz cutoff. Density of walls parametric. Waveguide fixed parameters: $a = 3 \text{ mm}$, $b = 300 \mu\text{m}$, $L = 10 \text{ mm}$, $WallsT = 200 \mu\text{m}$. Density expressed in tubes per square metre. metallic walls reference not plotted, superposing perfectly with 10^{15} m^{-2} density plot.

The transmission level stays at acceptable levels when the density is above 10^{13} m^{-2} , and is perfect for densities 10^{15} m^{-2} and above. We can notice another effect at lower densities, starting from 10^{12} m^{-2} : the cutoff becomes smoother, extending in the lower frequency. This red-shift happens because the walls are not conductive enough and the condition for Equation 3-1 is that the walls are perfectly conductive. In this case we need to factor in the penetration of the wave in the material that we can estimate with the skin effect formula:

$$\delta = \frac{1}{\sqrt{\sigma\mu\pi f}} \quad \text{Equation 3-2}$$

where σ is the conductivity of the material, μ the permittivity of the material and f the frequency of the E-M wave. δ [m] is parameter of the real decreasing exponential describing the intensity of the fields in the conductive material. We can see that the lower the conductivity, the bigger the skin effect, which translates in our case that at lower densities we cannot use Equation 3-1 with a since the effective value is larger. The effects are more intricate than only this, but explain roughly the behaviour of the cutoff with lower densities CNT. Although this effect can be interesting, the transmission

levels are too low to be of use in RF devices, hence VA-CNT forests density will be targeted to be above 10^{13} m^{-2} at least, 10^{15} m^{-2} for optimal results.

The other parameter of interest is the CNT walls size: the conductivity of the VACNT forest is much lower than that of metals. The anisotropy of the material also makes it hard to evaluate the penetration of the wave base on solely skin length: the skin length based on the equivalent conductivity of the VACNT forest used in the simulation model would be:

Table 3-1: skin length for different effective conductivities tied to VACNT densities at 50 GHz. 5 skin length is the length for 99% of the evanescent wave attenuation. The red shift is the shift of the cutoff towards lower frequencies.

Density [m^{-2}]	Effective σ [S.m^{-1}]	δ [m]	Skin length (5δ) [μm]	Expected Red shift [Hz]
1.00E+10	9.03E+00	8.40E-07	4.20E+00	1.40E+08
1.00E+11	9.03E+01	2.66E-07	1.33E+00	4.42E+07
1.00E+12	9.03E+02	8.40E-08	4.20E-01	1.40E+07
1.00E+13	9.03E+03	2.66E-08	1.33E-01	4.42E+06
1.00E+14	9.03E+04	8.40E-09	4.20E-02	1.40E+06
1.00E+15	9.03E+05	2.66E-09	1.33E-02	4.43E+05

Under these conditions, the maximal red shift observable should be 140 MHz for the lowest density of CNT, yet we can see on the simulations that this shift is several GHz. The increased red-shift effect can be explained by the anisotropy of the CNT material. The studied propagating mode here is the transverse electric mode (TE_{10}) and thus the electric field is aligned in the same direction as the carbon nanotubes while the magnetic field is in planes orthogonal to the CNTs. The bulk of the VACNT forest defines an anisotropic conductive medium. In the case of the simulations, the anisotropy is chosen to be perfect, with no off-axis conductivity.

Figure 3-4: a&b: EM fields at 75GHz in the 50GHz-waveguide with 200 μm thick walls of 10^{12} m^{-2} density CNTs. a: Electric field, b: magnetic field. The magnetic field propagates in the walls, when the electric field doesn't. c&d: EM fields in the waveguide with 200 μm thick walls of 10^{15} m^{-2} density CNTs. In this case the magnetic field is correctly confined in the waveguide.

From Figure 3-4, it's useless to check the walls thickness effect on high density carbon nanotubes: larger values of the variable change nothing to the transmission parameters, and if lower values can impact the transmission parameters, it is very hard if not impossible to fabricate extremely thin walls. Hence in this perfect case scenario the walls thickness has little to no impact on high density walls. The effect of the walls thickness can be better observed with lower density (Figure 3-5).

Figure 3-5: Transmission parameter (S_{21}) for perfect waveguide Designed for 50 GHz cutoff. Thickness of walls parametric (in μm). Waveguide fixed parameters: $a = 3 \text{ mm}$, $b = 300 \mu\text{m}$, $L = 10 \text{ mm}$, $\text{CNT density} = 10^{12} \text{ m}^{-2}$. Losses increase past 100 μm but are constant above this value.

To conclude on this part: it is very important that density stays above 10^{13} m^{-2} for the waveguide to display correct transmission parameter. At this density and above, thickness of the walls does not impact the transmission in the range of what is possible to fabricate: it is difficult to confidently produce clean VACNT structures with aspect ratio above 5.

Excitation of waveguides

The next step, is to devise a way to excitate waveguides. In the literature the most common excitation for SIW is the transition from micro-strip line to waveguide ([66],[7]) , and more generally transition from top to bottom (shorted coplanar stub [6], [67]), which is more easily done given the top-down fabrication approach of the waveguides. These approaches are not usable in our current topology since the propagation medium in the waveguide is air. Another approach as in [8] uses a pillar to short a coplanar line on the bottom metallic plane of the waveguide.

For VA-CNT base waveguide, two topologies for excitation were considered:

- Slot-fed bottom-up SIW
- Coplanar-pillar excitation

Figure 3-6: a: slot fed bottom-up SIW. b: Coplanar-pillar excitation. Both schematics are slice view from the side of the waveguide. in black: VA-CNT forests, in gold: metallization. Green arrow represents coplanar I/O. Red represents the propagation of the wave.

Both topologies are bottom-up fabrication. Two substrates are needed, one for each metallized cap of the waveguide made of VA-CNT.

Slot-fed SIW

In a first step, slots were considered to excitate the waveguides since the usage of slots is quite common in bulk waveguides for antennas or for coupling of different waveguides. The aim here is to adapt the work already done on slots to SIW, and more precisely to CNT based SIW.

The most common type of slot is the rectangular slot, often used for antennas. The tweak here is to excitate this slot with a transmission line on the top substrate. The slot is on metallized back of the substrate, when the access transmission line is on the top as can be seen on Figure 3-6 left part. Some work exist in the literature about feeding of waveguide using slots. In this case, the fields in the substrate being closer to that of a patch antenna when trying to excitate through a microstrip line, references on the feeding of antennas were also including mostly more exotic slot shapes. The type of slots studied include the ones in Figure 3-8.

Simulation setup

In general, the simulation setup will be as such: CNT walls 500 μm thick, thickness varying in the range of what is obtainable in growth (Between 100 μm to 1 mm). The width is dictated by the working frequency, and more specifically the chosen cut-off of the waveguide. In most of the case this cut-off is chosen to be 50 GHz, inducing a width $a=3$ mm. The substrates for both caps are alumina substrates, of which the thickness varies in multiples of 5 mils (127 μm). Mostly 3 thicknesses were considered: 5, 10 and 20 mils (127, 254 and 508 μm respectively).

The microstrip line used to excitate the slot is terminated with a coplanar line as it is easier to probe than microstrip line: given the topology, it would be very ill-advised to use coaxial connector since the pressure required to affix it to the substrate would very likely damage the carbon nanotube forests. The transition from coplanar to microstrip is done using a floating ground and a tapering to match the microstrip line [68]. Losses from this transition were computed below 0.1dB.

Figure 3-7: Coplanar to micro-strip transition. λ_g being the guided wavelength of the wave in the micro-strip line. The coplanar and micro-strip line dimensions are that of 50 Ω lines at the given frequency.

Many different designs were tested out and tried. Most of them are from the literature on the coupling of waveguides via slots, or the excitation of antennas using micro-strip-slot ([69]–[74]) and the derivations and fusions of some of the ideas. Most of the designs considered were not extensively looked into for the first results obtained

were unsatisfactory. For all the setups in Figure 3-8, extensive simulation steps were taken.

Figure 3-8: Example of types of slots considered. From top left to bottom right: Slit, Horizontal slot, Double slit fed with Y lines, H slot, Box slot, V slot, House slot, C slot and slanted slot.

The main difficulty in this setup for excitation is that as commonly studied in the literature, there is a propagation layer between the slot and the antenna which is not the case here where the antenna is directly under the microstrip, and directly in the metallization of the waveguide. Some simulations using a second substrate to use as a propagation layer were tried out, but the results of these are not conclusive. On the other hand, simulations have shown that the excitation tends to work better when going

through the VA-CNT layer used for slow-wave effect (see Chapter 3 for more), the layer probably acting as a propagation layer ersatz in these specific cases.

Figure 3-9: S-parameters of a House-O slot-fed waveguide (Coplanar line to waveport). a: With slow-wave layer. b: Without slow wave layer. $a=3$ mm, $b=500$ μm , $WallsT = 1$ mm, substrate thickness: 127 μm .

Of all the devices, the house-O slot was the most promising, Figure 3-9 show the results of a round of optimisation on the House-O type of slot. The transmission hovers above -3 dB while the reflection around -5 dB for the slow-wave case. In the case without slow wave, the transmission is approximately the same, but the reflection is lower. Yet the bandwidth is shorter, with only 50% of the cutoff frequency versus more than 100% of the cutoff. In both cases, the reflection is around -0.9 dB whilst the lines are simulated with lossy material.

In the case with the use of two coplanar inputs is as shown Figure 3-10. The bandwidth of the device is low, only 10 GHz, but the design was made with prototype fabrication in mind, and thus the focus was put close to the cut-off frequency.

These waveguides rely on a hard to tune excitation (micro-strip to slot to waveguide). Hence it is quite difficult to start fabrication with such devices, for this purpose another topology was studied, with less uncertainties on the excitation method.

Figure 3-10: S-parameters for a waveguide slot-fed with a House-O type slot on both ends. Focus was made on the beginning of the band, for future fabrication.

Coplanar-Pillar excitation

The second type of excitation is similar to [8]. The approach is, as opposed to the previous one, to have the coplanar accesses on the same substrate as the waveguide. Instead of having a slot coupling the waveguide, here the coupling is done via a stub short-circuited to the top plate of the waveguide (schematic side view on Figure 3-6).

Methodology

The excitation being made of VA-CNT, it adds a layer of uncertainty on the design. To this end some simulations were done to assert the risk caused by this uncertainty: mainly the impact of the density of CNTs on this excitation, the height mismatch between the walls and the pillar, the bad electrical contact. Simulations were conducted in the same pattern as previously: One excitation on one side, and a waveport on the other, then both excitations with their coplanar inputs. Another point of interest is the opening required for the coplanar line to reach the pillar. The opening in the wall cannot be too great for it would disrupt the continuity of the wall, yet common

RF sense would dictate that an opening lower than quarterwave should be acceptable. The hole cannot be too short either, for it can disturb the propagation in the coplanar line. Coplanar dimensions are fixed by the measurement method: RF probes usually have a fixed size: pitch and probe size (Figure 3-11).

The steps of the designing and fabrication of the devices is as follow: Waveguide design, excitations design, waveguide including the excitation + coplanar lines according to where the measurement shall take place, modification of design according to the fabrication constraints, fabrication, measurement, retro-simulations and repeat from step 1 from there. Three iterations were done for this design of simple straight waveguide.

Figure 3-11: Exemple of HF probe used for characterization of samples: Freq. range 0-67GHz, G-S-G 50 μm probe size 125 μm pitch.

As the design of the waveguide with waveport has already been done previously, it will not be done again in this part.

The excitation is designed to excite the TM modes of the waveguide as seen on Figure 3-12. By design though, this excitation can only excite modes with H-field anti-nodes in the middle of the waveguide. Furthermore, the fabrication method typically makes heights of the waveguides much lower than their width, thus the second superior mode of the waveguide is the TM_{30} , which implies that the passing bandwidth of the waveguide is higher than usual waveguides.

The main parameters for the excitation are the pillar width, the pillar position (its penetration in the waveguide), as stated before this excitation can only excite anti-node of H-field where the pillar is. Should the pillar be offset in the width direction of the waveguide, some interesting effects could arise (but were not studied here). As stated in the methodology part just before, some other parameters were also considered afterwards: conductivity of the pillar, height mismatch between the walls and the pillar, electrical contact between VA-CNTs and gold (top and bottom).

Figure 3-12: Simulation setup for the excitation in the waveguide. In black: VA-CNT forest. Golden color is gold, blue is substrate (Al_2O_3 , Si or Rogers). b is zoomed on the excitation pillar.

