

SYNTHESIS REPORT

FOR PUBLICATION

HIGHTC SUPERCONDUCTING THIN FILMS FOR MICROWAVE APPLICATION'S "SURF"

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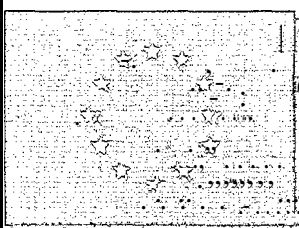
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2 SUMMARY

2.1 FIVE KEYWORDS ON THE CONTENT OF THE PROJECT

- Superconductors
- Surface Resistance
- Microwave Microelectronics Technology
- Microwave Components
- Cryogeny

2.2 ABSTRACT OF THE RESULTS AND BENEFITS OF THE PROJECT

Superconductors are known to provide very low electrical dissipation, since the resistance goes down to zero for direct current, and is several orders of magnitude lower than normal metals for alternative currents up to hundreds of gigahertz when they are operated below a critical temperature T_c . In 1986 a new family of phases has been discovered leading to compounds which can be used at liquid nitrogen temperature (i.e. 77 K or -196 °C). The easier accessibility of this temperature (versus liquid helium which was previously required for superconductors) urges up new applications for these materials. Early results on YBCO thin films deposition promoted electronics applications and more specifically microwave systems for radar, telecommunications...

The industrial and technical objectives of this project were:

- availability on the European market of microwave quality High Temperature Superconducting thin films:

- surface resistance R_s lower than 0.5 milliohm at 10 GHz and 77K
- low loss dielectric substrate
- homogeneous large areas (50 mm)

- supply of microwave components using these films - i.e. components which cannot be made without the use of superconductors and whose lack on the European market would lead to a technical dependency:

- multiple tap delay line: basic component for analogue signal processing:
 - direction finding systems (goniometers)
 - "instantaneous" digital frequency meter
 - microwave spectrum analyser
 - high frequency correlators for spread spectrum communications
- high Q microwave resonator ($Q > 1000000$) for low phase noise oscillators:
 - improved communications
 - safer air traffic control
- filter banks for mobile communications
- demonstration of microwave detection and slow wave effect

The goal of the project was to cope with three problems concerning the application of superconductors to microwave industrial problems:

- 1) to define really pertinent devices. Operational devices must be defined from specific needs and require a careful analysis of the whole problem (expected performances, volume, weight, including cryogeny).

- 2) to produce homogeneous, reproducible films. Most of the published results correspond to small samples (typically 10 x 10 mm) 50 x 50 mm areas are highly desirable for practical applications.

- 3) to make these devices. Operational devices imply very tight specifications defined by end-users, which adds design difficulties independently of the superconducting aspect. They require top level modelling.

The initial specifications were:

- 1- YBCO thin films: 500 nm thick,
- 2- surface resistance $R_s < 0.5$ milliohm at 10 GHz and 77 K,
- 3- homogeneous area: 050 mm
- 4- ultimate objective: $R_s = 0.1$ milliohm
- 5- multiple tap delay line : more than 16 taps with 0.1 to 1 ns delay between taps
- 6- narrow band filter: relative bandwidth $< 0.5\%$
- 7- 3D high Q resonators : $Q > 300000$ updated to 1000000 at 10 GHz
- 8- slow wave effect demonstrator : slowing factor > 5
- 9- microwave superconducting detectors competitive with semiconducting diodes

Most of these specifications (1,2,3,5,6,7) have been met except the more exploratory ones (4,8,9). The material specifications (1,2,3,4) provide a two orders of magnitude improvement versus the values of the metallization used in microwave devices at room temperature. This implies to reconsider the design of some usual components : planar resonators can replace metallic cavities with equivalent performances for the design of filter banks and with a considerable gain in weight and volume especially at frequencies below 5 GHz.

At the completion of the research (01/03/1996), several objects are available:

- Low R_s superconducting films: $R_s < 0.4$ milliohm
- C band narrow bandwidth filter: bandwidth = 0.5 to 1%
- High Q 3D resonators (10 GHz): $Q = 1300000$
- Tapped delay line (up to 10 GHz) : delay = 4 nanoseconds

3 THE CONSORTIUM

The objectives of the project required two kinds of know-how which were supplied by the different partners:

- material mastering
- devices design and fabrication

3.1 Names And Addresses Of The Partner Organisations

THOMSON-CSF LCR: J. C. Mage

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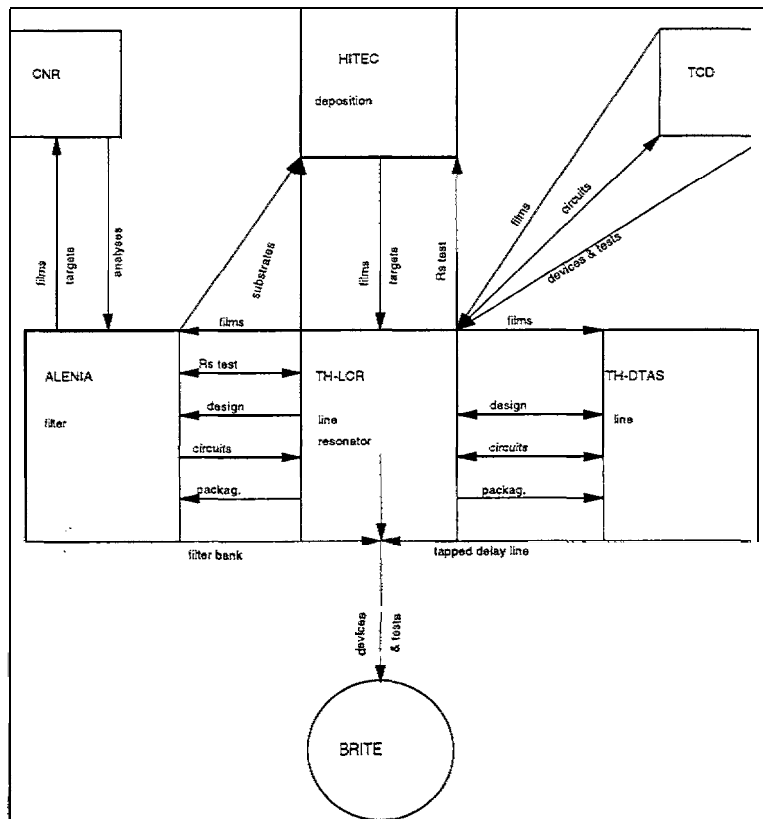
THOMSON-CSF MICROELECTRONICS (TCM): Mr Roussel

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THOMSON-CSF RADAR CONTRE MESURES: Mr Canal

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3.2 Consortium Description



The core of the consortium was constituted by two big industrial partners: the French company **Thomson-CSF** and the Italian one **Alenia**. Both of them are involved in the microwave business (radars, communications and satellites). Two divisions of Thomson were involved in the project: the LCR (Central Research Laboratory) and *Thomson-Sintra DTAS* (devoted to the production of components for analogue signal processing).

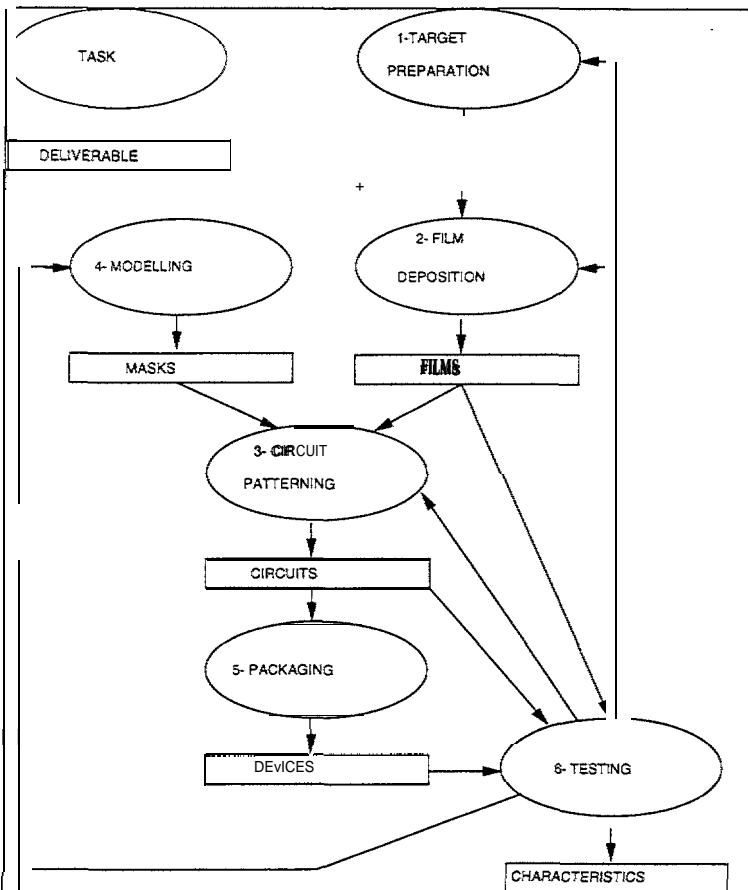
Around this core, **Hitec** is a German SME involved for seven years in material processing (special materials and thin film coatings) and connected with the nuclear research center of Karlsruhe (not directly involved in the project). Trinity College Dublin (**TCD**) is the Irish university already involved in superconductors through a SCIENCE project. **CNR-ITSE** (presently **CNR-ICMAT**) is the Italian National Research Center associated with Alenia.

Partnership structure:

- Hitec is a target, deposition equipment and film supplier for the consortium and for the merchant market
- Thomson is a manufacturer and end-user of 3D resonators and tapped delay lines
- Alenia is a manufacturer and end-user of filter banks
- the contribution of TCD concerns the *exploratory* aspect of the project
- the contribution of **CNR-ITSE** (presently **CNR-ICMAT**) is complementary of Alenia's activities

This structure has been maintained over the three years of the contract except that:

- *Thomson DTAS* (presently renamed *Thomson Microsonics*) was replaced by
- Thomson **CSF** Microelectronics (**TCM**): manufacturer
- associated with Thomson Radar & Contre-Mesures (**TRCM**): end user



Tasks:

- the target preparation (task 1) was mainly effected by Hitec.

- all the partners (except Th-DTAS) were involved in task 2 (films), each with a specific activity:
 Thomson-LCR: lowest surface resistance
 Alenia: reproducibility
 Hitec: large areas (up to 50 mm)
 TCD: very thin layers (less than 100 nm)
 CNR-ITSE: alternative materials

- all the partners (except Hitec and CNR) were involved in tasks 3, 4, 5,6 (device design, fabrication and testing) with different devices:
 Thomson-LCR: multiple tap delay line
 high Q resonator for radars
 Alenia: filter bank for satellites
 TCD: microwave detection
 slow wave effect
 TCM: pre-development
 multiple tap delay lines

Table 0.4. I: initially planned contribution of the different partners to the different tasks %

task \ partner	country type							total per task
	1 (C) Th-LCR F Industry	2 (PR) Alenia I Industry	3 (PR) Hitec D SME	4 (PR) TCD IRL Univ.	5 (AP1) TCM F Industry	6 (AP2) CNR-ITSE I Research		
- 1-targets	1	4	8	0	0	3	16	
-2-films	10	6	12	2	0	8	38	
-3 -patterning	3	4	0	0	3	0	10	
-4-modelling	3	2.5	0	3.5	4	0	13	
-5-packaging	5	2.5	0	1.5	2	0	11	
-6-testing	3	3	0	4	2	0	12	
total per partner	25	22	20	11	11	11	100 %	

The main tasks remained unchanged during the completion of the project, and the relative contributions of every partner to every task were almost unchanged. Minor modifications were necessary in the definition of the subtasks:

- task 1: targets - unchanged
- task 2: films:
 - subtask 2.1: *low Rs films* becomes low **Rs** and large areas films
 - subtask 2.2: reproducibility - unchanged
 - subtask 2.3: *large area films* becomes alternative substrates
 - subtask 2.4: very thin films - unchanged
 - subtask 2.5: film deposition and characterisation - unchanged
- task 3: patterning:
 - subtask 3.1: *chemical etching* is re-oriented to ion milling (3.3)
 - subtask 3.2: *ionic etching* is renamed plasma etching
 - subtask 3.3: is created" alternative etching methods"
 - subtask 3.4: industrial aspect is renumbered from 3.3 to 3.4
- task 4: modelling - globally unchanged except a major contribution of Thomson to filter modelling (4.3)
- task 5: packaging - globally unchanged
- task 6: testing - globally unchanged except an increased contribution of Thomson to resonant lines testing (6.2)

4 DESCRIPTION OF THE ACHIEVEMENTS

Among the new superconducting phases - all of them belonging to the family of the cuprates - we chose to deposit thin films of $YBa_2Cu_3O_{7-x}$ or YBCO - because this compound has a critical temperature $T_c = 91$ K well above liquid nitrogen temperature and its chemical stability was sufficiently established.

Among the deposition methods we selected sputtering because it had already provided good results and it was appropriate to large areas (see 4.2.). In order to get homogeneous films a special configuration is required i.e. Inverted Cylindrical Magnetron or ICM. A plasma is ignited inside a hollow cathode in order to prevent any back sputtering of the film during deposition. The special cylindrical targets which are needed by this process have been optimised during this project (see 4.1.).

High quality films require an accurate control of the substrate temperature during deposition, which is achieved by means of a heating substrate holder which has been developed for this purpose (see 4.1. & 4.2.).

As these films are intended for microwave devices, their microwave properties i.e. the surface resistance must be carefully controlled. This measurement was performed thanks to a specific method using a dielectric resonator which has been improved in order to make possible mapping of large films. A single crystal of rutile is used as resonator in order to probe small areas of the film thanks to the high permittivity of rutile (see 4.5.).

Microwave devices are designed thanks to CAD's which solve the Maxwell's equations and which have been carefully evaluated during the project (see 4.3). These devices are then patterned by means of different etching techniques which have been tested (see 4.4.). The circuits are packaged in order to meet both microwave and cryogenic requirements (see 4.4.). Then they can be tested by microwave network analyser (4.5.).

4.1 TASK 1: TARGETS FOR ICM SPUTTERING (HITEC)

The objectives of HITEC-Materials were based on the following topics:

- a) Improvement of small targets (diameter 50 mm)
- b) Development of a larger sputtering gun ICM 150 for deposition of large areas.
- c) Fabrication of large cylindrical targets with diameter 100 mm.
- d) Development of devices required for sputtering of superconducting films: - heaters, complete sputtering

4.1.1 Shaping Of Targets By Pressing And Sintering

The standard way for the fabrication of targets is cold axial pressing and sintering. However, cold axial pressing of $YBa_2Cu_3O_{7-x}$ powders leads only to bodies with green densities in the 60% range of theoretical density (TD). Sintering of axially pressed targets performed in the temperature range up to the decomposition temperature of the peritectically melting $YBa_2Cu_3O_{7-x}$ phase leads to bodies with densities up to at least 72 % TD. HITEC-Materials developed a special near net-shape isostatical cold-pressing and sintering technique to increase the densities of ring targets required for the Inverted Cylindrical Magnetron, depending on their size up to 8S %TD. During the project we enlarged the size of the hollow cathodes up to diameter 100 mm. These targets can now be fabricated routinely with very constant properLies.

4.1.2 Targets Bonded Into Copper Rings

The sputtering with improved targets fabricated by cold isostatical pressing and sintering showed that the mechanical stability and the thermal conductivity should be increased even further. The low heat conduction of the target to the target housing leads to overheating and glazing of the sputtering surface of the target. Moreover, the target becomes non conducting. The heat transfer to the target housing could be increased by a higher heat conduction of the target and/or by a better heat transfer to the water-cooled target housing. We developed a bonding technique of the hollow cathodes into copper rings at elevated temperature. First, the mechanical stability is increased by a pre-stress. So the target can withstand higher thermal radial stresses and is protected against mechanical damages during handling and mounting. Second, smaller thermal resistivity between copper ring and copper target housing occurs than between standard $YBa_2Cu_3O_{7-x}$ target surface and copper target housing. Meanwhile most of the superconducting targets for hollow cathodes are ordered at HITEC-Materials as targets bonded into copper rings.

4.1.3 Targets Fabricated By HIP

Our work on shaping of targets by cold isostatical near net-shape pressing and sintering showed that the densities achieved are not higher than about 88% TD. It is known that sintering under a simultaneously applied pressure leads to higher densities. Based on this knowledge we started densification of YBCO by Hot-Isostatic-Pressing (HIP). For performing HIP, the targets have to be vacuum-tight sealed into a container which will not react with the target at densification temperature, The density of this sample is about 95 % TD.

4.1.4 Improvement Of Sputtering Heads

With the ICM 100, which was available and which had been tested over an extended period of time at the beginning of this work, it was possible to deposit YBCO on substrates with diameters of up to about 30 mm. The new version of ICM 100 shows smaller tolerance levels, and for mounting of target only the magnet ring and the target fixing support have to be dismantled. In the beginning of this work, a prototype of ICM 150 had just been designed and set up. This is a larger sputtering head with a ring target of diameter 100 mm. The ICM 150 was developed for the sputtering of $YBa_2Cu_3O_{7-x}$ films on substrates with diameters ranging from 2 to 3 inches.