For demonstration purposes, the focus of the simulation was not on the bandwidth, but on the cutoff of the waveguide. A first simulation of a waveguide with coaxial excitation was done (Figure 3-13), while ignoring losses through the walls of the waveguide, and in the substrate (Al_2O_3). The S-parameters show steep cutoff and flat transmission in the bandwidth from 50 to 130 GHz. Theoretically, the bandwidth

should reach 150GHz (appearance of the second superior mode), but a cavity effect occurs before due to the length of the waveguide.

Figure 3-13: a: S-Parameters of the waveguide with Pillar excitations. Coplanar line feeding with Coaxial input. b: Dimensions in micrometre (top view of the excitation): red VA-CNT, Yellow gold, Green substrate. Waveguide width ($a=2900\text{ }\mu\text{m}$), waveguide height ($b=100\text{ }\mu\text{m}$). Metallization thicknesses: $3\text{ }\mu\text{m}$. Dimensions in μm .

Figure 3-14: Mask design of the first batch of samples. a: overview of the 2x2 inches mask. Blue the gold patterning. Green is the catalyst patterning, for CNT growth. 6 Devices from top left to bottom right: 50 GHz 1.5 cm long, 50 GHz 2 cm long, 100 GHz 1 cm long, 100 GHz (slow-wave) 1 cm long, 50 GHz (slow-wave) 2 cm and 50 GHz (slow-wave) 1.5 cm long. b: Zoom on 100 GHz device.

As stated before, the design was modified to fit better the fabrication constraints and devices were duplicated at other frequencies. The coplanar lines inputs were also normalized at these frequencies to be ground-signal-ground G-S-G (16-32-16). The batch was designed to be fabricated on 2"x2" (approx. 5x5 cm) Alumina substrates.

Compared to the previous coaxial input design, this design was altered to have thicker walls (500 μm instead of 100 μm), thicker excitation pillar width (200 μm vs 100 μm), uniformized access lines (as stated just before), VA-CNT support pads on the sides, to support the weight of the top metalized cap without damaging the pillar or the walls. Additionally, there are also 3 samples to be used for Slow-wave propagation, which will be explained later in chapter 4.

Fabrication was done on 2x2" alumina substrate (thickness 254 μm , $\epsilon_r = 9.8$, $\tan \delta = 0.006$): Lithography (Blue mask of Figure 3-14) then gold deposition followed by lift-off to get the bottom substrate with coplanar accesses. Then a second lithography is done, with the green mask of Figure 3-14, catalyst deposition then lift-off. The sample is subsequently partitioned according to the different devices, and individually the VA-CNT forests are grown in the Black-Magic CVD machine.

Fabrication steps raised concerns at several steps: 1) the surface quality of the aluminas was not good, causing lithography scattering with commonly used AZ5214 photoresist in the cleanrooms. Additionally, the thickness of the photoresist makes thicknesses of gold of more than 1 μm difficult to lift-off. This was bypassed by using a thicker negative photoresist Ma-N 1440. 2) Then again, the surface quality of the alumina being poor, it can be seen on Figure 3-15 that the gold deposited on it has too a poor surface quality. This induces additional losses in the lines, and causes the CVD growth on it to be difficult to become repeatable. 3) The growth of CNT happening at 675°C, the gold surface blackens a bit, and so does the naked alumina. Some carbon is deposited during the CVD process (Figure 3-16). This carbon layer can be conductive and cause short circuits in the lines.

Figure 3-15: Microscope photos of fabricated waveguides on Alumina. a: Overall view of 100 GHz device. b: Zoom on access line.

6 samples were made in this batch, and were later measured in XLIM (Limoges, France).

Figure 3-16: Naked alumina substrate after growth process in TCVD machine

Figure 3-17: SEM pictures of 100 GHz sample. a: Walls separation for coplanar access, b: top view of excitation, c: side view of excitation, d: zoom on top of walls. The surface quality of the VA-CNT forests is very rough due to the surface aspect of the gold on the aluminas.

On Figure 3-17 the surface quality of the gold can be seen to be poor due to the surface quality of the alumina substrate. In addition to adding losses in the lines, the VA-CNT growth on top is also uneven, raising concerns about the electrical contact between the top of the walls and the cap.

Figure 3-18: Simulated S-parameters of waveguide designed for having a cut-off frequency of 50 GHz, with slow-wave reducing the cut-off frequency to 38 GHz. The simulation takes into account lossy lines and was optimized for the lower part of the bandwidth. This is the simulation of the fabricated devices at 50 GHz.

Measurements on this samples were unsuccessful as the transmission of the coplanar access lines was measured to be lower than -20 dB for each line. These losses come from the poor quality of the gold deposition of the lines, the relatively low thickness (slightly less than 1 μm) and the carbon deposition due to the growth process.

Second batch

Figure 3-19: SEM images of direct growth of CNT on devices fabricated on HR Si wafers. a: Excitation pillar, b, tilted side overall view

To circumvent this problem of the lossy lines, the second batch of sample was fabricated on High resistivity silicon wafers (Thickness = 350 μm , Resistivity > 3 $\text{k}\Omega\cdot\text{cm}$, double side polished, 4" diameter). Since the previous batch failure was determined to

be the gold quality and underlying substrate problems, the design from the first batch was not altered.

From the previous samples the growth was better, as we can see on Figure 3-19: the surface of the VA-CNT forest is rather smooth, and the patterns are well defined. Yet we can also observe some parasitic growth of CNT on the edges of the gold, and of amorphous carbon on the surface of the gold. Raman spectra of these growths concluded that this is probably amorphous carbon, and since this growth only happen on the gold, this should not cause any issue for the device operation (Figure 3-20 Figure 3-21) as amorphous carbon is non-conductive.

Figure 3-20: a: Optical and b: SEM view of the carbon parasitic growth. c: Raman spectrum on the green circle. The hilly aspect is due to the gold, We can see the two peaks of carbon, with the D one higher, possible meaning amorphous carbon.

Yet even while ignoring these parasitic growths of carbon on the gold, we can still see a roughness on the gold surface on a micrometre scale which can be detrimental for the transmission parameter of the coplanar lines, and thus the whole device.

Figure 3-21: Left: Spot 1, Right: Spot 2. From top to bottom: SEM Perspective picture, Optical top view, Raman spectrum. From both Raman spectra we can conclude that these parasitic formations seem to be amorphous carbon covered in gold.

Some measurements were done to test the transmission parameters of the coplanar accesses this time, to avoid the issue of having extremely lossy lines making the full devices S-parameters irrelevant.

Figure 3-22: S-parameters of access lines of device 1, port 1. The transmission this time is still poor, but acceptable.

To follow the logic of the experimental protocol: simulations of the devices done as designed supposing infinitely large substrate, simulations of sample as was measured, simulations of another set to try to understand where losses and effects come from.

The theoretical response we should get is as seen on Figure 3-13. Figure 3-23 shows the measurement of one samples of the second batch of fabrication. Measurements were done on 8 samples, but the measured parameters were always similar to this one. 5 100 GHz cut-off waveguides, and 3 50 GHz cut-off waveguides.

Figure 3-23: Measured S-parameters for waveguide as seen on SEM picture. Design for having a 100 GHz cut-off. The two black curves are the reflections (S11, S22), while the red/blue curves are the transmissions (S21, S12).

On Figure 3-23 the adaptation of the ports is good, with -10dB on the reflection, yet there should be a cut-off visible at 100 GHz that is not visible here. The shape of the S-parameters should be similarly shaped as in Figure 3-13 as both devices are waveguides but designed for different cut-off frequency. Of all the devices of this batch, the ones designed for a 100 GHz cut-off were the most promising ones in terms of fabrication: the excitation pillar having the same height as the walls.

Figure 3-24: Simulation of S-Parameters (Reflection S11 and transmission S12) of device as modelled after measured sample. Top: with perfectly conductive lines, Bottom: Including line losses.

The first round of retro-simulations on the measured samples are made with the assumption that the fabrication is perfect as in the CNTs are perfectly vertically aligned, have a density of conductive ones of 10^{15} m^{-2} and there are no contact issues between the CNTs and both the ground planes.

Figure 3-24 show the parameters to be extremely poor for a perfect case scenario, indicating the design itself is a problem in the functioning of the devices.

Third batch

Following the problem on the design of the waveguides that occurred on the second batch, the waveguides were re-designed. In addition to that, an alternative to the fabrication method was found in using transfer techniques mainly developed for flip-chip interconnects as in [75]. This method was modified to use higher density grown carbon nanotubes instead of the PECVD very short and low-density ones. This allows the fabrication of the waveguide while bypassing the CVD process which exposes the gold to very high temperatures ($>650\text{ }^{\circ}\text{C}$). The process does involve solder but works with temperatures under $200\text{ }^{\circ}\text{C}$. The principle behind the technique is to grow VA-CNT forests without any patterning on a sacrificial substrate. Then the pattern of the waveguide is deposited as gold on the surface of the VA-CNT forest by e-beam, using a shadow mask. The same pattern of solder (Indium or SAC [Tin, silver, Copper]) is deposited on the device substrate. Using a flip-chip setup, the CNTs with the patterned gold on it are aligned then flipped on top of the device with the patterned solder, the device is heated up to the melting point temperature of the solder, and pressure is applied while the temperature ramps down. Afterwards the CNT substrate is peeled off, and the patterned forest stays on the device substrate. This technique presents the advantages of being at lower temperatures, and to have a good metallic contact between the base of the CNTs and the ground plane. On the other hand, this method relies on shadow masks, which lowers the minimum resolution we can get for the patterned VA-CNT forests, and the transfer technique struggles when the surfaces of CNT to transfer are too large. Additionally, the pressure applied to the CNTs tend to bend them, which reduces their height.

Figure 3-25: Characterisation of transferred CNTs. a: SEM top view of transferred patterned VA-CNT forests. b: SEM side view of transferred CNTs. c: Raman spectra of CNTs before and after transfer.

The transfer process in itself doesn't damage the CNTs as seen on **Error! eference source not found..** The macroscopic patterns suffer slightly from the transfer in non-detrimental way for RF devices (the feature size of the defect being much smaller than the wavelength). On the microscopic scale the CNTs retain their vertical alignment and their size.

In terms of design, the use of this technique requires thus that the minimum pattern size to be at least 100 μm, and was chosen to be 200 μm for safe measures. The supporting pads were also changed to be made of a compound of smaller shapes Figure 3-26, yet this should have very little impact on the parameters of the waveguides.

Figure 3-26: a: VA-CNT forest pattern for waveguide. b, simulation setup for waveguide, without alignment marks and support pads. Support pads are designed as clusters of triangles for transfer technique to work. Triangles were chosen to also showcase the ability to get very sharp designs with direct growth or transfer techniques.

Once again, the designs were made for devices working at 25, 50 and 100 GHz, and this time again the access lines were homogenised to G-S-G (26-50-26 μm), to fit the 125 μm pitch probes used in the measurement setups between 0 and 110 GHz (Figure 3-11). Simulations before and after taking the losses of the line into account are in Figure 3-27.

Figure 3-27: a: S-parameters for a waveguide designed for cut-off at 50 GHz, ideal case. b: S-parameters for a waveguide designed for a cut-off at 50 GHz with slow-wave reducing the cut-off frequency to 38 GHz, lossy case.

The measurements were made on the same setups as for the two previous batches but this time yielded results showing the expected response of waveguides (Figure 3-30). The measurements were done on RF absorbent to minimize the direct

transmission in the substrate. The waveguides were closed using caps of three types: polished stainless steel, roger dielectric plate with 80 μm copper metallization, and dummy silicon substrate with 1 μm gold metallization. The samples included direct growth VA-CNT samples on High resistivity silicon, transferred VA-CNT on Alumina, transferred VA-CNT on high resistivity silicon. Additionally, some samples underwent post-treatment to cover the top surface of CNT with either SAC or gold, and some of them had the cap already stuck on using solder method (yet the pressure used to stick the caps seem to have been too great as, it was not possible to see the waveguides as in Figure 3-28, implying that the waveguides were crushed).

Figure 3-28: Measurement of samples. a&b&c&d: e.g. of samples measured, a: 100 GHz silicon transferred CNT, b: 100 GHz silicon transferred CNT with SAC solder on top, c: 50 GHz Alumina transferred CNT, d: 25 GHz direct growth CNT. Bottom samples with cap set on for measurement. e: 50 GHz Alumina sample with rogers 80 μm thick copper metallization as cap on RF absorbant. f: Side view of 50 GHz silicon sample with 1 μm metallized si dummy wafer as cap on RF absorbant.