The most important changes to improve ICM 150 were the following:

- flat layout of the imer anode
- magnet positioned at the outer anode
- positioning of the cathode cooling near the target
- direct coding of the copper block of the imer anode
- reduction of tolerance limits for mechanical working
- decreasing of outer dimensions of cathode to fit the anode through a DN 150 flange
- increasing the mounting depth up to 200 mm.

The first new ICM 150 was delivered to Thomson for sputtering larger areas. Especially the changes in the layout of the imer anode and the arrangement of the magnets lead to a higher homogeneity in sputtering rates.

4.1.5 Development Of New Heaters

For the deposition of superconducting and c-oriented YBCO films substrates have to be heated up to temperatures in the range of 700 to 800° C. This requires surface temperatures of the heater up to about 950° C, depending on the kind of coupling from substrate to substrate heater. Other requirements for the heaters were:

- heaters have to run in O₂-atmosphere
- a homogeneous temperature profile has to be present on the surface of the heater
- heaters have to be thermal shock resistant
- heaters should have an active area of up to 2 to 3 inches
- heaters should not contaminate the lower and upper surface of the substrate
- both side deposition of films on the substrate should be possible in sequence.

We designed and setup a metal plate heater with diameter of about 60 mm.

The most important features of this heater are:

- heater surface and housing made from high temperature/high corrosion resistant steel
- special O₂-resistant wire as heating coil
- temperature measurement with thermocouples on surface of heater
- special winding of the heating coil in order to obtain a constant temperature profile on the surface
- additional radial thermal shielding.

The measurement indicates that a homogeneous area of diameter 40 mm is present. The highest deviation in temperate is about 5K. The life time of the heater was increased when exchanging the Cr/Ni heating wire for a Molybdenum wire. Operation up to 950° C surface temperature is now possible without problems. However, large substrate heaters working at temperatures higher than 830° C require a shielding and cooling device for the magnet ring of the ICM to prevent them from damages. We developed a substrate heater working with infrared radiation. The installed power is about 2000 VA. The housing of this substrate heater is completely water-cooled. Only the surface is warm. The characteristic of this system is that the substrate is directly heated by the radiation of the lamps. Using this technique of cold surface and direct heating, the contamination of substrate surface were drastically reduced. This system allows the deposition of films on both sides of the substrate. The complete heated surface is about 150 mm in square. The substrate temperature and the required power are controlled by thermocouples.

4.1.6 Conclusion

The state of the art at the beginning of the project was the fabrication of small ring targets (diameter 50 mm) with densities in the range of 75 to 80 % ρ_D . The disadvantage of these targets was their low density which caused problems in sputtering, e. g. mechanical stability and thermal conductivity. By optimisation of the fabrication process for the precursors which have to be calcinated, e. g. from Y₂O₃, BaCO₃, and CuO, and developing a near net shape isostatic pressing and sintering process. We succeeded in manufacturing targets with densities up to about 87 % ρ_D . By the performed characterisation work using X-Ray diffraction, metallography, differential thermal analyses, and resistivity measurements, we were able to demonstrate that the targets are of high quality in composition and homogeneity. With the help of additionally developed bonding techniques into copper ring, we managed to further increase the mechanical stability and the thermal coupling to the target housing. Furthermore, the targets bonded into copper rings are easier to mount in the target housing, and lead to a more uniform sputtering.

For sputtering large areas, we improved the small ICM 100 and developed an ICM 150 for working with hollow cathodes of diameter 100 mm. With this large sputtering head, deposition of YBCO films homogeneous in thickness and composition could be performed up to about 3 inches, At HITEC-Materials the ICM 150 is also used to sputter CeO₂ buffer-layers. These ICMS can be run in the wide pressure range from 0.2 to 2 mbar. Beyond the improvement of sputtering heads to deposit films on large substrates, we also developed two kinds of substrate heaters for working with substrates up to 3 inches. The first one is a metal plate heater which allows working with surface temperatures up to 950° C. The heating coil is fabricated from Molybdenum wire to ensure long life. The second one is a heater which is working with infrared radiation lamps. With this heater substrates can be heated up to about 820° C without contaminating their surfaces. This heater was developed with the aim to achieve deposition of films on both sides of the substrate. This heater has been successfully used for the deposition of CeO₂ buffer-layers.

4.2 TASK 2: FILM DEPOSITION

The critical parameters have been identified and optimised in order to allow the reproducible deposition of films with:

- Normal resistivity: $\rho < 220 \mu\Omega \cdot \text{cm}$ at 300 K
- Critical transition temperature : $T_{R=0} > 88 \text{ K}$.
- Sharp DC transition: $\Delta T < 1 \text{ K}$.
- DC $R(T)$ slope : $R(T=300\text{K}) / R(T=100\text{K}) -3$.
- Critical current density : $J_c 24.106 \text{ A} \cdot \text{cm}^{-2}$ at 77 K
- Surface resistance (10 GHz 77 K): $R_s \leq 0.5 \text{ m}\Omega$

This six criteria correspond to increasing difficulty. The first criterion is a very simple routine test which permits to eliminate very poor films. The four next ones are routine tests for superconductors. The last one represents the main goal of the project and it has focused most of our efforts.

General comment about film deposition

All the partners agreed with the fact that YBCO films are much more difficult to deposit than other usual materials. This is obviously due to the nature of YBCO which is first an oxide - with all the problems of oxygen stoichiometry -, then an unstable oxide - with all the problems of decomposition, formation of complexes, ageing of the target. This induced a poor reproducibility. The global yield could be very low due to the low yield of every step of the process. The global yield which is equal to the product of the partial yields tends rapidly to zero as soon as the success ratio of every step was less than 50% and as the number of steps increases. Thus, we should get a yield of 6.25 % for 4 steps, which was not that far from reality at the beginning of the project. Most of the problems are now well defined and efficient solutions have been proposed

- 1- surface state of the substrates: crystal quality, accuracy of the cut, cleaning
- 2- quality of the vacuum during the deposition
- 3- voltage of the plasma
- 4- surface temperature of the target
- 5- ageing of the target: oxygen stoichiometry, redeposition of an amorphous YBCO phase on the target
- 6- nature of the species in the plasma
- 7- temperature of the substrate

Fires on MgO

The YBaCuO films were grown at Thomson by DC hollow cathode magnetron sputtering method using a gun made by HITEC Materials in Karlsruhe (ICM 100).

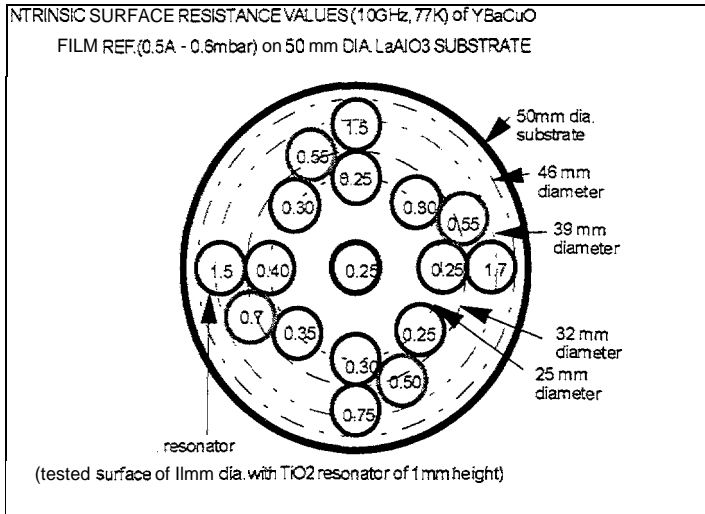
The average composition of YBaCuO films normalised to yttrium cation is $Y_1Ba_{1.92}Cu_{2.95}$. The precision of these results is better than $\pm 4 \%$ (absolute value). The film composition was determined by Rutherford Backscattering Spectrometry (RBS) in random and channeling geometry and X-ray Diffraction (XRD). The oxygen contents, directly measured by RBS, were compared to the ones deduced from the c-axis lattice parameter. We can see there is some degree of inconsistency between the oxygen content deduced from the c-axis by X-ray diffraction, RBS analyses and $T_{R=0}$ measurements. The c-axis parameter was measured by XRD, using the radiation coming from a copper X-ray tube in the Bragg-Brentano geometry. In this configuration, only lattice planes parallel to the substrate surface can be detected. It was found that all the MgO films were oriented with c-axis perpendicular to the substrate surface and no sign of extra phases was observed. RBS with a 2.2 MeV He beam in channeling geometry is a technique that provides information on the quality of the crystalline structure of the deposited films. The minimum yields, χ_{\min} , which are the ratios of the yield for perfect alignment (channeling) to that of random incidence, show that post-oxygenation has a strong influence on crystallographic properties. χ_{\min} of 5.7 % for oxygenation annealing 500 °C corresponds to the best values mentioned in the literature up to now.

The use of MgO substrates versus LaAlO₃ encounters several disadvantages such as higher a surface resistance for films, higher fragility, poor stability of the surface state especially versus moisture, poor reproducibility. In LCR, films have been deposited on 30 x 30 mm MgO substrates, 40 % exhibit a surface resistance R_s lower than 1 milliohm, 20 % exhibit a surface resistance R_s lower than 0.5 milliohm. In order to get a R_s lower 0.5 milliohm it is necessary to control very accurately the temperature of the substrate holder $T_s = 716 \text{ °C} \pm 1 \text{ °C}$. This very stringent condition explains the very poor yield of deposition on MgO. Thus deposition on lanthanum aluminate which gives more reproducible results has been developed (see below).

Fires on Lanthanum Aluminate

This study with an ICM 100 gun has permitted to define optimal deposition parameters which have then been transferred to the ICM 150 gun for larger area deposition which was readily operational. Nevertheless some different results were obtained and required a fine tuning of the set-points:

- each cationic composition does not vary independently from each other.
- thickness variation is dependent on sputtering parameter.
- surface resistance variation is dependent on sputtering parameter
- best thickness variation does not correspond to best surface resistance variation.



Surface resistance homogeneity R_s (milliOhm) on 50 mm films (LCR). We must do a compromise between excellent surface resistance and thickness homogeneity. With the following conditions, we can produce with yield of 50% YBaCuO films on LaAlO₃ (100) of which surface resistance is:

- $R_s < 0.35 \text{ m}\Omega$ over diameter 25mm
- $R_s < 0.7 \text{ m}\Omega$ over diameter 39mm

The deposition parameters on LaAlO₃ are:

- temperature of the substrate holder : 785 °C
- total pressure : 0.6 mbar
- target-substrate distance : 23mm,
- Oxygen / Argon flow = 200/ 100 seem
- plasma current /voltage = 0.5 A / 260 V

The thorough study of the variation of the composition versus the radius for films deposited from the ICM 150 under different deposition conditions (pressure, plasma power,...) shows that low R_s homogeneous films can be produced with a reasonable yield for a 40 mm diameter. All these results mean that the size of the homogeneous area is limited by the homogeneity of the plasma itself.

Among 40 films on LaAlO₃ substrates: diameter= 50 mm & $R_s < 1.1$ milli-ohms:

- 36 films exhibit a surface resistance R_s lower than 0.6 milliohm
- 32 films exhibit a surface resistance R_s lower than 0.5 milliohm
- 20 films exhibit a surface resistance R_s lower than 0.4 milliohm
- 10 films exhibit a surface resistance R_s lower than 0.35 milliohm

Films on sapphire

The deposition of YBCO films on sapphire requires buffer-layers to prevent reactions of the film with the substrate. After careful evaluation of the properties we selected CeO₂ as material for buffer-layers. We varied the properties for CeO₂ deposition on R-plane sapphire and found that the substrate temperature has to be 780° C to growth (200) - CeO₂ films. The deposition of YBCO on CeO₂/sapphire was performed with similar sputtering properties as used for film deposition on MgO. However, we used the radiation heater to heat the substrates. The film analyses indicate that good films cannot be deposited on thick CeO₂ buffer-layers. We succeeded in sputtering of (001) oriented YBCO films with transition temperatures of 86 K. However, microscopy indicates that the films have a strong tendency to get rents after some weeks of storage. In order to improve the film quality, the thickness of the buffer-layer has to be decreased to about 30 nm or less, to obtain stable films. The results from sputtering on CeO₂/sapphire and working with the infrared radiation heater indicate that a deposition of YBCO on both sides of the substrate is possible.

The results on the last films demonstrate that it is possible to get a surface resistance R_s lower than 1 milliOhm on double side CeO₂ buffered sapphire substrates. This process must be exactly controlled in order to get a reasonable yield.

4.3 TASK 4: MODELLING

Several available electromagnetic softwares have been evaluated. Only true Maxwell equations solvers have been considered since other classical softwares were known not to meet our requirements:

-2.5 D Bounds.m Element Methods:

- (a) Sonnet™
- (b) Microwave Explorer™ from Compact Software
- (c) Momentum™ from Hewlett-Packard
- (d) Antenna design from Thomson.

- 3D Finite Element method (these codes require very long CPU times and large memories):

- (e) HFSS™ from Hewlett-Packard
- (f) Thomson LCR code

A three pole microwave filter in microstrip configuration has been chosen for benchmarking. Concurrently with the modelling, we have processed and tested two filters from the same layout. Our goal is to know whether this kind of software is sufficient for our microwave planar applications, lines and filters. This work has been carried out with an exchange of technical analyses and modelling results with each of these three companies.

The evaluation considered

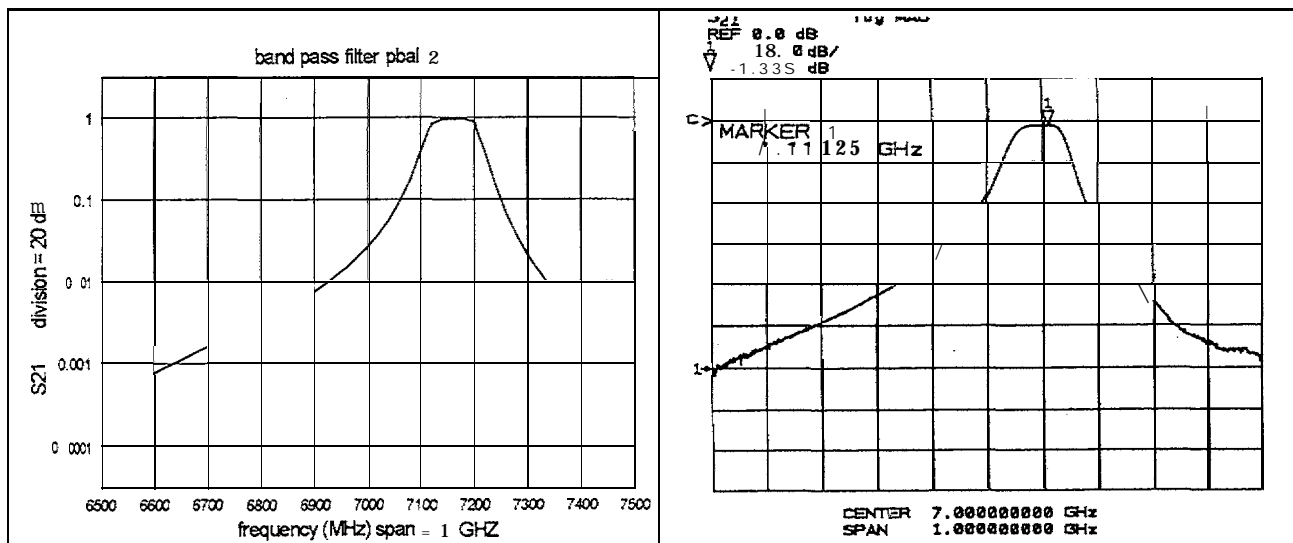
- the accuracy of the electromagnetic kernel,
- the Input and Output capabilities,
- the computing time
- the user interfaces
- the possibility to develop an optimisation feedback loop.

Two softwares (a and b) give the right transfer function with a shift in the pass-band frequencies. After several runs of modelling and discussions with the three companies, we were able to solve the frequency shift problem (a, b) but not the problem on the transfer function (c). We think that the absence of vertical walls in the software (c) is the cause of the problem. The problem of frequency shift (a, b) has been solved by an optimisation of the mesh.

The second point of our evaluation concerns the time that we have to spend to get the right transfer function. We saw in the previous paragraph that we have to optimise the mesh sections, that increases the time of analysis. All capability which allows to reduce the number of processing steps, the number of frequency steps in one analysis, and the number of mesh cells without changing the accuracy are necessary to converge quickly to the right solution. Among the input capabilities:

- Adaptive Frequency Sampling (c) gives the possibility to enter a start and a stop frequency and the software optimise the number and the value of the frequency steps to fit the transfer function.
- Edge mesh (b, c) allows to minimise the number of cells without changing the accuracy by keeping only one cell all around each resonant line.