Of course, the question of the contact of the cap on the CNT using simply caps set on top of the waveguides seem rather unsightly. The quality of this contact was confirmed qualitatively on Figure 3-28 with the cap holding on the waveguide quite

strongly and on Figure 3-29, with the side picture showing the inside on the waveguide below the cap. Additionally, three devices at 50 GHz were tested: one with the cap just set on, just as Figure 3-28 & Figure 3-29, one with a stuck on cap by transfer method, and lastly one with the cap set on, and pressure added manually on the cap. The results are given on Figure 3-29: the devices with stuck-on caps where exposed to too much pressure during the process and have their transmission parameters degraded. Between the devices with the caps pressured or not, the difference is ever so slightly in favour of the pressured cap waveguides. Yet, this pressure is quite intense, and typically squished the VA-CNT forests of the waveguides.

Figure 3-29: a: Device with polished stainless-steel cap set on without glue, held by the cap. The mechanical contact is good. b: Transmission (S_{21}) parameter of three devices designed for cut-off at 50GHz. Green: Stuck cap, Blue: set cap (as in the left part of the figure), Red: cap with pressure applied to it while measuring (applied manually).

Additionally, the measurements were done on two setups: 0-67 GHz and 75-110 GHz. The results shown on 0-110 GHz band were done in three steps: 0-67, 67-75 and 75-110, with the 67-75 measure done with the 75-110 setup, which introduces high level of noise on the lower end of the band due to sources instabilities (as can be seen in Figure 3-30). This noise is known to be the sources, and should not be considered as the waveguides own response.

Figure 3-30: Representative S-parameters for third batch of samples. a: (S21 and S22) Device designed for cut-off at 50 GHz, b: (S12 mag and phase) device designed for cut-off at 25 GHz, c: (S11,12,21,22) Device designed for cut-off at 100 GHz. Dimensions of waveguides: 25 GHz: 30 mm long, 6 mm wide. 50 GHz: 15 mm long, 3 mm large. 100 GHz: 10 mm long, 1.5 mm large.

Many samples were fabricated and measured (~25), but the results were highly consistent hence only some examples will be discussed. The results for the device designed for a cut-off at 25 GHz shows that the cut-off is clearly visible at the expected frequency, yet the transmission level is extremely low at around -40 dB, but still stays some 40 dB above the non-passing transmission level (Figure 3-30). The phase of S12 there shows a smooth evolution which is a sign of transmission in this bandwidth. On the other hand, the phase is also smoothly varying in the non-passing band, from around 3GHz to the cut-off. This shows that there is signal transmitting through the

ports, but not in the waveguide. The -80 dB S12 level before the cut-off is thus not noise, but parasitic transmission through another medium than the waveguide. This shape is very similar on devices at higher frequencies, but at higher levels (-60 dB for 50 GHz devices, -40 dB for 100 GHz devices).

The change in transmitted power most probably comes from the difference in length of the waveguides, with the 25 GHz being longer than the 50 GHz itself being longer than the 100 GHz one. This effect is later studied through simulations and is attributed to a lack of perfect contact between the top of the CNTs and the top cap of the waveguides (Figure 3-33).

The 50 GHz waveguide was measured on a larger band, and shows a very steady albeit low transmission level from 50 to 110 GHz which corroborates the discussion above on the mode excitation of the waveguide, allowing for a larger bandwidth (usually, the second superior mode is at maximum twice the cut-off frequency of the waveguide, but with the current means of excitation, it is pushed to three times the cut-off frequency). The devices at 100 GHz all exhibited the same response with a dip at 100 GHz, but very low difference in transmission before and after the cut-off. As for the 25 GHz devices, the cap used was made of a cut and polished steel plate and required pressure on the cap for the signal to appear, meaning that the contact between the cap and the CNT was uneven on the length of the waveguide. These reasons are why most of the focus was directed towards the devices designed for a cut-off frequency at 50 GHz, as the results were most promising on these.

Retro-simulations

The different parameters that could lead to lower transmission are expected to be either or all of the following: Poor line quality/fabrication, substrate resistivity/dielectric constant off-specs, poor electrical contact between the CNTs and the base substrate, poor contact or gap between CNTs and cap substrate, lower CNT

density/conductivity than expected, waveguide dimensions off-specs, waveguide height off-specs.

- Poor line quality: lower transmission/reflection parameters overall if the line is very lossy, or abnormally high reflection and poor reflection.
- Substrate resistivity or dielectric constant off-specs: overall lower transmission/reflection in the lines and if the substrate is really conductive, reflection very high, transmission non-existing.
- Poor electrical contact between CNTs and base: Lower transmission, higher reflection due to mismatch between excitation pillar and line.
- Poor contact between CNTs and cap: lower transmission due to leaking, possibly some parasitic resonance in the reflection, due to pillar resonating.
- Lower CNT density/conductivity: Lower reflection, smoother cut-off, possible red-shift of cutoff.
- Waveguide off-specs: pillar impedance varying would imply higher reflection and lower transmission, or non-flat transmission in the passing band.

Two devices were specifically chosen for these retro-simulations, dubbed 50 Al S2 and 50 Al T3 Figure 3-32 (S and T referring to the thickness of the walls in the design (200 and 500 μm)) for those two devices were tested with the cap set without pressure, which allowed for better reproducibility of the measures. SEM imaging from the side also revealed that the height of the walls was not constant but instead made the top surface of the waveguide slightly slanted (One pillar is 120 μm tall while the other is 192 μm tall in device T3).

Figure 3-31: Fitting the simulations based on SEM imaging of the samples.
Top: T3. Most of the smaller unwanted carbon growth was ignored. **Bottom: S2.** The shape and size of the excitation pillar was adjusted, as was the height of the walls.

The design being somewhat similar to the devices, the only unknowns on the design remaining are: the density of the carbon nanotubes (more precisely, their equivalent conductivity) and the contact imperfections. Given the conditions of the samples due to growth and transfer, DC measurements of conductivity were done on both the conductive layers, and non-conductive showing no problems on both parts (gold conduction is good, and no conductive layer deposited on top of the dielectric substrates). At the same time, DC conductivity measurements were done from the top of the VA-CNT forests and the conductive layer below, showing resistances varying from 30 to 60 Ω , mostly dependant on the pressure applied with the probes, which stays consistent with the expected resistance of the of VA-CNT forests of such densities.

Figure 3-32: Measured vs simulated S-parameters for device T3 (top) and device S2 (bottom). Line: measures. Dotted lines: simulation. Simulations done with 1-2 μm gap between top of VA-CNT walls and top cap, VA-CNT density of 10^{15} m^{-2} .

Figure 3-33: a: E-field at 50 GHz for device T3 with a gap of 2 μm between the top of the VA-CNTs and the top cap of the waveguide. The propagation in the waveguide is evanescent, but propagation appears between the cap and the top of the waveguide walls. b: Simulated transmission for waveguide with and without gap between top of VA-CNT forest and cap.

Of the effects of the gap between the walls and the cap of the waveguides, the relatively high non-passing transmission (Figure 3-33): the fields plot show that the transmission seems to occur in the gap between the VA-CNT and the cap but decreases rapidly over distance, which seems to corroborate the first assumption and the results showing lower out of band signal power in longer devices. Furthermore, the

simulations with and without this gap also corroborate this assumption with the cut-off being interrupted by a plateau on the simulation of device with gap.

The problem arising from this effect is more about the transmission in the passing band being lowered by a few dBs rather than the cut band being relatively high at -55 dB. On Figure 3-33 we can see the difference between the two simulations amount for 3 to 10 dB, while the transmission of the better devices usually reached -15 to -13 dB in their passing band.

To circumvent this fabrication issues in a fourth batch of sample, the following steps were taken: increasing the metallization layers thickness (to reduce possible contact issues with the probes), improve the process control (to have better conforming lines), add a process step to stick the caps to the CNTs using metallic solder with low pressure flip-chip (to minimize if not neutralize the unwanted propagation on top of the VA-CNT walls without squashing the CNTs).

Other excitations

Other excitation types were briefly considered, such as the bracket slot on a trapezoidal waveguide (etched in silicon wafer), or the TM₁₁ excitation of VA-CNT waveguide, very similar to the one previously studied. The former doesn't add much for the waveguiding part of the thesis, yet can be of interest for slow-wave waveguides, and will thus be described later. As for the latter, the excitation method uses a shorted coplanar stub in the waveguide (Figure 3-34), instead of a VA-CNT pillar to short the line to the cap. A cut-off frequency of 50 GHz of the TM₁₁ mode requires dimensions of approximately $a=b=4$ mm which is not currently easily possible to fabricate. Yet it is possible to use this same excitation for resonant TE₁₀₁ cavities which doesn't have constraints on b (the height of the waveguide), rendering the process possible to fabricate with our current means.

Figure 3-34: Coplanar access and excitation for TM₁₁ and TE₁₀₁ cavities. Dimensions vary depending on frequency.

Cavities

The idea behind the cavities is their simplicity, with a single resonant frequency to study (Figure 3-35), but also the high sensitivity to the cavity dimensions and quality affects the quality factor, which can be used reversely to characterise precisely the conductivity of the CNT walls. The designing of cavities follows Equation 3-3, hence for a TE₁₀₁ any constraint on b disappears. In the facts, the higher b, the higher the quality factor of the cavity.

$$f_r = \frac{u'}{2} \sqrt{\left(\frac{m}{a}\right)^2 + \left(\frac{n}{b}\right)^2 + \left(\frac{p}{c}\right)^2}$$

Equation 3-3

Design

Figure 3-35: Structure and dimensions of a 150 GHz square cavity. Substrate is 525 μm thick high resistivity silicon wafer, bottom face metalized. Metalizations for bottom and top layers are 1 μm thick. Line is 300 μm long.

Using Equation 3-3 several cavities were designed for testing over a range of frequency, from 30 GHz to 280 GHz (**Table 3-2**):

Table 3-2: Cavity dimensions, and coplanar accesses dimensions fixed by the probes pitch. Several designs have a square and a non-square variations.

Frequency[GHz]	a [μm]	b [μm]	c [μm]	Pitch [μm]	G-S [μm]
30	7500	300	6708	125	33-50
30	6500		7825		
40	5000		5669		
50	3750		5000		
60	3000		4523		
70	3000		3062		
70	2500		4160		
80	2500		2835		
85	2500		2500	100	
90	2000		3015		
100	2000		2268		
120	1500	200	2261		
150	1500	150	1342	75	18-28
150	1200		1809		
175	1200		1226		
175	1100		1368		
200	1000		1134		
240	900		869		
240	800		1001		
280	750		765		
280	675		881

Figure 3-36: S-parameters for cavity designed to work at 150 GHz (dimensions in Table 3-2). There is a slight frequency shift due to the excitation and the opening in the cavity for the coplanar accesses. In full line: opening in the cavity for access reaching the top of the cavity. In dotted line: opening in the cavity reaching halfway through the top of the waveguide. Horizontal line is -3 dB for reference.

Similarly, as for the waveguide design, the process starts with perfect simulations (metallic grounds, cavity walls are perfect conductors), then added losses one by one to check the critical points of the design. Then some more simulations were run to check the impact of the equivalent conductivity of the CNT.

Figure 3-36 shows the simulated S-parameters of a sample cavity design. With $a=1500\text{ }\mu\text{m}$, $b=150\text{ }\mu\text{m}$ and $c=1342\text{ }\mu\text{m}$, Equation 3-3 yields a resonant frequency of 150 GHz. The resonance is red-shifted due to the opening and excitation perturbing the cavity. The resonance can be fine-tuned back to 150 GHz by slightly shortening the cavity, yet it was left as is as the actual resonance frequency matters little.

Figure 3-37: S-parameters for a cavity designed to work at 150 GHz (Dimensions in Table 3-2 & Figure 3-35). Compared to the perfect case in Figure 3-36, The transmission and reflection are lower due to losses and the resonance red-shifted slightly, while the Q factor is visibly lower.

The quality factor from the perfect simulation is 347.8, while the one for the real case scenario (Figure 3-37) drops to 85.48, which is largely explained by the finite conductivity in both caps of the cavity and of the carbon nanotubes walls. To further look into this, a simulation with varying densities of carbon nanotubes was done with results in Figure 3-38.