Sonnet has been selected because of the very good agreement between modelling and a real three pole filter used for bench marking (figure below).



microstrip 3 pole filter:

Sonnet model

LCR experiment

4.4 TASK 3 & 5: PATTERNING AND PACKAGING

Several patterning methods have been evaluated paying attention to an industrial criterion - reliability-:

- 1 chemical etching
- 2 laser ablation
- 3 plasma etching
- 4 ion milling

I) Chemical etching has experienced serious problems of reproducibility:

- under-etching
- incomplete etching
- residual phases

These chemical processes are very sensitive to subtle differences in the quality of the films. LCR focused its activity on low Rs films regardless of other quality parameters with an industry oriented point of view since its role consists in a technology transfer to an industrial unit of Thomson. Finally chemical etching is up to further advice regarded as not reliable and not industrial for superconducting films.

2) Laser ablation was more exploratory. In spite of interesting results, the industrial cost seems prohibitive.

3) Plasma etching provides a clean pattern, but the superconducting properties of the films were often altered by this process so that ion milling which is more controllable has been preferred.

4) Ion milling has been tested using an ion beam produced by a special gun. On the contrary of plasma etching, the velocity and the direction of the ions can be accurately controlled. The samples must be carefully fixed with a vacuum grease in order to avoid any overheating which would destroy the superconducting properties of the films by oxygen depletion. The end of etching is visually monitored *in situ*. Then the quality of etching is controlled by *ex situ* DC resistance measurement. This process is by itself more expensive than chemical etching because it requires a more complex apparatus and a longer processing time, but it is much more reliable so that finally the global patterning cost is lower. This process has been transferred to TCM.

Metallisation of the reverse side is made with gold according to the following steps:

- 1- removing of any amount of silver paste by polishing
- 2- cleaning
- 3- ion etching
- 4- titanium layer
- 5- gold layer
- 6- electrolytic loading by gold

A special (XBD iron alloy) with matched thermal expansion is used for the enclosure.

Table 5. I : general issues for packaging

requirements	laboratory solutions	industrial final solutions\
good thermal conductivity between the parts of the case	- iridium joint - gaseous atmosphere	
thermal expansion matching		XDB steel
microwave input / output	- SMA connector - soldered semi rigid cable	SMA
dry enclosed atmosphere	over-pressure of helium gas in the case	He sealed
mechanical and electrical contacts	Au contact resistance $< 10^{-8}$ ohm.cm ²	Au
substrate fixation	clamping with spiral springs in the four comers of the case	5025 E Ablefilm
low metallic losses for high Q resonator	Copper	Copper

We realised cases for the lines, for the high Q resonator, for the filter, for the slow wave and the detector.

4.5 TASK 6: TESTING

4.5.1 TESTING OF DELIVERED FILMS

The surface resistance R_s is measured at 10 GHz and 77 K thanks to the dielectric resonator method (see figure--->). The most accurate results have been obtained with a flat rutile resonator (diameter= 7 mm, height= 1 mm). The very high permittivity of rutile (105 at 77 K) permits to probe a small area (diameter 5 mm) of the film. The choice of the single crystal with a low dielectric loss (10^{-5} at 77K) results in a high accuracy on R_s (5 μ Ohms). Thomson has delivered 106 samples :

53 films with $R_s < 0.5$ mOhm

Alenia has delivered 44 samples :

8 films with $R_s < 1$ mOhm

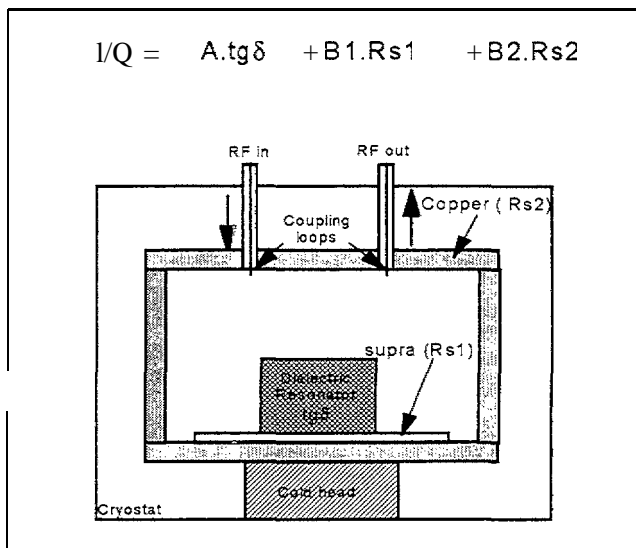
Hitec has delivered 58 films:

3 films with $R_s < 1$ mOhm

double side on buffered sapphire

CNR delivered 14 films

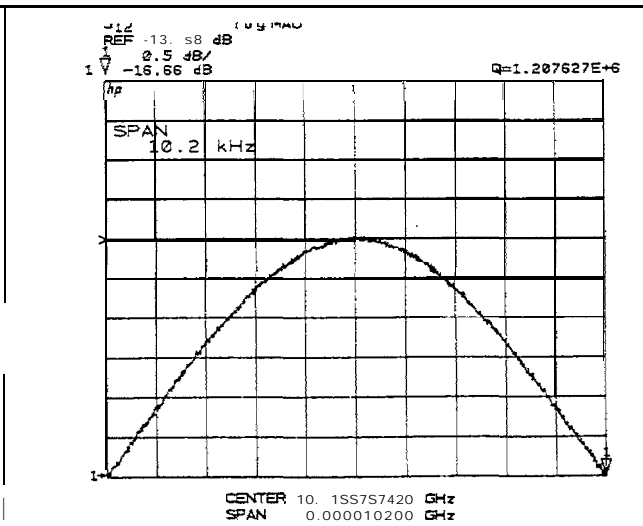
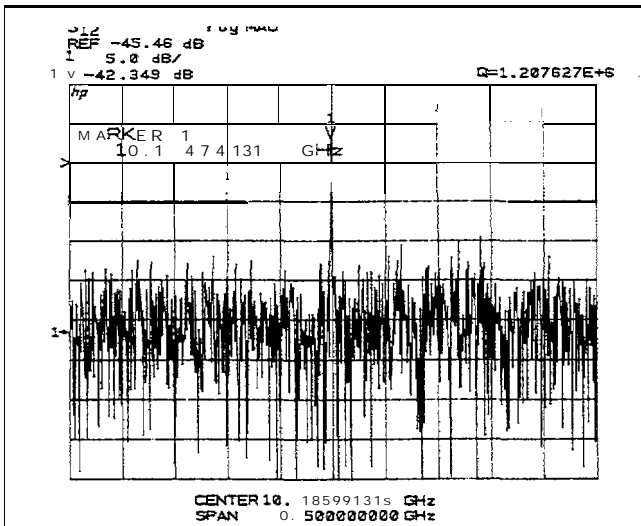
TCD delivered 16 films



Surface resistance: dielectric resonator method

4.5.2- TESTING OF 3D RESONATOR (THOMSON LCR)

The resonator measurement has been made with a network analyser HP 8510B. For cryogenics tests (77K), the resonator is dipped in a storage dewar filled of liquid nitrogen (N_2). A helium (He) gaseous over pressure in the resonator cavity avoids any water or oxygen condensation problem.



Transfer fiction (S_{21} parameter) of the 3D resonator: wide span (0.5 GHz left) and narrow span (10 kHz right)

The quality factor value is 1200000 at 10.2 GHz.

There is not other resonant mode over more than 500 MHz.

This device experiences the effect of about fifteen fast temperature cycles without decrease in performances. This result is encouraging for new superconductor oxide industrial perspectives.

The 10 GHz 3D resonator is regularly tested particularly for demonstration purposes. The quality factor is quite reproducible (between 1,100,000 and 1,300,000). Q as high as 1,900,000 have been measured in the LCR laboratory.

4.5.3- TESTING OF FILTERS (ALENIA AND LCR)

Classic design (microwave computer assisted design)

A three pole end coupled filter has been tested. The agreement between the modelling and the experiment is poor, probably because of the lack of homogeneity of the YBCO film. The central frequency F_c is shifted by 200 MHz due to the moderate accuracy of the modelling, the bandwidth B W is increased from 100 MHz to 150 MHz, due to the moderate surface resistance of the YBCO film, and a spurious rippling of about 5 dB occurs in the pass band. The conclusion is that classical microwave CAD is inadequate for HTSC filter modelling, exact solution of the Maxwell's equations through a 2.5 D software is necessary.

Advanced desire (2.5 D boundary element method)

Testing filters is an important point for evaluating the accuracy of 2.5 D modelling and of technology (see 4.3.). The accuracy on the central frequency F_c depends on the size of the mesh. This explains a 60 MHz shift. In order to get a better accuracy, we can either refine the mesh within a reasonable computing time or establish a two run process with rule of the thumb between modelling and experiment. The reproducibility of these processes seems sufficient to eliminate any tuning elements even for very selective filters. Concerning the shape of the transfer function, even for a very narrow bandwidth ($BW < 1\%$) in spite of the very low couplings required, the accuracy of the modelling is excellent:

-3 dB bandwidth= 95 MHz versus 90 MHz

- insertion loss = 1.3 dB (and 0.7 dB for the best filter) versus 0.6 dB

- rippling < 0.1 dB

- out of band rejection > 60 dB

Moreover even the defects of the transfer function (slightly non symmetrical with zeroes out of the band) are accurately modelled by Sonnet. These defects correspond to interference between very weak signals (< -60 dB). These results are possible because Sonnet takes into account the effect of the enclosure around the circuit, Several filters patterned with the same mask exhibit the same transfer function.

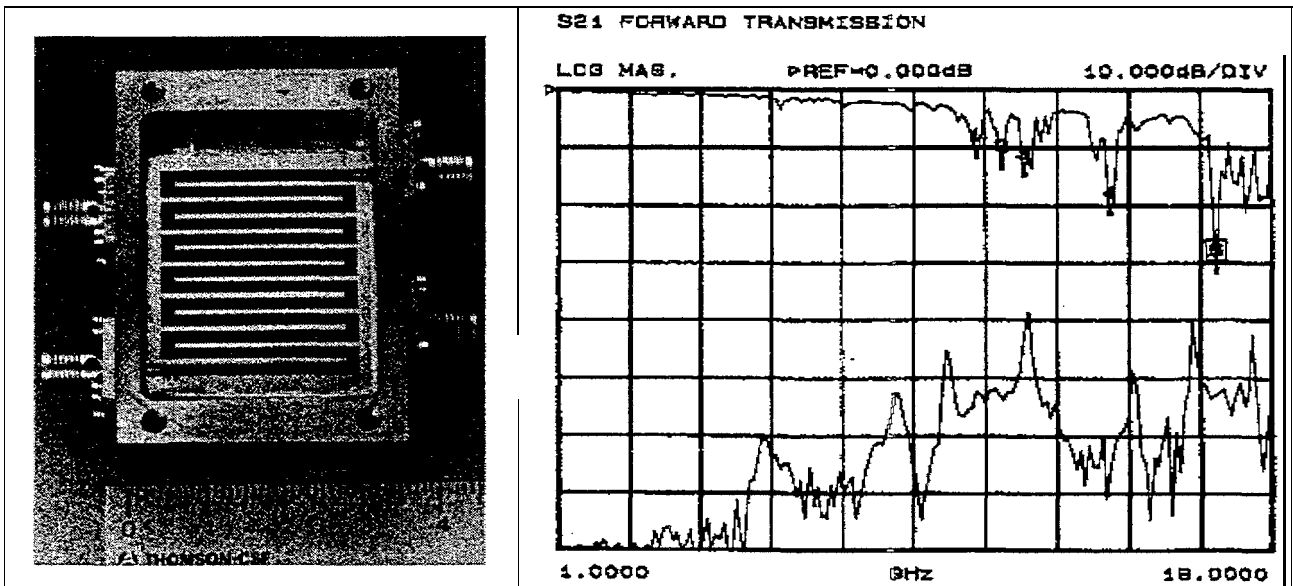
4.5.4: TESTING OF TAPPED DELAY LINE (THOMSON LCR & TCM)

Measurements made in TCM stress two directions :

① the integration of meander line or other microwave circuits in a more industrial way is possible :

- use of TCM procedures for CPW (coplanar waveguide) line packaging,
- use of a conductive adhesive film to maintain the substrate and to ensure ground contact,
- use of automatic or semi-automatic wire bonding means.

② rejection of parasitic resonances above 11 GHz



Tapped delay line : open box (left),

transfer function (right)

Nevertheless, integration of microwave circuits patterned on YBCO (e.g. filters) has been successfully performed and no spurious mode occurs up to 11 GHz. The insertion loss is very good, typically less than 3 dB at 10 GHz and 1.6 dB for the best line versus 7 dB for an Aluminium line. This corresponds to a corrected surface resistance:

$$R_s = 1 \text{ m}\Omega.$$

4.5.5 TESTING OF DETECTORS AND SLOW WAVES (TCD)

The noise performance of a detector ultimately determines its usefulness and it is the general goal of detector design to maximise this signal to noise ratio. The output noise voltage of a receiver limits the maximum detectable power, quantified by the NEP (noise equivalent power) value. In general, the lower the NEP, the more sensitive the receiver, although the ultimate limitation is imposed by the temperature and that of any background radiation.

High temperature superconducting detectors have NEP values in the range $10^{-12} - 10^{-14} \text{ W/Hz}$.

4.6 Initially Planned Vs Actually Accomplished Objectives

task	subtask	partner	objective	initial	updated	achieved	
1. targets	1.1. target preparation	Hitec	density	>95 %	90 %	86% / 95% (HIP)	
		Hitec	size	> 100 mm		= 100 mm	
		Hitec	thermal contact		$T_{\text{target}} < 400^{\circ}\text{C}$	copper clad	
	1.2. target analyses	Th LCR	spurious phases	< 5740		< 10/0	
	1.3. alternative phase	CNR	versus YBCO	lower R_s		no	
2. films	2.1. low R_s	Th LCR	surface resistance	100 $\mu\Omega$	200 $\mu\Omega$	190 $\mu\Omega$ (3 %)	
	2.2. reproducible	Alenia	yield $R_s < 500 \mu\Omega$	> 10 %		> 50 0/0 *(2.1)	
	2.3. <i>large areas</i>	Hitec	diameter	> 50 mm		=40 mm*(2.1)	
			Hitec	alternative substrates			sapphire
	2.4 very thin films	TCD	thickness	<100 nm		=20 nm*(2.1)	
	2.5. analyses	CNR	1 phase epitaxy	> 90 %		>99.95 %	
3. etching	3.1 chemical	Th LCR	1 μm resolution / no damage	+ no residue		<i>not reliable</i>	
	3.2. plasma	Alenia	1 μm resolution/no damage	+ no residue		<i>not reliable</i>	
	3.3 industrial	Thomson	1 μm resolution{ no damage	+ no residue		ion milling* (3.1)	
4. modelling	4.1 lines	Th LCR	Zc, attenuation			2.5 D model	
	4.2. 3D resonator	Th LCR	ultimate Q			2 D model	
	4.3. filters	Alenia	fc, BW, IL, rippling, rejection			2.5 D model*	
	4.4. <i>slow wave</i>	TCD	slowing factor			analytical model	
	4.5. detectors	TCD	sensitivity			RSJ model	
5. packaging	5.1 lines	Thomson	coplanar grounding			wire bonding	
	5.2. 3D resonator	Th LCR	high Q	>1000000		1300000	
		Th LCR	vibration sensitivity			lab demo	
	5.3.filter	Alenia	spurious modes	> 2 f_c		@ 2 f_c	
	5.4.slow wave/detector	TCD	strain control	no break		lab demo	
6. testing	6.1. films	Th LCR	surface resistance	resolution < 10 $\mu\Omega$		190\pm10 $\mu\Omega$ @10GHz	
	6.2. resonant lines	Alenia	surface resistance	resolution < 1 $\mu\Omega$		106\pm1$\mu\Omega$* @ 6 GHz	
	6.3 3D resonator	Th LCR	quality factor Q	>300000	>1000000	= 1300000	
	6.4. filters	Alenia	bandwidth	< 0.5%		= 0.9 % *	
		Alenia	insertion loss	< 1dB		= 0.7 dB *	
		Alenia	rippling	< 0.5 dB		= 0.1 dB *	
		Alenia	rejection	60 dB		> 60 dB *	
		Alenia	filter bank	>5 channels		?	
	6.5.delay line	Thomson	delay	> 1.6 ns		3.9 ns	
		Thomson	insertion loss			1.6 dB @ 10 GHz	
		Thomson	spurious modes	> 10 GHz		> 11 GHz	
		Thomson	tap number	> 16		= 3	
	6.6. slow wave	TCD	slowing factor	> 5		?	
	6.7 detectors	TCD	sensitivity	> semicond.		?	
bold letters = excellent results <i>italic = poor or no result</i> normal = passable						* = Th LCR	

5 EXPLOITATION PLAN

5.1 PRACTICAL APPLICATIONS OF PROJECT RESULTS

This BRITE project was essentially a material oriented project including microwave devices in order to demonstrate the potential applications of superconducting materials.