Density	Fr	S21r	Q
10^{16} m^{-2}	149.1	-3.5216	121
10^{15} m^{-2}	148.95	-3.3077	92.8
10^{14} m^{-2}	148.25	-4.9137	72.0
10^{13} m^{-2}	145.85	-11.221	64.0
10^{12} m^{-2}	138.9	-16.52	22.5

Figure 3-38: Transmission of 150 GHz cavity versus density of VA-CNT forest. Left: plotted transmission parameters, Right: remarkable figures.

The cavities are rather sensitive to the equivalent conductivity of the VA-CNT forests, and thus implicitly to their densities. As opposed to usual methods [76][65] to determine the density of the forests, this could be of use to determine the density of conductive CNTs in the VA-CNT forest.

Conclusion

Waveguiding in VA-CNT based waveguides has been demonstrated both in simulations and in measurements of prototypes. If the fabricated devices are still lossy, their performances match that of the simulations when taken into account all the imperfections. This proof of concept in itself is very important as it showcases the possibility to use VA-CNT forests for waveguiding.

This opens the way for more work on VA-CNT based waveguides, and specifically on the slot-coupled waveguides: since the concept of guiding is sound, it is worth optimizing the excitations.

Single waveguide cavities are a very simple resonant device, but as such can be a very powerful characterization tool. The excitation being simple and not relying on CNTs, it is possible to predict very accurately their resonance frequency, peak and quality factor, making any changes on these parameters easily detectable. Extensive simulation work has been done to show that the variation of both quality factor and

resonant frequency depends on the density of conductive CNTs. This can be a way of characterization of VA-CNT forests.

CHAPTER 4: SLOW-WAVE

Introduction to slow-wave effect

Wave velocity in RF refers generally to the phase velocity of the wave $v_p = \lambda f$ [m.s⁻¹], λ [m] the wavelength in the propagation medium and f [Hz] the frequency of the wave. It is understood that λ is dependent on the propagation medium, and specifically the dielectric constant ϵ (or the refractive index $n = \sqrt{\epsilon_r}$ in optics, where ϵ_r is the relative dielectric constant of the medium). If the phase velocity carries no information (and thus can sometimes be greater than the freespace velocity c) it is important in RFs for resonant systems, as the length of such systems usually are set in multiple of the quarter-wavelength which reduces proportionally to the wave velocity.

$$v_p = \lambda * f \quad \text{Equation 4-1}$$

where v_p [m.s⁻¹] is the phase velocity of the wave, λ [m] its wavelength and f [Hz] its frequency.

At a given frequency, some devices have set dimensions, like antennas, stubs, filters, and this can only be changed by changing the effective dielectric constant of the propagation medium ϵ_{eff} (which, to simplify, is the dielectric constant factoring in the topology of the fields and of the transmission device) or working at higher frequencies. Since the frequency is usually set, the only solution is to change the wave velocity is to get slower waves. A widely used example of this kind of engineering is in phone antennas, using this effect to keep the antenna dimension below that of the handphone.

The easier way to produce slow-wave is with the use of high-k dielectrics ($k = \epsilon_r$). A lot of work was done on those, motivated by their use in CMOS technologies. If high-k refers to higher than silicon-dioxide ($\epsilon_r = 4$), high-k dielectrics specifically used for RF slow-wave applications reach above $\epsilon_r = 20$, if the reference in the CMOS field is Silicon Dioxide with a ϵ_r of 4, for RF the most commonly used substrate is Alumina

ceramic with a ϵ_r varying between 9 and 10. Yet changing the material can introduce higher losses, and the increase on the dielectric constant usually changes the impedance and Q-factor of the devices, adding starker constraints on the design. Another way of doing so, is to use meta-materials to increase either the inductance $\mathcal{L}$ or capacitance C of the transmission line, effectively increasing the dielectric constant. It is possible using specific topologies to change only the impedance or the capacitance of the medium, which allow the modification of the wave velocity without increasing the impedance. For instance, looking at Equation 4-2 & Equation 4-3, a meta-material increasing C while leaving $\mathcal{L}$ would result in a slow-wave propagation with a lower characteristic impedance.

$$v_p = \sqrt{\frac{1}{\mathcal{L}C}} \quad \text{Equation 4-2}$$

$$Z_0 = \sqrt{\frac{\mathcal{L}}{C}} \quad \text{Equation 4-3}$$

where v_p [m.s⁻¹] & Z_0 [Ω] are respectively the phase velocity of the wave and characteristic impedance of a lossless transmission device on a medium of effective capacitance and inductance resp. C and $\mathcal{L}$ (both functions of frequency).

State of the art, high-k dielectrics, meta-materials

High-k

The research for High-k dielectrics is that of searching for a dielectric with a very large bandgap. Most of the state of the art dielectric materials showing the highest ϵ_r are binary or ternary oxides. As stated before a very commonly used dielectric for RF is Alumina ceramics Al_2O_3 which exhibit dielectric constants of up to 9.4 with $\tan \delta$ values of 0.006 for typical commercial alumina substrates at low frequency (MHz), diminishing around 8.8 and 0.004 in the tens of GHz.

In the binary oxides, it is interesting to point out HfO_2 , as it is extensively studied to replace SiO_2 in Si CMOS transistors and is currently used in DRAM. The dielectric

constant of this material reaches around 30 [77], in addition to its very attractive stability in temperature, and thermodynamically inability to react with silicon.

Other high-k binary compounds include ZrO_2 with a dielectric constant measured at around 25 [78], Ta_2O_5 or TiO_2 with dielectric constant respectively 22 and 80 [79], or more exotic oxides with lanthanides or rare earths La_2O_3 , Y_2O_3 , Pr_2O_3 , Gd_2O_3 and Lu_2O_3 which have dielectric constants of resp. 30, 15, 18, 14 and 11 [79]–[82]. Ta_2O_5 also show ϵ_r around 100 in a defect-free crystalline form [83], but requires process reaching 800 °C to produce.

There are plenty of binary oxides showing good high-k properties, but there are even more ternary oxides and larger oxides. But worth noticing among them are STO (SrTiO_3), BTO (BaTiO_3): 2000 and 2000-6000 dielectric constants [79], [84]. CCTO is a complex ferromagnetic oxide ($\text{CaCu}_3\text{Ti}_4\text{O}_{12}$) reaching up to $\epsilon_r = 10000$ but dropping dramatically at higher frequencies (< 10 at 30MHz) [85]. The main issue with these super-high-k dielectrics is their wide variations of ϵ_r with frequency, making them unsuitable for mm-wave RF applications. The tantalum counterparts of STO and BTO, SrTaO and BiTaO display ϵ_r of resp. 20 and 50 [86], but there again the frequency within which the measurements are conducted stays below 1 kHz.

A filter was made using a ceramic presenting an ϵ_r of 90 in [104]. The group uses a commercial ceramic from Trans-Tech presenting an ϵ_r of 90, in addition with a $\tan \delta$ of 0.0009 to fabricate a SIW filter working in the C-band (4-8GHz). This is interesting since this ceramic has also a low thermal expansion constant, which is very important for RF devices.

meta-materials

Meta-materials with very high ϵ_r are researched for high refractive index medium, for tera-hertz and optics. It is possible to increase dramatically the effective dielectric constant with strong capacitive coupling while decreasing the diamagnetic response by

using, for instance, slit-ring resonators. It is possible to engineer quite precisely and quite unnaturally the refractive index of the material through tuning of ϵ and μ . In [87] they managed to produce quasi 3D medium with refractive index of 33.2 peak, while still staying above 8 at lower frequencies, resulting in ϵ_r around 120 in the mm band.

It is possible to design metamaterials quite precisely, playing with ϵ_r with planar resonators and μ_r by using diamagnetic materials, thus engineering complex refractive indexes. Some interesting cases with negative indexes or even Zero index were demonstrated [88], [89].

The recurrent issue with the very high-k dielectrics is that they rely on ferromagnetic effects to reach these values, but thus usually lose this property when the frequency increases. Some other simpler oxides can still reach very high values, such as TiO_2 and Ta_2O_5 in their crystalline forms, but the dimensions typically required for RF applications are larger than what the fabrication methods can output: several hundred of microns thick substrates of few centimetres square. Stacked planar resonators have the advantage to work at higher frequency, as they are made for optical purposes. Plus, at lower frequency the refractive index, ϵ_r & μ_r have all their imaginary part null, since the feature is way below the wavelength. The main issue with these, and meta-materials in general is the cost of producing them: stacking several layers of metallization on dielectric. In addition, adding any kind of SIW inside this kind of substrate is tricky, as the metallization of the waveguide will interfere with the resonators.

CNT for slow-wave

Here we try a different approach to slow-wave. Instead of increasing the dielectric constant of the material in which the wave propagates, the goal is to increase the effective dielectric constant of the wave in the propagating medium. In the transmission device, the wave is not in free-space, as such interactions with elements

of the propagation device will affect the wave travelling velocity, which is thus related to $\epsilon_{r,\text{eff}}$ instead of ϵ_r of the medium. The wave propagating in a transmission line have more constraints than free-space propagation: for instance, the wave propagates only in the substrate plane, and thus the medium needs not be isotropic.

Figure 4-1: Slow-wave in rectangular waveguide. a: isometric view of a waveguide. b: schematic front view of slow-wave waveguide. Vertically elongated metallic structures occupy a portion of the waveguide, defining a portion of the waveguide where the vertically aligned E-Field cannot exist but in plane H-field can. c: Front view simulation of such waveguide, with field intensities plotted. The greyed part is the region occupied by the vertically aligned metallic structures (simulated with bulk isotropic metal).

In practical, it has been achieved by fabricating a meta-material to increase greatly the capacitance of the transmission line, while keeping the inductance untouched. According to Equation 4-2 this would reduce wave velocity and characteristic impedance. For transmissions lines design, this means reduction of lateral and longitudinal dimensions. Figure 4-1 describe how this is done in a rectangular waveguide. We consider the TE₁₀ propagation in a rectangular waveguide: the first propagating mode E-field is vertically aligned while the H-field is circling orthogonally to the E-field. Part of the waveguide is filled with vertically elongated

metallic structures, with a sufficiently high aspect ratio so that the vertically aligned E-field sees this region as a metal and thus cannot exist within, while the orthogonal H-field can exist inside it with minimum interference. In terms of capacitance and inductance of the waveguide, in theory H is untouched, hence leaving $\mathcal{L}$ unchanged, while the effective height of the waveguide is reduced for E, increasing $\mathcal{C}$ which reduces v_p according to Equation 4-2.

The simulation of the result presented in Figure 4-1 uses for the meta-material region a bulk equivalent material. In this case the material is an imaginary anisotropic material, but simulations like this have been done with metallized via in [7], showcasing similar behaviour: The electric field is confined in the part without the metallic pillars, while the magnetic field propagates in the pillared region of the waveguide, while some interactions between the metallic parts and the field are visible.

Figure 4-2: Comparison of waveguide parameters with and without slow-wave. a: Phase velocity. b: Transmission. The red-shift of the cut-off frequency is visible on both (50 $\rightarrow$ ~35 GHz). The phase velocity also stays lower with the slow-wave. The transmission dips at 70 GHz with slow-wave, indicating the second higher mode TE_{20} of propagation in the waveguide, which is as expected twice the cutoff frequency ($a > 2 \cdot b$).

Simulations were done on a waveguide using VA-CNTs as walls, as described in Chapter 2, and VA-CNT filling a ratio α of the waveguide ($\alpha = 1$ is a fully filled waveguide, $\alpha = 0$ is a normal empty waveguide). The ports were defined as waveports and as explained in Chapter 2, with a small anisotropic metallic buffer for the simulation

to run. The dimensions of the test waveguide are the same as the ones of the first proof of concept waveguide in chapter 2: length=10 mm, width=3 mm, walls thickness = 200 μm , guide H = 300 μm and CNT density = 10^{15} m^{-2} . A simulation with $\alpha = 0$ was compared to one with $\alpha = 0.5$ on Figure 4-2: the red-shift of the cut-off frequency is clearly visible, from 50 GHz to ~38 GHz. In addition, the transmission remains as high, yet the cut-off sharpness is slightly softer. The shift of cut-off shows the wavelength to be shorter in the width direction of the waveguide, while the phase velocity converging to a lower value shows the wavelength to be shorter in the length direction of the waveguide: this slow-wave effect allows size reduction in both length and width of waveguide devices.

The filling ratio α of course interacts on the cut-off red-shift and the phase velocity reduction as seen Figure 4-3. The trend of the parameters seems to be linear for the phase velocity and quadratic for the cut-off frequency. Furthermore, not shown here, the transmission degrades from too high values of α : for values of α 0.6 and above, the beginning of the passing band is below 0.5 dB and while the value is extremely low for $\alpha = 0.9$, this might be due to simulation instability (as we can see a glitch in the phase velocity). Yet for $\alpha = 0.6$, the transmission stays at acceptable levels, and the size reduction of the devices is approximately 2 in both axes.

Figure 4-3: a: Phase velocity versus frequency, for α varying from 0.1 to 0.9. b: Variation of cut-off and phase velocity flat versus α .

Use of slow-wave

In addition to the obvious advantage of reducing the feature size of the devices, slow-wave can have other non-trivial uses: travelling wave-tubes and antenna arrays for example. For travelling wave tubes, slow-wave will allow for higher gain per unit length, as the wavelength is reduced for a given frequency: in traveling wave tubes, the idea is to use the anti-nodes of a standing wave to amplify a signal. The shorter the wavelength, the more anti-nodes per unit length it is possible to use, and thus the slower the wave, the more gain per unit length the traveling wave tube can output. For antenna arrays, and more specifically patch antennas, the importance of slow-wave comes from the fact that the array repetition matrix has to be halfwave of the medium of propagation, usually air. In the case of waveguide feeding of the antennas of the array, if the waveguide filling is air, the width of the waveguides will be at least freespace halfwave, which makes the feeding with waveguides impossible if not for the use of slow-wave to reduce the dimensions of the waveguide.

Slow-wave in waveguides and SIW

Impact of slow-wave on slot excitation

As introduced in chapter 2, the slow-wave layer in a waveguide tends to act as a propagation layer, thus facilitating the adaptation of the waveguide to the micro-strip line above. This is important for interposers (more in Chapter 4), since the feeding of the waveguide should be possible through both substrates (top and bottom), which causes problems due to the asymmetry of the device.

A solution for this matter is to separate the slow-wave layers on both substrates, to keep a symmetry in the device. Simulations on this kind of devices show good results and are easier to adapt than both the other solutions (through the full slow-wave layer, and on the opposite side). The results for this are shown in chapter 2. Yet this solution raises a question on the fabrication process to reach this kind of three-layer structure.

If multi-layered growth [4] or use of different buffer layer for different growth speed is possible, it adds complexity to an already non-trivial process. Another solution would be to use transfer as in chapter 2, with different sizes of carbon nanotubes. It was proven experimentally that this is possible.

Impact of slow-wave on coplanar-pillar excitation

In coplanar pillar excitation, the impact of the slow-wave layer is less important than for slot-excitation. If the impedance is indeed changed it doesn't completely change the functioning of the excitation as opposed to the slot excitation and, if not natural, it is possible to have the slow-wave layer either on the top or bottom of the waveguide Figure 4-4.

Figure 4-4: Setup (a) and transmission parameters (b) of coplanar-pillar excited waveguide with slow-wave layer on the bottom.

The trivial way of fabricating these slow-wave waveguides is of course to fabricate the waveguide as in chapter 2, then instead of adjusting a metallic cap on top, to adjust a metallic cap with a layer of VA-CNT forest on top. The height of the waveguide and that of the forest will thus define the α ratio. In the case of the inverted one, the concept is the same, but the top cap will have the shape of the waveguide, requiring an additional step of patterning, and of alignment when flip-chipping. That is why the trivial method was preferred for both fabrication and simulations.

The adaptation and design issues aside, this topology and fabrication behind raises several issues: the electrical contact between the two layers of VA-CNT, and more particularly the electrical contact between the excitation pillar and the top VA-CNT layer; the anisotropy of the VA-CNT layer is here of more importance than it is for simply guiding the wave and must be looked into; we know CNT growth of CNTs usually yield a layer of more horizontal CNTs atop the forest, which in this case would be in the middle of the waveguide.

Study of the various electrical contact between VA-CNT and cap

In most of the cases, bad electrical contact will be modeled by introducing a gap between the layers, as it will most of the time be realistic considering the fabrication method Figure 4-5 (affixing the cap to the VA-CNT waveguide, the VA-CNT on the bottom substrate due to buffer layer for direct growth, or due to solder or tape for transferred CNTs, or non-homogeneous growth as is the case for the second batch of fabricated samples in chapter 2).

Figure 4-5: The 5 considered possible electrical contact issues in the slow-wave waveguide. Each color represents one different gap for the simulations of the poor electrical contact.

In chapter 2, the real case scenario of the bad contact between the cap and the VA-CNT walls was already studied. Yet, the simulations here will be on the lossy

waveguide, without the fabrication pads, considering infinite substrate. In this case, each gap as represented Figure 4-5 is tested alone to check if the impact on the transmission values and shape are important. Then the effects deemed important will be simultaneously simulated to check if they interact with each other. This approach is chosen to reduce the number of rather computationally expensive simulations: on a local computation machine, each setup can take up to several hours of computation time, and the process must be repeated each time the design is altered.

To automatize the process, a quantifier was chosen to attribute the quality of the waveguide. This was chosen to be the average of the transmission parameter on a bandwidth to be defined from 38 GHz to 76 GHz (From the cut-off, to twice that frequency). Generally, this method was used for most of the fine tuning optimisation of the waveguides, varying parameters of the waveguide against one or multiple chosen operators.

Then a single value of 5 μm of the gap size for each of the different possible contact error as listed on Figure 4-5 is done and compared against the reference waveguide (Figure 4-6) to assess to which extent these imperfections impact the functioning of the waveguide (**Table 4-1**). Since the gap size was chosen to be big, the values are all large. From **Table 4-1** we can see that none are negligible, but the most predominant ones are blue/purple/orange. The gap value was chosen very large to exhibit large differences in the different setups. In practical, these gaps are expected to be much lower, around 1 μm for orange and red, and even lower than that for green/blue/purple.

Figure 4-6: S-parameters for reference waveguide. a: simulation setup dimensions, with also $a=3$ mm, $b=170$ μm , Length=30 mm, walls-thickness=200 μm , metal thickness=1 μm gold, substrate: 525 μm thick intrinsic silicon. b: S-parameters, with average value of S21 over the 38-76 GHz frequency range (grey zone). Dimensions in μm .

Table 4-1: Operator value for the different possible electrical contact issues as described Figure 4-5. Operator is Mean(S12(dB),38-76GHz).

Error type	Ref	Green	Red	Blue	Purple	Orange
Operator value	-2.7901	-10.677	-9.2632	-24.420	-16.122	-14.531

A simulation with more realistic gaps was done in Figure 4-7, with values of what is expected to be a rather bad case scenario of electrical contact errors. The parameters are completely damped to unsatisfactory levels. As it stands, backup measures must be taken to ensure the functioning of the slow-wave waveguide. For green, blue and purple contacts, direct growth CNTs should have only the 2-8 nm buffer layer of non-conductive Al_2O_3 , and thus should not pose any problem. Yet, considering the results from [39], it can still be problematic for longer growth CNT. To circumvent this, the use of transfer CNT switches the denser part of the CNT towards the grounds of the waveguides, while the electrical contact is ensured using electrically conductive solder. As for red and orange gaps, it is possible to put pressure on the cap, to minimize the gap forming in between the two VA-CNT layers, as those layers are somewhat elastic up to a few newtons of pressure [90]. Those two gaps can further be reduced using metallic solder, but only on CNT parts of the bottom part of the slow-wave SIW.

Figure 4-7: S-parameters of slow-wave waveguide with gaps as follow: Green 0.5 μm , red 1 μm , blue 0.5 μm , purple 0.5 μm and orange 1 μm , compared to the reference waveguide.

Study of density effects

The second unknown effect comes from the density. It was analysed in chapter 2 but should be done again with this topology. To be thorough, simulations should be done with two different densities, as the two VA-CNT forests are on different substrates. Yet, there is no reason why one forest would have lower density since the fabrication method is the same, both on gold, hence density is always consistent on both substrates for all the simulations.

Several values of density were compared against the reference waveguide with the use of the operator (Figure 4-8): the transmission stays almost unchanged for densities down to 10^{13} cm^{-2} , which leave a large headroom for fabrication in our case. At lower densities of 10^{12} cm^{-2} the transmission level drops to very low values, but the shape of the waveguide response is still clear, whereas below these values, the transmission is not exploitable anymore.

Figure 4-8: Effect of VA-CNT forest density on transmission. a: Operator=mean(S12[38-76 GHz]) versus density, b: S12 versus frequency for each value.

Study of anisotropy effects

In chapter 1 it has been developed that the growth of CNTs via TCVD and supergrowth method yield approximately vertical CNTs: their overall aspect ratio is extremely high, but they growth is vertical due to the high density of the forests, making

the z axis the only possibility of growth direction. In [39] it has been theorized and observed that CNTs are extremely tortuous and not entirely straight. On the other hand, the slow-wave phenomenon described here relies heavily on the anisotropy of the material.

A run of simulations was done on the reference waveguide, with different anisotropy values. The anisotropy of the material is defined as follows: 1 means the bulk material is completely anisotropic, the conductivity off z-axis is null. Other values of anisotropy mean the conductivity off z-axis is $\sigma_x = \sigma_y = \sigma_z * (1 - ratio)$. On Figure 4-9 we can see the micrometre scale of non-anisotropy, while the CNTs length is in the hundreds of micrometre range. The electrical contact between two adjacent CNTs is usually poor, hence the anisotropy ratio is chosen to represent the off-z-axis variation of the CNTs in comparison to their length. In Figure 4-9 this value is higher than $1 - \frac{200 \text{ nm}}{200 \text{ }\mu\text{m}} = 0.999$. Hence simulations running around this value were done, with the results in Figure 4-10.

Figure 4-9: SEM imaging of TCVD grown VA-CNT forests from the side. a: 21.03kX magnification, b: 296X magnification.

Figure 4-10: Effect of VA-CNT forest anisotropy on transmission. a: Operator=mean(S12[38-76G Hz]) versus anisotropy factor. b: Transmission parameter versus frequency, for given anisotropy factors.

The transmission decreases very fast with anisotropy lower than 0.9999. Yet looking at the transmission parameter, the transmission parameter retains its shape, while exhibiting a slight blue-shift. For lowest values of anisotropy, the transmission goes up again, with the cut-off visibly blue-shifted back to 50 GHz: higher anisotropy slow-wave layers act more as a lossy metal than a slow-wave layer. Which is somewhat consistent with Figure 4-8, showing the waveguiding properties can be kept with densities 100 times lower than the reference, hence 1/100 of the reference density in the off-axis direction cause the off-axis conductivity to be non-negligible anymore.

The anisotropy is an important factor for good properties of the waveguide, with harsh criteria of less than 0.05% of the conductivity off-axis. Then, increased value does not deteriorate the shape of the waveguide response, only its loss level.

As for the fabrication of test devices, the design of the waveguides without slow-wave is also adaptable to slow-wave usage, by replacing the normal metallic cap with a one on which a VA-CNT was grown on the metallic plane. The excitation and input lines features are to be changed accordingly but the changes are within a few microns, and can be ignored with minimal losses (2-3 dB).

Slow-wave μ TL

Another type of waveguiding device are the micro-strip transmission lines. The wave propagating in the substrate of a μ TL is mostly TEM, which means it is possible to use the same principle as for the slow-wave waveguides to generate slow-wave propagation in a μ TL with the use of VA-CNT forests below the line Figure 4-11. This idea is also supported by the work done in [10], where simulations of a μ TL with a propagation layer made of an alumina membrane filled with copper nano-wires was run.

Figure 4-11: Field representation (E in green, H in red) in μ TL and principle of slow-wave microstrip. a: Normal μ TL. b: Slow-wave μ TL.

If the topology exists in the literature, it uses top-down processes. The idea is to replace the anisotropic membrane made of copper nanowires embedded in a honeycombed alumina substrate with VA-CNT grown CNTs. Then the surface of those is to be covered with a dielectric layer, for instance SiO_2 .

For the same reasons as stated before, these simulations also require a small anisotropic metallic layer to be run by HFSS. Similarly, simulations were done to determine the effect of this layer to be negligible.

A first simulation using waveport feeding of the μ TL was done to exhibit the slow-wave effects. The slow-wave μ TL is setup with a 200 μm thick layer of VA-CNT using the bulk equivalent model as presented in chapter 1, with a 5 μm thick layer of SiO_2 ($\epsilon_r = 4$) on top and a metallization thickness of 1 μm . It is compared to a microstrip on 92pc

alumina substrate of the same dimensions as in Figure 4-12 and the results in **Table 4-2**.

Figure 4-12: Simulation of slow-wave μ TL versus reference designed on alumina. Both simulations are fed with waveports, have supposedly infinitely large substrates, the bottom ground plane is supposed perfect. The substrate dimensions are: 1000x1000x205 μm (LengthxWidthxThickness). a&b: simulation setups for no-SW(a) and SW (b). c&d: Waveport excitation mode for so-SW(c) and SW (d). e: Phase velocity in the μ TL. f: Attenuation constant in the μ TL.

Table 4-2: Extracted parameters from the simulations of no-SW and SW μ TL

Type	No-SW	SW
Width [μm]	31	8.5
Z_0 @ 50GHz [Ω]	95	100
Phase velocity @ 50GHz [multiples of c]	0.41	0.28
Attenuation constant @ 50GHz [Np/m]	17.7	42.5
$\epsilon_{r,\text{eff}}$ @ 50 GHz	5.949	12.76

The effective dielectric constant is more than doubled with slow-wave, while the width of the line is divided by almost 4 to keep the same impedance. The attenuation constant though is much higher, which can be problematic for resonant devices (lowers the Q-factor considerably). It is also worth noticing the properties are lowered proportionally to the ratio between the full substrate thickness and the thickness of the VA-CNT layer just as it is the case for waveguides. Yet, as opposed to waveguides, it is more relevant to keep the dielectric layer thickness to a fixed value, and to change the VA-CNT layer thickness, since the dielectric layer is chosen to be one to be sputtered, for fabrication purposes (see later).

Figure 4-13: α , Z_0 , $\epsilon_{r,eff}$ and v_p versus ratio cntT/(cntT+oxideT). Substrate dimensions: 1000*1000 μm (L*W). Oxide T = 2 μm . Line W = 20 μm . Metal layer: 5 μm .

In Figure 4-13 the parameters of a fixed width line were plotted against the ratio of the substrate filled with VA-CNTs. The oxide layer on top is Silicon Dioxide of fixed thickness 2 μm , while the VA-CNT layer thickness varies from 30 to 200 μm . The ratio is the thickness of the CNT layer on the total thickness of the substrate (CNT layer + oxide layer). First: the simulations tend to become unstable for ratios closing on 1. Then, the characteristic impedance increases a bit with the ratio and fixed width line, so to compensate the dimensions should go larger to reduce it. As for the attenuation constant α , it increases sharply when the ratio approaches 1. The losses generated by high ratios can be a problem for some devices, some care must be taken. The phase

velocity decreases linearly with the ratio, thus the $\epsilon_{r,eff}$ increases quadratically with it.

The values reach as high as 60, but with α values very high.

slow-wave stub

Figure 4-14: Simulation setup and S-parameter results for coplanar-fed slow-wave μ TL. VA-CNT thickness: 100 μ m, oxide Thickness: 5 μ m, metalization thickness: 1 μ m, μ TL length: 2 mm. Taper length to match coplanar pad = λ_g . At 50 GHz, transmission level is -0.47 dB. Dimensions in μ m.

To better exhibit these changes the idea is to fabricate a resonant device. The choice went for an open stub, as it is easy to design and fabricate. Additionally, as for waveguides, it is important that the device has coplanar inputs for coaxial input would exert too much pressure on the sample and crush the CNTs. The coplanar-micro-strip transition is the same as used for waveguides. A simulation on a simple slow-wave line was done as on Figure 4-14, which shows the transition works well for this design. Two μ TL lines were run to extract the losses from the transition and the line to be respectively 0.0498 dB and 0.185 dB.mm⁻¹.

Table 4-3: General parameters of quarterwave open stub for a 105 μ m thick substrate. SW substrate is 100 μ m thick layer of VA-CNT of 1015 m⁻² density and 5 μ m SiO₂. No-SW is 105 μ m thick 92pc Alumina.

	Line W [μ m]	Stub L [μ m]	Cop W [μ m]	Cop Gap [μ m]	Cop pad [μ m]	f_r [GHz]	S12 @ f_r [dB]	Q
no-SW	106	575	41	20	280	49.8	-22.413	86.91
SW	30	390	18	40	175	49.3	-31.213	65.85

As previously, the no-slow-wave reference will be of a same height substrate using 92pc Alumina substrate. The dimensions of the line are depending on the substrate, to match $50\ \Omega$, but the line length is constant and chosen to be 3 mm, to ensure the stub is far away from the coplanar pads. Then the dimensions of both devices are as in **Table 4-3**.

Figure 4-15: S-parameters of the quarterwave open stub. a: no-SW. b: SW.

The resonance is characterized in **Table 4-3**, and the parameters are plotted in Figure 4-15. In the case of the no-SW stub, the coplanar pads also act as resonators thus generating other resonance peaks. In the case of the SW stub, this effect does not appear, probably thanks to the slow-wave layer. In the latter case, the frequency response of a stub is very neat. In both cases, the resonance peak is located close to 50 GHz for the dimensions of the stub as given in **Table 4-3**. We note the dimensions of the stub being around 50% shorter for the slow-wave device, and its width more 3 times thinner. The quality factor is reduced though. The size reduction is consistent with the results on the μ TL alone, and thus it is possible to get even greater size reduction in both length and width of the stub. It is of importance to note that the quality factor is tied to the attenuation constant, and thus will only get lower with ratio increasing. Furthermore, the use of wet processes for fabrication is extremely complicated, with the layer of VA-CNT. Thus, fabrication is planned to be done using shadow mask

processes, which forbids to small dimensions, hence the choice to keep the ratio relatively small.

Measurements for pre-fabrication

To summarize the process flow for the stubs and the associated constraints in

Table 4-4.

Table 4-4: Constraints and limitations associated with each of the process steps necessary for the fabrication of the slow-wave stub.

Process step	Constraint/limitations	Critical ?
Gold metalization (bottom ground)	None	-
Growth of VA-CNT forest full plate (or localized below the lines)	Temp > 600 °C, gold roughness	Roughness of ground is irrelevant. Conductivity can be overcome with thick layer. Thickness of forest critical.
Oxide deposition	Only dry process: PECVD. Require long or multi-step deposition	Wet process forbidden. Thickness of Ox layer critical. Surface smoothness critical.
Metallic lines patterning/deposition	Only dry process: shadowmask constraint on dimensions	Dimensions on the line are not critical. Small variations are OK.
Probing/measurement	Pressure applied must be low	Can destroy the sample.

From **Table 4-4** we can see two steps are particularly important: the control over the thickness of the forest and the control over the thickness and smoothness of the oxide layer. The forest thickness is possible to control within a dozen of micrometres through quick recipe optimisation (see chapter 1), yet the precision required here is larger than that. Though it is possible to mitigate this effect to a certain extent through changing the oxide layer thickness on top. The thickness of the oxide layer can be controlled very precisely in PECVD process for oxides. The deposition is slow enough to have a good control on the thickness. The last important factor is the oxide surface roughness.

Surface roughness of the oxide is directly dependant on the surface roughness of the VA-CNT layer. We say in Chapter 1 that if the average roughness of the surface is acceptable, at the micrometre scale the surface is extremely rough. Although, this surface looks better for supergrowth CNTs. For good quality transmission lines, the typical roughness admissible is around ± 50 nm at these frequencies (50 GHz).

Figure 4-16: AFM of TCVD grown CNT forest. a: overview. b: Profile along the red line on the top image.

Figure 4-16 show a typical result for an AFM imaging of TCVD grown CNTs. The roughness is rather high, but deposition of SiO_2 usually smoothes the surface. It is still possible to reach the goal of ± 50 nm roughness which was done then measured again in Figure 4-17.

Figure 4-17: AFM imaging of TCVD grown CNTs with 2 μm of sputtered BN deposited on top. a: overview. b: profile along the red line on the top image.

The overall roughness jumped from ± 100 nm to ± 500 nm. Given the shape of the layer as seen on the overview in Figure 4-17, it is possible the deposition aggregates CNTs on the top part, and then forms the layer starting from the peaks, thus creating domes. This is further corroborated by the ridge, symptom of aggregation of CNTs. Another example of this effect can be seen on a CVD sample with 1 μm gold deposited on top as on Figure 4-18: the gold layer penetrates a bit the forest, while pushing small bundles together.

Figure 4-18: SEM imagings of CVD grown VA-CNT forest with 1 μm thick layer of gold deposited on top via e-beam evaporation. At the micrometer scale, the gold seems to penetrate inside the forest a bit. On a larger scale, the forest seem to be squashed a bit and the top is fractured.

After 2 μm deposited, the highest difference is greater than 1 μm , this kind of roughness is making any μTL fabrication on top impossible. A backup solution to this issue is to use supergrowth CNTs, as it was shown in chapter 1 that the surface roughness is better than that of TCVD grown CNTs. Furthermore, some recipes involving the passing of the precursor gas into a DI water bubbler yields extremely smooth surface CNTs (Figure 4-19).

Figure 4-19: Supergrowth CNT with H_2O bubble. Surface is shiny.

AFM measurements were done on the sample picture in Figure 4-19 before and after deposition of $\sim 1 \mu\text{m}$ thick SiO_2 via PECVD in Figure 4-20.

Figure 4-20: AFM imaging of supergrowth CNT. a: 5x5 μm probed site. b: zoom in on a flat surface. c: trace horizontally in the surface describe top right.

The surface of the device before PECVD deposition is very smooth, with some glitches probably due to localised overgrowth, which can happen because the catalyst layer is not clean enough or too old when the growth is done. Overall, the surface roughness is below the required levels of $\pm 100 \text{ nm}$ which is a good thing. The only problem with supergrowth is the difficulty to control the layer thickness for low values as the growth is too fast: $\sim 1 \text{ nm}$ in 120 s. If the glitches are troublesome, it's probably possible to not have them with a clear catalyst, or with a quick low energy plasma attack of the surface.

Figure 4-21: AFM imaging of supergrowth CNT with 1 μm PEVCD deposited SiO_2 layer on top. a: overview. b: overview with profile line. c: profile line along the line from top right.

The same effect happens as with the TCVD CNTs: the bubbly effect, though the effect is much less pronounced, with a min-max difference of 250 nm with only 1 μm thick layer of SiO_2 . It might be possible to smooth this out with thicker depositions or multiple depositions.

In conclusion, the fabrication of SW- μTl seems compromised by the surface roughness of the VA-CNT layer: if the current measurements are not completely against the possibility, it seems compromised to manage to get a surface roughness in the acceptable range through this method. A good way to circumvent the roughness issue is to change the topology of the transmission line.

Other topologies

The point is to change the fabrication and/or the topology of the line to solve the issue of the surface roughness of the bottom side of the line. It is important to note that the critical part is not the roughness of the top part of the oxide layer, but the bottom part of the metallic line.

Inverted lines

The idea is to revert the topology of the line: instead of having the ground plane below, it is on top on a flip-chipped substrate. Several variations of the topology exist (Figure 4-22), all with the main difference from the reference topology to have the line fabricated on the bottom substrate, then the ground plane fabricated on another substrate which is then flip-chipped on top.

Figure 4-22: topologies of SW- μ TL. a: reference topology. b: inverted line with CNT grown on the line and flip-chipped ground+oxide. c: flip-chipped ground+oxide+grown CNT. d: flip-chipped ground+grown CNT + oxide. Color code: Grey=Substrate, gold=metallization, black: VA-CNT layer, red: oxide.

Simulations of perfect excitations of the lines as described in Figure 4-22 bottom right are performed and results are shown in Figure 4-23. The equivalent dielectric

constant is rather high, and increases linearly with the VA-CNT layer thickness for constant oxide layer thickness. The losses though seem to increase quadratically. The simulations were done at 50 GHz. Yet as it shows in Figure 4-23:right, for a VA-CNT thickness of 250 μm , the line stop being excited passed ~ 50 GHz. As opposed to the reference design, the medium above the line is not air anymore, but silicon. The simulations were done with infinite substrates, yet the oxide and VA-CNT layers were of fixed width of 3 times the line width: since all the fields are localised around the line, this has little impact on the results. Additionally, here again an anisotropic imaginary metal was necessary to run the simulation, and as before, simulations were run to assess the impact of this layer on the results, which were deemed negligible.

Figure 4-23: Simulations of the system as on bottom right of Figure 4-22. Dimensions: substrate thickness: 500 μm , line w: 30 μm , line l: 2 mm, oxide thickness: 3 μm , oxide: silicon dioxide. CNT layer is of fixed width: 3 x line w = 90 μm . a: ϵ_r and losses versus the thickness of the VA-CNT layer. b: S-Parameters for CNT thickness = 250 μm

If the ratio VA-CNT to oxide becomes too great, the waveport excitation fails to excite the mode in the transmission line, but rather excites the evanescent mode in the substrate.

The bottom left design (as in Figure 4-22) shows very bad propagation hence results are not shown here. Top right is considered and the results are in Figure 4-24 and Figure 4-23. Same as for the previous results: the transmission stops for CNT

thickness values of 250 μm and above. The plotted H-field in Figure 4-24 shows the evanescent wave in the bottom substrate. For the ϵ_r and losses graph, the scale was purposely left as the one in Figure 4-23 and shows the dielectric constant variations are very similar until the waveguide mode excitation drops, with slightly lower losses for the bottom right setup.

Figure 4-24: Simulations on the setup as described Figure 4-22 - b. Dimensions: substrate thickness: 500 μm , line w: 30 μm , line l: 2 mm, oxide thickness: 3 μm , oxide: silicon dioxide. CNT layer is of fixed width: line w = 30 μm . a: ϵ and losses versus thickness of the VA-CNT layer. b: S-parameters for CNT thickness = 250 μm . c: Side view of H-field in the device at 50 GHz. d: front view of H-field in the device at 50 GHz.

If line simulations are promising, the feeding of such lines is difficult: the design is made with the intent of flip-chipping the top ground with a part of the slow-wave multi-layer propagation layer. Yet since the bottom line is fabricated on a substrate, the probing for measurement is done from the top, requiring a part of the line not covered with the cap, and a transition from coplanar to microstrip. Since the substrate has a

higher dielectric constant than the air above, the fields will be predominantly in the substrate, making the transition tricky.

The simulations with coplanar inputs were made after Figure 4-22 bottom right topology with some modifications for fabrication (Figure 4-25): The oxide layer is the same size as the μ TL instead of the slow-wave layer, to facilitate the fabrication and not have to deposit oxide on CNTs, which was proven to be rather difficult.

Figure 4-25: Simulation setup of the coplanar-input inverted slow-wave μ TL.
a: isometric view of the coplanar input and the line, the top substrate is hidden. b: Top view of the coplanar input and line, with layers transparent.
Dimensions: substrate thickness: 500 μ m, CNT thickness: 100 μ m, line length: 2 mm. The line dimensions were chosen to have a 50 Ω characteristic impedance in the μ TL. Dimensions in μ m.

The transition coplanar to μ TL is extremely short, but can be lengthened without additional losses according to simulations. Two different line lengths were simulated, to extract insertion and propagation losses: respectively 0.0756 dB/mm and 4.1279 dB. The insertion is very bad due to wave being confined in the substrate in the coplanar line. Another design (Figure 4-26) was done to try to adapt the transition better by displacing the transition to the μ TL under the slow-wave layer but the transmission stays below -5 dB.

Figure 4-26: Coplanar inputs with transition to μ TL under the slow-wave layer.

To conclude on the reversed topology slow-wave μ TL, the concept is sound and can reach up to $\epsilon_r = 16$ at 50 GHz with losses under 0.7 dB/mm. Nevertheless, too high thicknesses of slow-wave layer make the μ TL drop. The topology can work, but the excitation of the lines is a problem, as the line is fabricated on a substrate with dielectric constant above that of air and thus will contain most of the fields of the E-M wave, making the transition to the slow-wave μ TL difficult, as the fields in the latter are above the substrate in the slow-wave layer.

Conclusion

In waveguides or in microstrip transmission lines, extensive simulation work was done to show that the use of CNTs for slow-wave can yield effective dielectric constants of propagation more than 30. If the losses induced by extreme slow-wave implementation reach critically low levels, in more moderate cases it has been shown that it is possible to reach dielectric constants about 25 without too much additional losses. In practical, the size reduction of the devices is approximately half as long and one third the width for quality factor only 20% lower.

The advantage in comparison of other slow-wave implementations is that, as opposed to high-k dielectrics, the propagation medium is air, which allows for higher

quality factors at low filling factor for waveguides. In addition, the effective dielectric constant is very stable in frequency, supporting very high frequency, as opposed to micro-pillars meta-materials where the resolution of the pillars forbids too high frequencies.

Yet, preliminary fabrication steps have shown the fabrication of such devices requires a control over the process that is not yet in our grasps, especially the surface roughness requirement is quite stringent and is of critical importance.

Slow-wave is a nice addition to the set of passive devices, to enhance them by reducing size for better packaging and integration, especially at lower frequencies where waveguides are still bulky. In addition, slow-wave is necessary for certain implementations, such as waveguide feeding of antenna arrays, and thus expands the usage of the technology.

This further expands the scope of the use of CNT for passive devices, other more advanced devices will be studied under chapter 4.

CHAPTER 5: ADDITIONAL PASSIVE RF DEVICES USING CNT

In the previous chapters, carbon nanotubes were used for basic transmission purposes, with the purpose of serving as the framework for devices in the millimetre band. Basic passive devices and mostly the transmission function are primordial to the integration and packaging of novel devices up to 300 GHz.

The range of passive devices includes the transmission devices, not forgetting all the bends, couplers, mixers and splitters, with the more advanced designs such as butler matrix or rat-race coupler. But there are also filters and resonators to adapt impedance. The advantages of using carbon nanotubes for SIW is the possibility to go towards high frequencies without any problem regarding the resolution of the walls, as each nanotube features radii lesser than 10 nm. Also, the fabrication process allows fine patterning, and the large density of the nanotubes bypass the slightly larger equivalent dimensions intrinsic to the designs using large metallic pillars. These allow for design of precise devices, and the high quality factors make the technology efficient for filtering.

Carbon nanotubes SIW filters

The cavities as presented in chapter 2 could already be considered to be very simple first order filters. The objective is to present here more refined filtering devices. Filters were designed by partners in France, and fabrication was started for several types: order 2, 4 and 6, and some with step in the height of the filter. As the filters are based of the SIW design as described in chapter 2, they display a similar excitation with coplanar-pillar contraption. Figure 5-1 shows simulations for order 6 filter in lossy setup. The first post is doubled, to increase size of the posts. For single post, the first one

should have a radius of 164 μm , which can be difficult to fabricate using either transfer technique, or direct growth technique with high aspect ratio.

Filters were designed using as described in [91], then further optimized in HFSS by varying the dimensions.

a

b

c

Figure 5-1: Design and S-parameters of the lossy filter (order 6). Dimensions in μm . a: top-view of the filter. b: zoom in on the excitation. c: S-parameters of the filter. $Q = 9.96$. Bandpass filter designed for 33-37 GHz pass. Dimensions in μm .

Waveguides as such were designed for order 2, 4 and 6. Yet, the coplanar-pillar excitation as designed is efficient for waveguide heights of 400 μm and if this fits the order 6 filter, the 4th order filter has a ~ 750 μm cavity height and the 2nd order one ~ 1 mm. Although this adds complexity to the fabrication process, it is still doable using

multiple transfer with different heights (Figure 5-2), or if there is only one step: growth on the cap and alignment.

Figure 5-2: 2-stepped transition for taller filters. a: in principle schematics. b: Fabrication by layers. The steps allow for height transition between optimized transition height and filter height.

Simulation results for this design are in Figure 5-3 for a 4th order lossy waveguide. The height in the filter is 762 μm while the height of the SIW for the coplanar-pillar excitation is 512 μm .

Figure 5-3: Design and S-parameters of a 4th order CNT SIW filter with one transition step. a: S-parameters. b: isometric view, zoomed on excitation. $Q = 13$. Bandpass filter designed for 34-36 GHz pass. Dimensions in μm .

Some filter prototypes are under fabrication. If it was possible to reach heights of 500+ μm with TCVD growth, the original intent is to use supergrowth CNTs, for their higher profile and higher densities.

Smart Interposers

Interposers are substrates with main goal to transmit signal vertically from a motherboard below to a circuit above [92]. The interest in the technology has grown in

the past few years with many new 2.5D devices and applications. Typical interposers only transmit the signal from the motherboard below to ICs and dies above (Figure 5-4). The transmission is done with TSV in thin substrates, using bumps to electrically connect the different layers. The goals are multiple: for instance, the motherboard can be a computing silicon die, with an interposer to add additional functions fabricated on a different substrate. Another example in the recent commercial applications would be the latest video card from ATI, using an interposer to connect both the GPU and the RAM chips on a single interposer to the motherboard below.

The smart interposer, on the other hand, boasts all the basic functions of the interposer (basically only vertical transmission of the signal inside the substrate), but also integrate passive functions in the substrate, such as transmission, filtering, coupling, splitting.

Figure 5-4: Traditional interposer (a) versus smart interposer (c). Schematic representation.

Most of the work done on interposers focuses primarily on the vertical transmission of the signal, and mostly the way to decouple the different TSVs to increase the TSV density in the substrate [93], [94] with the resulting goal of increasing the data transmission rate from the motherboard to the top chip [95]. Mostly these are used for typical GHz electronics, but are sometime specifically tuned and designed for RF [93], [96] or optoelectronics [97]. The choice of substrate can be silicon, glass, quartz or ceramics. The closest there is to smart interposer in the literature comes from [94], where some capacitors are fabricated alongside the TSVs to filter-out the interferences caused by neighbouring TSVs, allowing for higher density TSV integration.

Figure 5-5: 1,2 &3 : Fabrication process of the functionalized interposer. Growth or transfer of VA-CNT layer on two metallized substrates; Alignment and flip-chipping of both substrates together. 4: Side schematic view with different layers. A: Top substrate with top interface. B: top slot metallization (fully metallized except waveguide excitation slots). C: Top substrate VA-CNT forest patterning the waveguides and functions, can be used for TSVs, and slow-wave. D: Same as C, for bottom substrate. E: Same as B, for bottom substrate. F: same as A, for bottom substrate.

The idea of smart interposers is made possible with the waveguides introduced in chapter 2. As stated, it is possible to excitate the waveguides with slots, and this from both sides of the waveguides working between 25 to 100GHz. Additionally, the high conductivity, and the fabrication method allow fabrication of TSVs to support the basic functionality of a normal interposer, but also supports a wide range of passive functions that are possible to integrate in the interposer itself. Instead of having one substrate, it requires two substrates with 1 or more layers of VA-CNT sandwiched in between (Figure 5-5).

Most the results on the simulations on this kind of device was also studied in chapter 2. Other devices, such as filters, can also be fitted inside. Couplers, splitters and bends were studied with the coplanar-pillar excitation and are useable with this technology. Some more complex and advanced passive devices can also be implemented in this technology, thanks to the ease of patterning the VA-CNT forests, be it with direct growth method or transfer. The comparatively very high densities of the forests also allow for very sharp designs with effective dimensions of the waveguide not exceeding the geometrical ones, as opposed to micro-fabricated pillars SIWs. For example, Butler matrixes and rat-race couplers can be fabricated as the curvature and the precise lengths are easily attainable.

Nano-antennas

As opposed to the previous work that was done, this work also makes use of the quantum properties of the carbon nanotubes to generate slow-wave propagation. Carbon nanotubes in VA-CNT forests can be used for fabrication of quarter-wave monopole antennas on substrate [41], [98] 100 GHz and above, limited by the size of the antennas (around 700 μm and below). At these frequencies and efficiencies, the main goal for the technology is onboard wireless transmission. In addition, relying on specific properties of the CNTs as described in [58], [99] known as quantum

capacitance and kinetic inductance, a slow-wave effect is induced in carbon nanotubes, which could lower the working frequency of the antennas without requiring difficult or impossible to fabricate straight antennas. According to the work in [99] a single nanotube dipole antenna could exhibit slow-wave effects resulting in phase velocity being $1/100^{\text{th}}$ that of the freespace celerity. Additional work conducted in [29], [41] expanded the calculations to multiple CNT antennas which mitigates the slow-wave effect to half to a third of the freespace celerity. The lower the number of CNTs, the higher this slow-wave effect while the resistance and thus dampening of the antenna also increase. Designs were thought of to reduce as much as possible the number of CNTs in the antennas while keeping mechanical stability necessary for the high aspect ratios required for monopole designs, hence the triangle-star shape.

Figure 5-6: Nano-antenna fabrication and characterization. a: SEM imaging of nano-antennas in transmission. b: Simulations design based on the SEM imaging of the monopoles. c: Measurement in the 220-330 GHz band. d: Simulations in the 220-330 GHz band.

Fabrication was done according to the design specifications in [41], [98] and measurements were also done in IEMN (Lille, France), and retro-simulations were done to match the fabricated monopoles (Figure 5-6). Measurements show a very faint transmission maximum at 250 GHz with a matching reflection dip. The width of the peak, and the absence of it on the simulation seem to go against the device working. More work is underway to verify this data. Retro-simulations though find a matching behaviour for frequencies around 400 GHz. This mismatch is either due to the density of the CNTs fabricated not matching the simulations (10^{14} m^{-2}), or the length chosen in the simulations not fitting.

Figure 5-7: Simulation of the half-wave feed CNT monopole antenna. Top: Simulation setup with dimensions and surfacic current plotted on the line. Bottom: Reflection coefficient on the port, with the radiation pattern inserted. VA-CNT forest density: 10^{13} m^{-2} .

Anyway, the results being poor, the design was questioned after new insight about CNT growth came, especially about the height attainable, and the density. The impedance of the antenna was evaluated to be $190\ \Omega$, and a halfwave line input was designed to adapt the antenna to the $50\ \Omega$ input coplanar line (Figure 5-7). The design focuses on a single monopole, without transmission thus. The half-wave input ensures good impedance matching but is much more sensible to imperfections in fabrication and non-conformity and at the same time narrows the bandwidth of the monopole antenna. Nevertheless, the response of the monopole antenna made of carbon nanotubes is very sharp, and the radiation pattern is matching that of a monopole, with slight asymmetry caused by the intrinsic geometrical asymmetry of the access. A simulation was also done without the antenna, to confirm the resonance is due to the antenna and not the half-wave match.

In the design of the monopole in Figure 5-7 the density of the VA-CNT forest composing the monopole was chosen to be $10^{13}\ \text{m}^{-2}$, and the height is $105\ \mu\text{m}$. The resonant frequency of the antenna is $200\ \text{GHz}$ while the impedance of the input is $50\ \Omega$ at this frequency. To compare, freespace wavelength at $200\ \text{GHz}$ is $1500\ \mu\text{m}$, which makes a quarterwave length of $375\ \mu\text{m}$. Using low density carbon nanotubes here allow the antenna to be only $105\ \mu\text{m}$, which is a length reduction of a factor 3.57.

Design & fabrication

For simplicity's sake, the fabrication designs do not use this half-wave input design which is extremely sensitive to imperfections. Instead a simpler impedance adaptation is used (Figure 5-8).

Since monopole antennas are not directional at all (as in Figure 5-7), ways to make the antennas more directional were studied: reflectors and Yagi-Uda antennas. The idea behind the reflectors stem from the fact that VA-CNT walls can be used for E-M shielding [100] and their use as walls for SIW as in chapter 2. Yagi-uda antennas

using VA-CNTs work well, and it is possible to fabricate different height VA-CNT pillars thanks to the transfer method of CNTs. Yet Yagi-Uda design requires very precise heights for the reflector and the directors, and thus is very difficult to fabricate using VA-CNT methods. A design with same height reflector and directors has a lower directivity without the issue of being difficult to fabricate.

Figure 5-8: a: Mask design of nano-antenna used in transmissions. Orthogonally set inputs, and different spacing ranging from 1 mm to 4 mm in between monopoles. b: CNT reflector for nano-antenna.

Flexible devices

Yet another way to use carbon nanotubes in RF devices is to use them on flexible substrates in transmission lines, to take advantage of their conductivity and high mechanical resistance. Flexible electronics and RF devices are need for wearable electronics. One of the main issues with flexible electronics is the wear of the lines during and after deformation of the substrate. Usual metallic lines cannot be used for they would break and shatter. The detrimental effect of this is even more pronounced for RF devices, as even after deformation, breaks and rifts will dampen the RF signals much more than low frequency and DC signals. General idea is to use ink-jet printing and conductive inks, which are much more flexible than metallic lines. Yet, most commonly silver nanoparticle ink is used, but is still subject to cracks and tear, which makes it very difficult for RF devices.

The main idea is to use a conductive polymer: poly (4-styrenesulfate) (PEDOT:PSS), and to mix in metallic CNTs in solution inside to improve the conductivity of the PEDOT. CNTs are intrinsically anisotropic, and so is their conductivity, which can be a problem for electric lines. Since the layer of PEDOT is supposedly lesser than the length of the CNTs, they will tend to arrange in the plane of the lines and not vertically, improving the conductivity in the direction of interest.

Two kinds of devices were designed, fabricated on PDMS and measured: 50 Ω μ TL and a 13 GHz patch antenna. Designs and simulations were done using ANSYS HFSS. The PDMS substrate has dielectrics properties as follow: $\epsilon_r = 2.68$ & $\tan \delta = 0.04$ at millimetre-wave frequency range [101]. PDMS substrates of 180 μ m for μ TL and 360 μ m thicknesses are fabricated by spin-coating a film of PDMS on a 3" silicon wafer coated in C_4F_8 anti-adhesive layer, and then cured at low temperature. The metallization of the devices was fabricated using ink-jet printing. Since the PDMS substrate are hydrophobic, an O_2 plasma at low power treatment is required prior to printing, to render the surface hydrophilic: contact angle before and after plasma is respectively 115 $^\circ$ and 40-50 $^\circ$.

Figure 5-9: Topologies of the PEDOT usage for flexibility in RF devices. a&b: PEDOT inclusion in lines. c&d: Sandwiched line.

Both MWCNT/PEDOT:PSS and Ag ink are printed using ink-jet printing. The idea is not to fully replace the Ag ink metallization with PEDOT, as the conductivity difference is too big, but to add some PEDOT to increase the flexibility of the metallization. Several topologies were considered (Figure 5-9): PEDOT inclusion in full Ag devices, PEDOT sandwiches and its variations. PEDOT inclusions are designed so that the Ag ink line is broken into small parts that would resist better flexion, linked together with PEDOT inclusions. Sandwiched lines are made so that under flexion the Ag ink lines would break, but the PEDOT part of the line would hold, and ensure contact during and most importantly after the flexion.

Figure 5-10: Designs and simulation of flexible patch antennas. a&b: Designs with dimensions. Both patches have the same dimensions. c&d: Simulation results compared with antenna without any PEDOT. Left: Inclusion design. Right: U design. All dimensions in μm .

If the principle is obvious for lines, it is less for patch antennas. Some results were published in [28] for a patch antenna with PEDOT inclusion in the quarterwave matching line without much damping of the response of the antenna and good matching of the simulation results. Yet, the patch being large ($6900 \times 8510 \mu\text{m}$), its resistance to flexion is quite low. Hence different designs were explored (Figure 5-10). The idea is to keep Ag ink with higher conductivity where the currents are strongest, while replacing the rest of the patch with lower conductivity MWNT/PEDOT. When the adding of a PEDOT inclusion simply adds losses without impacting the resonance, the addition of PEDOT in the antenna patch itself seriously damps the resonance of the antenna. The resonance can still be seen, hinting the concept might be improved, for example by using a meshing of Ag ink patches or islets linked with PEDOT, or tessellation with squares or triangles.

Conclusion

Some other uses of CNTs for RF applications were briefly introduced which could complement and complete the SIW and slow-wave propagation devices that were presented in chapter 2 and 3. If the flexible RF are anecdotic and not possible to combine with the SIW technology, it still adds some insight on the possible use of CNT

in chaotic arrangement. The common point of all those technologies is that they rely lightly on the individual characteristic of carbon nanotubes, as opposed to typical applications using CNTs in the literature. As such, they suffer less from the lack of precise control over the processes of fabrication and placement of CNTs on a substrate.

CHAPTER 6: CONCLUSION

Summary

Carbon nanotubes, their properties, fabrication and uses have been explored, and more specifically in their vertically-aligned forest form. CVD is the method of choice to obtain such VA-CNT forests, and thus the VLS mechanism underlying the growth process was explored as well as the effectively used growth machine and method used in the thesis. Then a model to use for simulations of CNT in typical finite element modelling software was described as it was used thoroughly in the thesis.

In chapter 2, CNT-base silicon integrated waveguides were introduced as a possible answer to the problematic, and thus their properties, design, fabrication and characterization is discussed in length. Waveguiding is studied, and then two specific feeding topologies are examined. As the conductivity of the CNTs become an important factor in the functioning of the devices, another design based off the SIW presented before is devised to characterize more precisely the VA-CNT forests conductivity. This proof of concept is important as it validates the model used, it also shows the possibility of fabricating SIW using bottom-approaches which has better resolution than existing methods, allowing for new devices to be fabricated.

Then as an improvement of the devices introduced in chapter 2, in chapter 3 we focus on the study of slow-wave propagation, to answer other questions raised in the corollaries of the problematic: miniaturisation. CNT are used for their intrinsic geometric anisotropy to form materials with anisotropic conductivity, forming a layer in which electric field cannot propagate while magnetic field can. This is adapted to be fitted in the waveguides as described in Chapter 2, simulations show size reduction up to a factor 4 for waveguides using this method. Another guiding topology is then studied: micro-transmission lines, and how to achieve slow-wave in this topology using carbon nanotubes. Here we demonstrate via simulations effective dielectric constants for the media of propagation reaching 20 and above, while keeping losses under control. Size

reduction is always a goal in itself, and these results show it is possible to do so while keeping air as the propagation medium, as opposed to existing solutions. This allows for theoretically higher Q-factors and lower losses.

Then finally other RF passive devices using carbon nanotubes are introduced that can complement the array of functions that can be offered to answer the problematic. Filters, smart interposers, nano-antennas and more anecdotic flexible substrate electronics using carbon nanotubes are investigated.

Limitations

In chapter 2 some limitations on the VA-CNT forests were observed, but their impacts on the functioning of the devices at this point was deemed minimal. In the case of slow-wave propagation, these defects take more importance, as we can see the non-perfect anisotropy, added to the imperfect surface aspect can lead to tremendous losses. In addition, in the devices introduced in chapter 4, the dimensions of the elements are extremely precise and cannot be changed easily. The control over the growth of carbon nanotubes is still imprecise in terms of length and quality of nanotubes, and that lack of consistency makes the designing and fabrication of precise devices difficult. Yet, progress is consistently made in the literature on the control of growth and quality of CNTs which would impact positively the work done here.

Another point to raise is the high temperature process of the growth of CNTs. This can be dropped down to 500 °C (at the cost of sacrificing the quality of CNTs) with some tuning on the recipes but the temperature still is too high for most of the commonly used processes. Additionally, CNTs cannot be used with any kind of wet process for they would aggregate and lose their shape and properties. The use of transfer method can alleviate these problems, but comes with its own complexity and problems.

Future openings

The most important part here is to be able to devise a new method, or improve current method of fabrication of CNTs to reach a very consistent way of fabricating carbon nanotubes. Deeper insight into VLS mechanism could allow for more precise CVD growth of carbon nanotubes. TEM characterisation of CNTs could offer some valuable information on the quality of the CVD grown CNTs.

Additionally, the use of carbon nanotubes in metallic waveguide was not studied here as it does not fit in the problematic, as metallic waveguides are bulky and cannot be used for integrated devices. Yet metallic waveguides are extensively used, especially for high power applications, and it can be interesting to consider the use of carbon nanotubes for slow-wave in such devices.

Finally, the work in this thesis was focused on the millimetre wave frequency band, and if going lower in frequency is not suitable for waveguides, it would be interesting to check the behaviour of the CNT SIW in the terahertz domain, and their possible use for meta-materials fabrications. The very high density of VA-CNT forests should allow for increase in working frequency without much problem of resolution, and might also bring in the quantum properties of the carbon nanotubes in improve the devices.

CHAPTER 7: PUBLICATIONS

Theoretical study of CNT based waveguides: on the simulations and results coming from chapter two. The design, simulation, fabrication and characterization of CNT based waveguides. [103]

Dispositifs millimétriques passifs à ondes lentes à base de nanotubes de carbone : on the simulations and results coming from chapter 3. The design, simulations and preliminary experimental results of CNT based slow-wave devices [102]

CNT based smart interposers: patent application for some of the results from chapter 4. Concept and simulation results for interposers including RF passive components.

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