Concerning the material aspect the main result is the mastering of 2 inch YBCO film deposition with a surface resistance R_s lower than 0.5 milliohm. This result was necessary for the achievement of our objectives and can also lead to many other non-stated applications such as medical imagery by low field magnetic resonance (low noise HF antenna).

Concerning the demonstrator aspect, the first most dramatic result concerns the **Very High Q 3D Resonator**.

At the time when the proposal was first written down, no result in the world have been published in this field. Thus the project was very innovating. During the negotiation of the final proposal, Dupont (USA) published dramatic results in this field, compelling the consortium to update the specifications from $Q > 300,000$ to $Q > 1,000,000$. Now this ambitious objective is not only fulfilled, but passed beyond, and better results are still expected for the near future. The typical applications of these resonators are:

- microwave filters with a very high selectivity and a low insertion loss (BRITE project DiHiMiCo, start: 0 1/01/96)
- microwave oscillators with a very low phase noise:
 - radars with improved contrast by Doppler processing (air traffic control, wind shear detection, low RCS targets etc...)
 - very low jitter clock for high speed digital signal processing (Analog to **Digital** Converters - ADC - in the GHz range)

The need for low phase noise is real:

- it is necessary to improve - within the next five years - the nowadays specifications by 2 or 3 orders of magnitude.
- the superconducting resonator solution has been proposed to workgroups involved in radar and ADC future and has met a big interest, even if some potential users are reluctant to use cryogeny.
- no alternative solutions to these problems of jitter and phase noise is actually known.

The second result on delay lines demonstrates the feasibility of moderate delays with the possibility of integrating signal processing for: correlators, stop-band filter bank, envelop detectors, instantaneous digital frequency meters.

The third result concerning filterbanks still requires studies for the branching manifold in order to make complex multiplexer (a single filter is not interesting for the system designer: many problems, little advantage):

- filters for **cellular phone base station** (ACTS proposal) :
 - higher a selectivity,
 - lower insertion loss,
 - more numerous channels

An intermediate structure between 2D planar design and 3D resonator is now under consideration to supply outstanding performances especially versus power handling ability. This is now a very active field in the USA

- filters telecommunication satellites (up) and satellite telecommunications (down):
 - either lower weight, smaller volume for equal performances
 - or very improved performances: very low insertion loss with 3D resonator filters

The integration of high T_c superconducting devices in satellite systems is a long way passing through space qualification and the development of very reliable cryocoolers.

5.2 IMMEDIATE SPIN-OFF

This kind of components is interesting only for integration within analogue or digital signal processors such as mixers for correlators, diodes for envelop detectors, comparators for instantaneous digital frequency meters. The development of these detectors is subordinated to the development of advanced devices based on tap delay lines.

The involvement of TCM, RCM, Airsys, Alenia Spazio, GEC Marconi, TCC . . . confirm the interest of superconducting devices for oscillators and lines but also for filter banks: either band-stop or band pass filters in order to reject jammers or to avoid any interference which are becoming very troublesome. This purpose requires complex systems. The feasibility of the basic bricks of these systems have been demonstrated in this project. The design of new systems associated with the definition of new standards a long way...

Hi I? XC-Materials is producing targets for deposition of superconducting films and also offers sputtering equipment. So the tasks of the project fit very well to the delivery program of our company.

TARGETS: The improvement of the target density by near net shape isostatic pressing and sintering was welcome by our customers. Also the bonding of the targets into copper rings.

The turnover in these special targets increased and is about 60,000 ECU/pa.

SPUTTERING HEADS: We assume that the request for sputtering larger areas is at least partially triggered by the knowledge of our project for deposition of films for micro-wave applications. We received orders for ICMS in the range of 150,000 ECU.

EQUIPMENT: The turnover for our devices used in sputtering superconducting films as substrate heater, power supplies, complete sputtering systems is in the range of 250,000 ECU.

RATING: The project work performed in the last 3 years led to the following:

- optimization/improvement of our products in the branch of HTSC

- increased know-how
- additional product branch
- additional turnover

We consider our financial investment in this project as well placed with a good chance to amortise within the next 3 years.

Education of students in the microwave and the superconductor fields

- ongoing work to finish a Ph.D. thesis for Ms. Miriam McConnell in TCD
- an M. SC. thesis for Mr. James Egan in TCD
- stage T. Lebouar X 1993 in LCR
- stage S. Barrault X 1993 in LCR
- stages N. Voisin 1993, 1995 in LCR
- stage B. Janossy in CNR

5.3 ESTIMATED TIME SCALE FOR DEVELOPMENT

After the completion of the project at O 1/03/1996, the estimated time scale for development:

- Raw films :

The commercialisation of 2 inch YBCO films is not immediately available on merchant market because the best results from Hitec occurred very lately so that it is now too early for a real commercialisation. Thomson (which is not a film supplier) could deliver few samples on request. The opportunity of a specific effort on this subject is questionable since during the completion of the project, the Munich Technical University (Pr Kinder) has developed a very powerful co-evaporation system which permits to deposit films on very large areas (up to S⁴). These films are commercially available at a very competitive price and are now under evaluation by Thomson.

- Filter bank :

3 years development -spatial specification-

Alenia plans to develop filters associated with Alenia Spazio.

This kind of project could be funded by ESA.

- Filter bank :+

3 years development -telecommunications specification-

Thomson associated with GEC Marconi, Birmingham university and Wuppertal university have submitted last year an ACTS proposal which was successful but underwent problems due to the withdrawal of Ericsson who was the end user inside the consortium. Ericsson has been replaced by Thomson CSF Communications (TCC) which is a specialist of military communications now evolving towards civilian communications.

This project concerns essentially filters for UMTS base stations for macrocells (along the motorways) and hypercells (for low population density areas) and components for satellite communications.

The project is still under negotiation

- 3D resonator :

3 years development for low noise oscillators

Thomson LCR associated with TCM and the Limoges university (IRCOM) has submitted a 2 year proposal to the French DGA / DRET (Direction des Recherches et Etudes Techniques).

This project could start next year with Airsys as an end-user.

- Tapped delay line :

3 year development

Thomson-CSF Microelectronics with Thomson Radar Contre Mesures as an end user have been directly involved in the project. TRCM has now to define precise specifications for devices such as correlators, stop-band filter bank, envelop detectors and instantaneous digital frequency meters. The development of stop band filter banks is driven by the supply of such devices by Superconductor Technology Inc. to US Air Force Wright Patterson.

Not yet a precise deadline.

- Slow wave delay lines :

Demonstration yet to be done.

- Detectors:

Moderately convincing demonstration.

6 COLLABORATION SOUGHT

Collaborations are possible in several fields.

-telecommunications: components for cellular phone base stations.

==> We are seeking for a collaboration with a phone operator. Several contacts are being studied, but the choice is not yet defined. This collaboration would be performed within the now running ACTS project SUCOMS.

- filters for base stations are thoroughly investigated in the USA


- the filter structure designed within SURF is a first step towards base station filters:
the frequency is 2 GHz instead of 10 GHz

the microwave power is tens of watts instead of milliwatts

- the power handling capability is yet to be tested but is expected to meet the requirements.

- atomic clock / frequency standards

==> We are seeking for a frequency standard manufacturer. We have now a collaboration with NPL (National Physics Laboratory London) within BRITE project DiHiMiCo in order to study an oscillator for an atomic clock. A very low phase noise near the carrier is required. A cryogenic system is required to meet this objective. Our high Q 3D resonator can be a solution to this problem. Superconductors could be used in more sophisticated resonators in order to improve temperature stability

 astrophysics applications

==> We are seeking for a collaboration with an astrophysics laboratory in order to make a Perot-Fabry spectrometer.

The university of Nancy FR has measured our films in the sub-millimeter range with very interesting results (YBCO is still much better than gold in this frequency range). Such a spectrometer is intended to analyse atmospheric species or inter-galactic molecules

- Magnetic Resonance Medical Imaging:

==> We are seeking for a collaboration with a MRI manufacturer in order to develop YBCO antennae for low field magnetic resonance imagery. The interest in high Tc superconductor antennae in order to improve the signal to noise ratio. The "Institut d' Electronique Fondamentale" IEF Orsay FR has tested our films at 100 MHz by making an antenna consisting in a resonant lumped LC circuit the Q of which is 7200. The low field resonant frequency is typically 4 MHz. The Rs of YBCO at 4 MHz is supposed to be 3 millions times lower than that of copper. The circuit design is accessible to the technology developed within SURF.

We would like to test this antenna in a MRI system.

7 REFERENCES

7.1 PUBLICATIONS RESULTING DIRECTLY FROM THE PROJECT

- ESA / ESTEC (Noordwijk NL 27-28 April 1993) ESA WPP-052 :
Workshop on: Space Applications of High Temperature Superconductors
"Advances in Application of High Tc Superconductors to Microwaves"
Y. Lemaître, L.M. Mercandalli, D. Mansart, B. Dessertenne, B. Marcilhac, J.C. Mage page 75
"Activities on High Temperature superconductive materials for Italian Space Programs" page 163
F. Graziani, N. Sparvieri et al.
- 8èmes Journées Nationales Micro-ondes 3C-2 (Brest FR 12-14 May 1993)
"Supraconducteurs et hyperfréquences : état de l'art" J. C. Mage
- Club CRIN (Aussois FR 12-14 Mai 1993):
- Applications des supraconducteurs a haute température critique:
"Composants passifs: état de l'art international et perspectives" P. Hartemann
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B. Marcilhac, J. C. Mage et Y. Lemaître, L.M. Mercandalli, D. Mansart, B. Dessertenne,
- 12ème colloque Optique Hertzienne et Diélectrique (Paris F 1-3 September 1993):
"Supraconducteur YBaCuO/LaAlO₃ : relation entre la croissance cristalline et l'impédance de surface"
Y. Lemaître, L.M. Mercandalli, D. Mansart, B. Dessertenne, B. Marcilhac, J. C. Mage
- European Microwave Conference (Madrid SP 6-9 September 1993) page 185:
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Y. Lemaître, L.M. Mercandalli, D. Mansart, B. Dessertenne, B. Marcilhac, J.C. Mage
- SITEF (Toulouse FR 19-23 October 1993):
"Supraconducteurs à haute temperature critique: Application aux composants passifs pour hyperfréquences"
J. C. Mage, Y. Lemaître, L.M. Mercandalli, D. Mansart, B. Dessertenne, B. Marcilhac
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- ISIS 3 International Summit on Industrial Superconductivity (Aylesbury UK 10-19 May 1994):
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- M'S HTSC (Grenoble F 5-9 July 1994) WD-PS 159 :
- Physics C 235-240 (1994) 643-644
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- M'S HTSC (Grenoble F 5-9 July 1994):
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- Journal de Physique III France No 4 (July 1994) page 1'285:
"Advances in the application of high Tc superconductors to microwave devices for analog signal processing"
J. C. Mage, Y. Lemaître, L.M. Mercandalli, D. Mansart, B. Dessertenne, B. Marcilhac
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"Superconducting properties of polycrystalline YBCO prepared by pyrolytic process"
"Growth and magnetic characterization of YBCO films"
N. Sparvieri et al.
- Physics C 220, 219-216 1994
"Twinning of YBCO thin films on different substrates and modification by irradiation"
T. Steinbom, W. Blau, M. Mc Connell et al.
- Solid State Communications, Vol. 89, N° 8, pp. 705-708, 1994
"Magnetic field dependence of Josephson photoresponse in HTSC thin films"
Gi. Schnieder, W. Blau et al.
- Applied Physics Letters Vol. 63 N° 20, pp 2827-2829, 15 November 1993
"Enhancement of Josephson photoresponse of granular high Tc superconductors thin films by deoxygenation"
Gi. Schnieder, W. Blau et al.
- J. C. Mage, Thomson, has given an invited lecture at EUCAS 95, 3-6 July 1995 Edimburg UK:
"Niches and Traps for Applications of High Tc Superconductors in the Microwave field"
- in LCR, high Q resonators and filters have been operated for demonstration for several visitors:
Mr Withers from Conductus USA, Ms Ceremuga university of North Queensland, Australia, Mr Bensasson E. U., Mr Martin E. U., DRA Malvern, French CNRS directors, Swedish ministry of scientific research.

7.2 PUBLICATIONS AND THESES RESULTING INDIRECTLY FROM THE PROJECT

Several theses have been or will be maintained in connection with this project.

For these theses, some of the films have been supplied by Thomson-CSF / LCR:

Louis-Anne de Vaultier thèse de doctorat de l'université de PARIS 6, 12 juin 1995:

"Transmission dans l'infrarouge lointain de couches minces YBaCuO, détermination de la profondeur de pénétration du champ électromagnétique"

Abstract: this work consists in building an experimental set-up to measure the transmission of electromagnetic waves through YBaCuO thin films from 5 to 250 K. The study of the superconductive transition leads us to establish a quality criterion for the samples. We correlate the film quality with the measurement of the X ray "rocking curve" and the measurement of the microwave surface resistance. We show that the apparent width of the transition is probably due to the superconducting gap which seems to exhibit a strong anisotropy. We show that the study of the low temperature data yields to the absolute value of the electromagnetic penetration depth. Finally we establish within a weak link model the extrinsic nature of the quadratic dependence versus the temperature for the penetration depth in films. We also show that the intrinsic dependence is linear. This implies that there exist low temperature excitations inconsistent with an isotropic gap.

Masato Tazawa, thèse de doctorat de l'université Henri Poincaré NANCY 1, 10 novembre 1995:

"Nouvelles possibilités d'étude d'un milieu très réfléchissant dans l'infrarouge lointain, application aux nouveaux supraconducteurs à haute température critique"

Abstract: This work started four years after the discovery of high T_c superconductors, when it has been possible to get single crystal plates thin enough and stable enough to permit transmission studies in the 10-40 cm⁻¹ spectral range of the far infrared. They were thin layers 200 Å to 500 Å thick deposited on MgO single crystals. It has been necessary first to characterise MgO as accurately as possible. Then we had to write programs to calculate transmission, reflectivity, absorption, etc. of stratified samples. The last and best results have been obtained with a grid spectrometer and a cryostat especially made for sub-millimeter waves and a new interferometric method.

We have been able to get the free carrier plasma frequency from 4 K to 300 K and the collision frequency of quasi-particles. It has been shown that the number of quasi-particles is given by a $(T/T_c)^{1.5}$ law rather than a more conventional $(T/T_c)^4$ law. Losses in the microwaves at low temperature are due mainly to the small number of residual quasi-particles, whereas in the far infrared they are mostly ascribed to the low frequency wing of the infrared oscillators that we have introduced in the model.

Xavier Lopez Garcia GPS Paris VII university 20 décembre 1995

"Corrélation entre la composition et les propriétés physiques des couches minces YBaCuO: étude des mécanismes d'oxygénation"

The thesis presents data relating to the correlation between the composition and the physical properties of YBCO films. Measurements have been made both in situ and ex situ, using nuclear reactions techniques and ¹⁸O tracers.

It has been possible to measure the oxygen stoichiometry of films subjected to different thermal treatments. Three important new facts emerge:

- 1) the presence of carbon impurities has a marked effect on the rate of oxygen exchange
- 2) exchange during oxidation and reduction is asymmetric. It is faster in the case of oxidation in accordance with other works on resistivity measurements
- 3) the diffusivity of oxygen in these c-axis oriented films is orders of magnitude higher than expected from data obtained from single crystals.

Sarfati IEF Paris XI 22 janvier 1996:

"Nouvelles générations d'antennes pour l'imagerie par résonance magnétique"

Myriam Mc Connell, Trinity College Dublin to be maintained in November 1996

"HTS films for microwave applications"

James Egan Trinity College Dublin

The dissemination of films and characterisations has led to several publications:

- *"Residual losses of superconducting thin films of YBaCuO in the far infrared and microwaves applications"* (films and microwave characterisation supplied by Thomson-CSF/LCR)

A. Hadni, X. Gerbaux, H. M. Cudraz, M. Tazawa, J. C. Mage, B. Marcilhac, L. Mercandalli, D. Mansart
Physics C 245 (1995) pp 219-230

- *"Microwaves Properties of MgO Single Crystals Computed from the IR and Measured by a Resonator Technique"*

A. Hadni, X. Gerbaux, J. C. Mage, B. Marcilhac
Infrared and Millimeter Waves Journal (July 1994):

- *"YBaCuO films epitaxially grown on MgO, LaAlO₃, SrLaAlO₄ and Al₂O₃ substrates, structural and superconducting properties in correlation with the microwave surface resistance and the far infrared transmittance"*.

(acknowledgement to Thomson-CSF / LCR for microwave measurements)

C. Le Paven-Thivet, M. Guilloux-Viry, J. Padiou, A. Pernn, M. Sergent, L.A. de Vaultier, N. Bontemps
Physics C 244 (1995) pp 231-242: