SYNTHESIS REPORT

FOR PUBLICATION

CONTRACT NO . . . . . : BRE2-CT92-0298

PROJECT NO..... : BE 5786

TITLE . . . . . : **IMPROVED TARGET MATERIAL FOR HIGH FIELD SPUTTER DEPOSITION OF HIGH QUALITY ITO FILMS WITH LOW RESISTIWTY AND HIGH OPTICAL TRANSMISSION**

PROJECT
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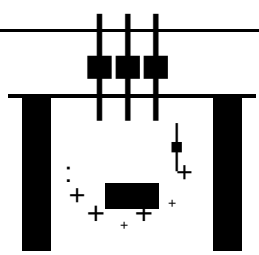
SAINT GOBAIN RECHERCHE

REFERENCE PERIOD FROM 01/01/93 TO 31/12/95

STARTING DATE : 01/01/93 DURATION : 36 MONTHS



PROJECT FUNDED BY THE EUROPEAN COMMUNITY
UNDER THE BRITE/EURAM PROGRAMME

DATE : February 29, 1996

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Trappes, February 29, 1996

IMPROVED TARGET MATERIAL FOR HIGH FIELD SPUTTER DEPOSITION OF HIGH QUALITY ITO FILMS WITH LOW RESISTIVITY AND HIGH OPTICAL TRANSMISSION

BRITE EURAM CONTRACT Nr BRE2-CT92~0298 1

SYNTHESIS

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KEY-WQRD : EEC - ERITE EURAM - TH!N FILM - PROCESSING (METALS) - PROCESSING [CERAMICS& GLASS) - SURFACE TREATMENT TECHNOLOGY - RECYCLING

ABSTRACT

For the manufacturing of flat panel display and electrochromic devices, an high density Indium Tin Oxide (ITO) material and an improved sputtering system have been developed then tested at the preindustrial scale. These material and equipment enable to increase the utilization rate of the ITO sputtering targets without deteriorating the performances of the ITO thin films. The origin of black nodules that grow on the target surface during a sputtering operation and that lead to premature shuts down of the sputtering process has, also, been elucidated.

The potential benefits that can be expected from this work are a reduction of the production costs of the flat panel display systems.

The problem for the recycling of the ITO targets also been studied and two recycling routes (for new scraps and for old scraps) have been proposed. These two recycling routes will enable a better management of the iridium worldwide resources.

1.- INTRODUCTION

For the manufacturing of flat panel displays, electro-chromic glazing and other large area coating files, electrical conducting layers with high degree of optical transmission are usually required. Thin films of Iridium Tin Oxide (ITO) having good optical and electrical properties, different deposition techniques have been developed to substrate with a thin film transparent and conducting ITO layer.

One of the them (semi reactive sputtering of an ITO target under a plasma) enables the deposition of high quality thin films but suffers from a major problem related to a reduced possibility of utilization of the ITO target in a sputtering machine. One estimates, indeed, that, due to the non uniformity of the plasma ring in the sputtering machine, only 20 O/! of the weight of a target weight can be transferred, without alteration of the thin film properties, on a substrate with the classical ITO targets and sputtering technologies. This results in frequent interruptions of sputtering process, for target replacement, and generates important quality of partially used ITO target.

The industrial experience also shows that "black nodules" grow on the surface of ITO target during the sputtering operations and that their occurrence modifies deposition conditions and the characteristics of the ITO thin films. The common practice is to stop the deposition **process to change** or clear the ITO target but, again, this operation has negative impact on the manufacturing costs for the flat panel display systems.

The objectives of the research program carried out by METALEUROP RECHERCHE, DEGUSSA AC, LEYBOLD AG and St GOBAIN RECHERCHE with the financial help of the European Corn mu n i ty, is therefore to ***develop artd test improved ITO mqteria!s and sputtering techniques for the manufacturing of high qua!ity I TO thin films.*** More precisely the quantitative objectives of this research program are :

- To develop and test a high density ITO material able to produce during an extended period of time, ITO thin films with a transmittance higher than 98 O/O; a resistivity **lower than 150~ohm.cm and a defect density lower than** 100 / cm^2.

- To **develop and test targets and magnetron designs able to increase the target utilization rate from** 20 0/0 to 30 0/0 without alteration of the thin film properties.

- To develop and test different procedures to recover valuable iridium from partially used ITO targets,

Benefits in term of :

- ➥ **Improvement of the performances of the flat panel display systems ;**
- ➥ **Reduction of flat panel display production costs ;**
- ➥ **Better management of worldwide iridium resources ;**

are expected from this program,

2.- TECHNICAL DESCRIPTION

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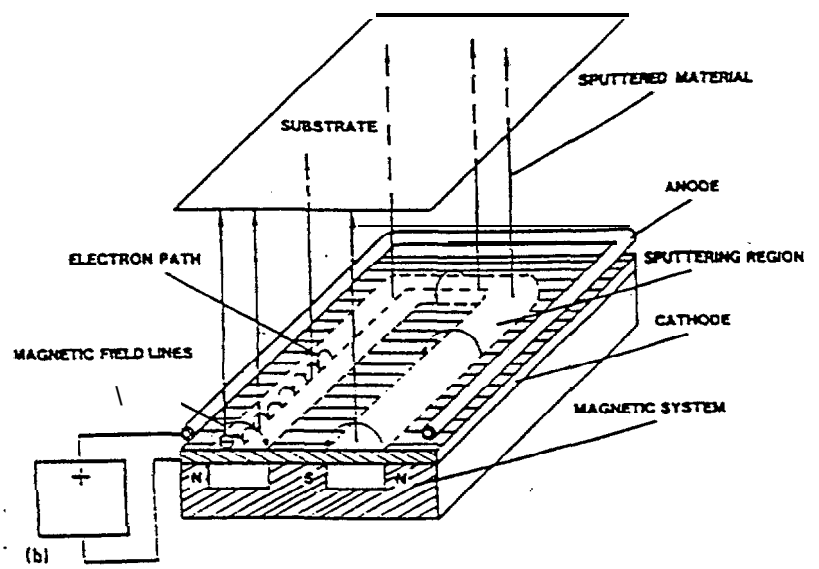
In the sputtering technic, the deposition of ITO coatings for the flat panel display application is performed on vertical in-line systems where rectangular magnetron cathodes are used with a fixed location of the magnets, The position of the permanent magnets defines the plasma ring (see fig. 1) at the surface of the ITO target and therefore the erosion zone where an efficient putter rate is generated. The arrangement is optimized for an uniform deposition on the substrate and for a low sputter voltage to guarantee good thin film properties especially in term of conductivity.

FIGURE 1 : PRINCIPLE OF THE SPUTTERING DEPOSITION PROCESS



The current procedures for ITO target production consists in mixing an In_2O_3 powder coming from an iridium producer with SnO_2 then carrying out a densification step by different methods such as the Sintering, the Hot Pressing {HP) and the Hot Isostatic Pressing techniques. These densification methods enable the processing of a ceramic material ready to be shaped in an ITO target with a density ranging from 80 O/. to 95 % of the ITO theoretical density (7,1 g/cm^3). The target density is a quality criterium since it is thought that a high density target is less prone to the nodules formation problem and has a longer life time than a low density target.

B) DE.C.RLIZLOMO.FIHEI.E.CHNICALULO.G RAM

The research program defined by METALEUROP RECHERCHE, DEGUSSA AG, LEYBOLD AC and St GOBAIN REICH ERCHE, for the development of an improved target material, included :

- The ***development of high purity*** {41V] /n_2O_3 ***and*** ***In_2O_3/ SnO_2 powders*** for ITO target application.

- The ***development of improved cfensification techniques*** for the manufacturing of I TO targets with density higher than 95 O/..

- The ***development of improved sputtering sysfems*** to increase of the I TO target utilization rate form 20 $^0/_0$ to 30 O/..

- Some academic studies for a better comprehension of the ***nodule formation prob!em*** which prematurely reduces the ITO target utilization time.

- The ***development of*** **cost** ***effective methods to recover*** valuable iridium from partially used ITO targets.

- The testing of the differ~nt products, methods and process defined, in a first step at lab scale, within the framework of a ***demonstration run*** carried out at the preindustrial scale.

3.- RESULTS

A) D_EYELO_WEiX_T_O_F_ HI_C.H_MJRIIY_lN 2&4N D-lN.$_2$(1$_3$/_S.MQ$_2$-FQWDERSAND D.ENS.LELCAJ.LO.NI.EC H.N.LQ.U.ES.F.O.R.IHE.ERO.CES.S.I lW_O.F_tUm-DINSl.LY_TARGtET

The procedure used for the development of an In_20_3 and In_20_3/SnO_z powders that better fit DE GUSSA'S requirements for the processing of high density ITO targets was a procedure in 7 steps which included :

- The characterization of competitive In_20_3 and lnz O~/Sn02 powders.
- The definition of a "wet chemical" route for the *martufacfuring of an iridium hydroxjde.* Within this framework, the influence of the nature of the chemical reagents and calcination conditions on the morphology of the iridium hydroxide used for the processing ot the In_20_3 powder has been studied
- The definition of a processing route for the *production of a mixed* $fn_20_3/Sn0_2$ *powder.* A particular effort has been made to improve of the tin distribution in the iridium matrix.
- The *selection of the most promising po walers and densification technology* on the basis of the mechanical properties of ceramic ingots densified by 3 dertsification techniques (Sintering , Hot Pressing and Hot Isostatic Pressing).
- The *confirmation of the previous choices* on the basis of the sputtering trials carried out on lab scale targets (PK75 targets, 1,5 kg).
- The *upscaling* of the In_20_3 processing routes and densification techniques from the lab scale (PK75 targets, 1,5 kg) to the pilot scale (PK500 targets, 8 kg).

2- The *manufacturing* of three PK500 targets for the demonstration run.

This procedure led to the definition of the two processing routes for the manufacturing of a In_20_3 powder (S1 200) and a In_20~/$Sn0_2$ powder (Wlvll 200] for target application and enabled the selection of the Hot Isostatic Pressure (HIP) technic as the most promising route to produce targets with a density higher than 95 O/..

FIGURE 2:

PERFORMANCES AND CHARACTERISTICS OF **S1 200** AND **WM1 200** POWDERS FOR ITO TARGET APPLICATION

POWDER	POWDER COMPOSITION	POWDER PROPERTIES			TARGET PROPERTIES	
		BET	TGA	HIP density	Bending St	Elect Res.
		(μm)	(m^2/g)	(%)	(Mpa)	($\mu.\Omega.cm$)
S1200	In_2O_3	2,7	1,3	0,25	98,6	87,3	430
S1200	In_2O_3	2,4	1,2	0,25	98,4	-	1 255
WM1 200	In_2O_3 - SnO_2	2,8	1,4	0,20	98,6	105,0	430

More precisely, it has been demonstrated that :

- **A good flexibility in term of morphology, grain size distribution and surface specific of the In_2O_3 powder can be achieved by changing the powder process conditions, mainly the $In(OH)_3$ precipitating conditions and calcination conditions.**

- **The performances of an ITO target are strongly dependent on the In_2O_3 powder and the densification technic. More precisely, the characteristics asked for a In_2O_3 for sintering application (high BET, multimodal distribution) are different from those asked for an HIP application (medium BET, low TGA) or in other words, a In_2O_3 powder that fits a target manufacturer for a .Sintering application may not fit the same manufacturer for a HIP application.**

- **The Sintering or HP techniques do not enable the processing of high (> 95 0/~) density targets. The maximum density that can be achieved with the sintering or the HP techniques, do not pass 90 o/~ of the theoretical density whereas the HIP process enables the processing of ITO target whose density (97 °/0) is higher that the goal density (95 °/0) defined at the beginning of the program.**

All these properties were, firstly checked out at lab scale (100 g ingots) then al pilot scale (1,5 kg targets) before being upscaled to the preindustrial scale (8 kg targets).

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A specific experiment was set up, in collaboration with the University of Darmstadt, to investigate the nodules growth problem, [t consisted in observing the progress of the nodule formation and growth with a video camera, aimed at the target surface, during a sputtering operation.

FIGURE 3: PICTURE OF AN ERODED I T OTARGET COVERED WITH NODULES (X 0,8)

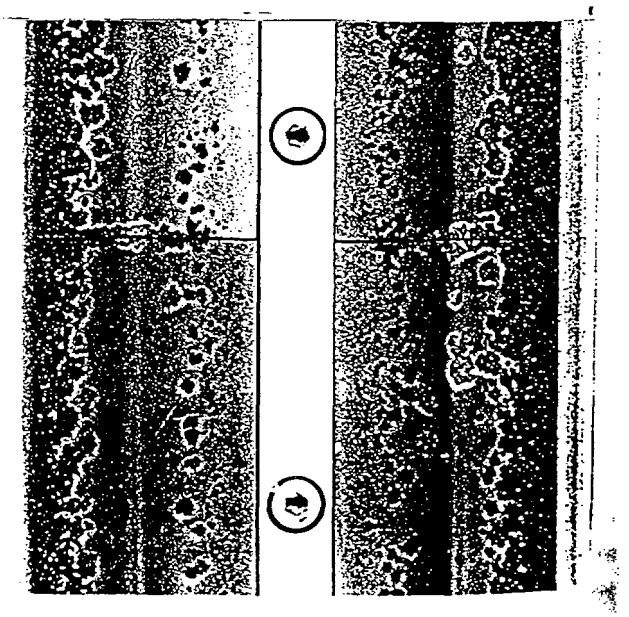
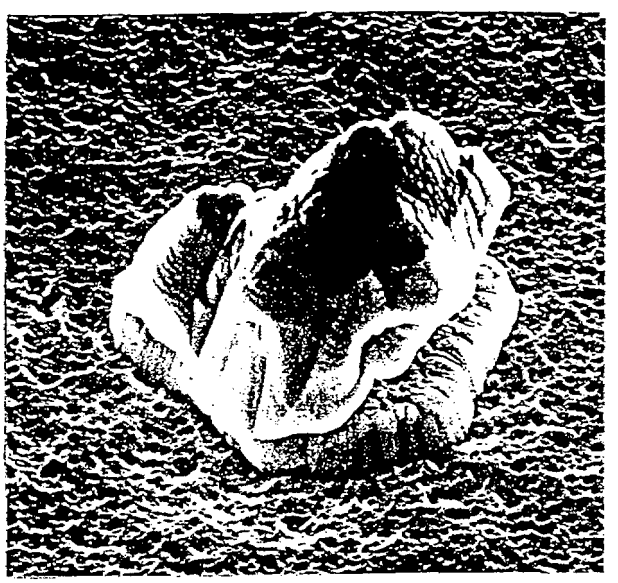


FIGURE 4: SEM MICROGRAPHY OF A NODULES (X **2000)**



This experiment clearly demonstrated that :

> solid particles from the target, ITO flakes coming from the masks or the redeposition areas for instance, or its environment, mineral dusts for instance, are responsible for nodules development on the target surface ;

> ceramic particles such as $Si0_2$, $A1_20_3$, C purposely introduced or naturally present in the ITO material can generate some nodules ;

~ a possible mechanism for nodule growth is that, in an early stage, nodules are cone-shaped with a ITO or ceramic particles on the top and that, in a second stage, they are stabilized by a redeposited ITO overcoat that is less sputtered than the surrounding material ;

and enabled therefore, to draw out some *recommendations for a beffer management of flie ITO suffering sysfems* to avoid the nodule formation problem :

> the sputtering machines should be designed and maintained in a way which reduces the particle contamination risks and minimize redeposition risks on the target ;

> th$_e$ thermal cycling of the target should be limited to reduce the risks of flaking of the back sputtered material ;

> the target manufacturing process should be controlled to avoid risks of particulate inclusions and to provide target microstructure resistant to the flaking phenomenon.

[t is, now, thought that a minimization of the particle sources in the sputtering process (ie an improvement of the machine cleanliness and the utilization of targets that generate a limited amount of particles upon sputtering) will enable, in a near future, to *considerably alievia fe fhe nodu!e formafion problem and reduce fhe periods of process shut down linked wifh fhis problem.*

c) ~ y I M P - u I I E R I N . G _ s Y s I E M s

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The main problem related to the sputtering system technology is the low target utilization rate. With the classical material and sputtering systems, only a slight amount, around 200L of the target weight is used for coating. After, the target has to be removed and replaced which results in process shut down and increases the flat panel display production costs.

The **conductivity of the ITO thin film being strongly dependent on the discharge voltage the solutions developed to solve the target utilization problem must guarantee a low discharge voltage to keep good thin film properties.**

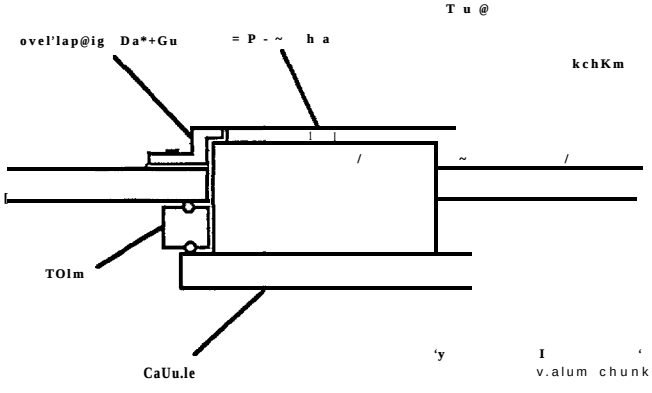
Three type of solutions have been proposed by LEYBOLD to increase the target utilization rate without modifying the ITO **thin film properties :**

- The Overlapping Dark Space **system.**
- The Movable Magnet Array system.
- The superposition of an Oscillating **Electromagnetic Field.**

The Overlapping Dark Space system :

The **idea of the Overlapping Dark Space is to increase the target utilization rate by using specially designed targets with ITO material only in the race track** zone (see *Fig. i).* The non sputtering areas of the target are replaced by a cheaper, easily removable material, e.g. an aluminum insert fixed by screws. For the edge areas of the target, an Overlapping Dark Space shielding is used.

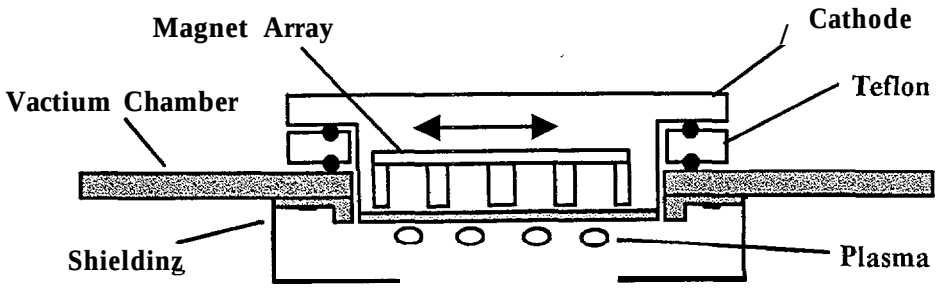
FIGURE 5: CROSS SECTION OF THE CATHODE WITH THE OVERLAPPING DARK SPACE SYSTEM



***The h40vabfe Magnet Array* system :**

Another possibility to increase the target utilization rate is to move the magnet array and scan with the plasma ring over the target. ~ig6 shows the principle of this arrangement. The movement is controlled by electrical engines.

FIGURE 6: CROSS SECTION OF THE CATHODE WITH THE MOVABLE MAGNET ARRAY SYSTEM



This solution has been extensively investigated, but on a double instead of a single racetrack target, to obtain a more homogeneous plasma, by :

> Moving the magnet array in one then two directions,

> Modifying the distances between the two race tracks to optimize the target utilization rate.

> Modifying the target dimensions.

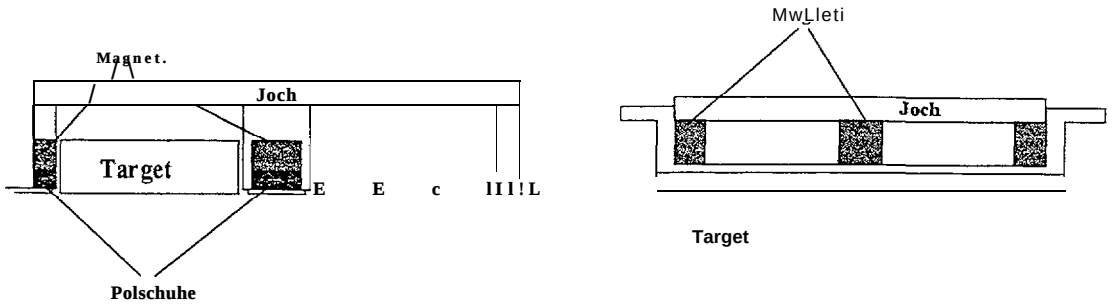
The Movable Magnet Array system has been tested in its optimized version.

Utilization of an Oscilfafing Electromagnetic Field :

A third solution to increase the target utilization rate is to superpose an electromagnetic field perpendicular to the target surface. This secondary field, created by a coil placed around the target, causes a drift of the racetracks depending on the direction of the magnetic field. With an oscillating superimposed magnetic field, the race tracks scan across the target which results in an higher target utilization rate.

This solution has been especially studied for ZPT cathodes (see & z) which are slightly different from the traditional PK cathodes. This type of cathode has been preferred because the gradient of the vertical magnetic field, created by the permanent magnets, is fewer, in the range when this component becomes zero, with type of cathode than with the regular ones.

FIGURE 7: SCHEMATIC OF THE P KAND ZPT CATHODES



ZPT Cath@ PK Cathode

D) SXEC.U-O-N-OhW41VL0 YD-S.WIIERI.NQSYSI.EM.F.0.I-IH E-D.EM.0N.SIIL4Tl.0.N. R.U_N

Fig. 8 gives an overview of the target utl!'izatlon rate and' ot the properties ot thin films obtained, by LEYBOLD, with the **3** improved sputtering systems tested within the framework of the Brite Euram project.

FIGURE 8:

OVERVIEW OF THE TA17GET utilization RATE AND THIN FILMs properties OBTAINED WITH THE 3 IMPROVED SPUTTERING SYSTEMS

	STATE OF ART	GOAL OF THE PROJECT	OVERLAPPING DARK-SPACE	MOVABLE MAGNET ARRAY	ZPT CATHODE	ZPT CATHODE WITH ALTERNNATING MAGNETIC FIELD
Target Utilization	~ 20 wt %	30 wt %	32 wt %	39,4 wt %	35 wt % (estimated)	38 wt % (estimated)
Discharge Voltage at a poowder density of 1,6 W/cm^2	280 V	< 300 V	260 V	295 V	360 V	349-360 V
Specif Resist ity R.T. 200 'C			I,o@cm)	0/.K2crn ~	260pf2crn \	291 cn

W * - [; 3

It can be seen that the best utilization rates (around 380/.) have been achieved with Movable Magnet Array and the Oscillating Electromagnetic Field systems but these major improvements are linked with an unacceptable deterioration of the ITO thin film properties (mainly the electrical conductivity which is related to the discharge voltage).

That is the reason why, the ***Overlapping Dark Space system, that gives the best results in term of thin film properties and that*** **also** ***enables a substantial! increase (from*** 20 ***to*** 32%) **of** ***the ITO target utilization rate, has been chosen as the improved sputtering system*** to ***be tested on St GOBAIA? RECHERCHE'S sputtering machine during the demonstration run.***

E) RES.U.LIS_O_F_tH E_DEM.O.N.SIIWLCWRW

A demonstration run has been carried out on a St GOBAIN RECHERCHE'S sputtering machine equipped with LEYBOLD'S improved sputtering system, to test, at the preindustrial scale, the high density ITO targets developed by METALEUROP and DEGUSSA. More precisely :

➢ Two batches of 8 kgofS1200 powders have been HIPed for the manufacturing of PK500 targets to be used without the Overlapping Dark Space system (PK500 1) and without the Overlapping Dark Space system (PK500 2).

➢ One batch of 8 kg of WM1 200 powder has **been** HIPed for the manufacturing of one PK500 target to be used with the Overlapping Dark Space system (PK500 3).

Fig. 9 pictures out the characteristics of the PK500 targets manufactured with the METALEUROP and DE GUSSA ITO material for the demonstration run together with some indications on the thin films properties.

It can be seen that :

> The powders developed by ~ETALEIJROP and DEGUSSA enable the HiPing of ITO targets whose ***mechanical properties (density; UTS; bending strength) are higher than those of competitive products.*** A reduced sensitivity to the nodule formation problem can be expected from this improvement.

> The powders developed by ~ETALEUROp and DEGt_JSSA enable the HIPing of ITO targets whose ***electrical conductivity thus thermal conductivity are higher than competitive products.*** This improvement can positively effect the thermal management of the ITO targets which are prone to heat during the sputtering operation.

FIGURE 9:

CHARACTERISTICS AND PERFORMANCES OF THE ITO TARGETS AND THIN FILMS PRODUCED DURING THE DEMONSTRATION RUN

TARGET	POWDER PROCESS	TARGET CHARACTERISTICS						FILM CHARACTERISTICS			
		HIP density (%)	Bend Str (MPa)	Elect. Res. (,ufl.cm)	Impurities (ppm)	Design (-)	Util rate (%)	Cathod volt (v)	Elect. Res. @200°c (@cm)	Elect. Res. @300 °c (,@.cm)	opt. trans @Q,c
PK 01	120 processing route	98.6	87.3	430	< 100	regular	23	280	233	150	98.2
02	20 processing route	98.4		1,255	600'	ODS**	32	286	251		97.5
PK 03	WM1 200 processing route	98.6	105.0	430	< 100	0DS**	-	286	256	227	97.1
Competitive product		97.0	88.0	1,200	1,000	regular	-	281	234	158	97.4
Goal of the project		> 95	max	min	< 100	-	> 30	-	150	150	98.0

* : 5i02 (500 ppm) in solution

** : ODS = Overlapping Dark Space system

> all the targets developed by METAL EUROP and DE GUSSA meet the ***goal purity for a 4N material,*** except for target number 2 which suffered from a SiO_2 contamination problem upon powder synthesis. This SiO_2 contamination problem is probably responsible for the high electrical resist ivity of the ITO thin films deposited from target 2.

> The ***improved sputtering system de ve!oped by LEYBOLD significantly increases the uti~ization rate of an /TO target*** without deteriorating the optical and electrical properties of the ITO thin films. The goal of 30 0/0 of utilization rate has **been achieved.**

> The optical and electrical properties of the ITO thin films deposited from METALEUROP and DE GUSSA targets are, at least, as good as competitive products but do not meet, today, the goal for an electrical conductivity of 150 'C ~.ohm cm under normal deposition conditions. It has been, however, demonstrated that this objective can be fulfilled by carrying out the deposition step at 300 "C instead of 200 'C.

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As already mentioned on ~ 3.c, one of the main problem related to the sputtering system technology is the low target utilization rate. With the classical material and sputtering systems, only a slight amount, around 20 0/0 of the target weight can be used for coating and the sputtering operations produce, as by products, large amounts of partially used ITO targets that cannot be used any longer for sputtering operations.

Two solutions have therefore independently been studied by METALEUROPand DEGUSSA to develop recycling routes **to recover valuable iridium from** ITO scraps.

The solution developed by DEGUSSA concerned the recycling of ITO "new" scraps by direct remilling of ceramic material then further HIPing. The pilot campaign carried out on 20 kg batches proved the feasibility of this concept since the performances of ITO targets and thin films produced from the secondary material weren't significantly different from those achieved with primary powders.

This solution that is cost effective but that can only be applied on in house scraps completes the second approach proposed by METALEUROP that can treat ITO scraps coming from different sources,

The idea developed by METALEUROP is reintegrate the iridium metal present in the **ITO** scraps in the METALEUROP'S iridium primary production line. **The hydrometallurgical** process developed for the **ITO** target recycling :

b The leaching off TO scraps in an acid solution, for a total dissolution the ITO material.

b The separation of the iridium metal from the tin metal in the acid solution with an organic extractant.

- The r$_{ecov}$e~$_{Y}$ ***of the va/uable iridium*** from the acid solution by an electrolytic process.

w The ***regeneration of the organic extractant.***

The validity of the process has been checked out during pilot scale campaign carried out on 300 kg of scraps. The overall recovery rate for iridium obtained during these trials was about 78 O/. thus approaching the goal (80 O/.) defined at the beginning of the project.

5.- CONCLUSION

This European funded program enabled :

☞ The development of high purity (4N) In_2O_3 powder (S1200) and In_2O_3 / SnO_2 powder **(WM1 200)** for ITO target application.

☞ The development of an improved densification technic (Hot Isostatic Pressing) for the manufacturing of high (95 °/0) density ITO targets.

@= The development of an improved sputtering system **(Overlapping** Dark Space system} to increase of the ITO target utilization rate form 20 to 300/..

m= A better comprehension of the nodule formation problem which prematurely reduces the ITO target utilization time.

☞ The development of cost effective recycling methods to recover valuable iridium from partially used **ITO** targets.

All the technologies and the products developed, in a first step, at the laboratory scale have been, in a second step, tested at the preindustrial scale. As refereed on Fig. 10 which gives an overview of the project goals and realizations, most of the quantitative objectives defined at the beginning of the program **have been achieved.**

The potentials benefits that can be expected from this study are :

- ***Reduction of the production costs of the flat panel display and electrochromic devices.*** It is thought that the application of the recommendations for a better management of the particles sources in a sputtering machine will substantially alleviate the problem caused by the formation of black nodules on the target surface during sputtering operations and reduce a cause for frequent process shut down. It is also thought that the utilization of the Overlapping Dark Space system will considerably increase the target utilization life, thus increasing the periods of time when the sputtering system works under effective steady conditions. The productivity of the sputtering systems should be increased. The Overlapping Dark Space system is now available on some LEYBOLD'S sputtering machines.

- ***Slight improvements of the I TO thin fi~rn properties brought by the utilization of high density ITO targets.*** The high density ITO which weren't available in the past on the market may **become available, should** LEYBOLD MATERIALS and DE GUSSA decide to invest a plant using the HIP techniques.

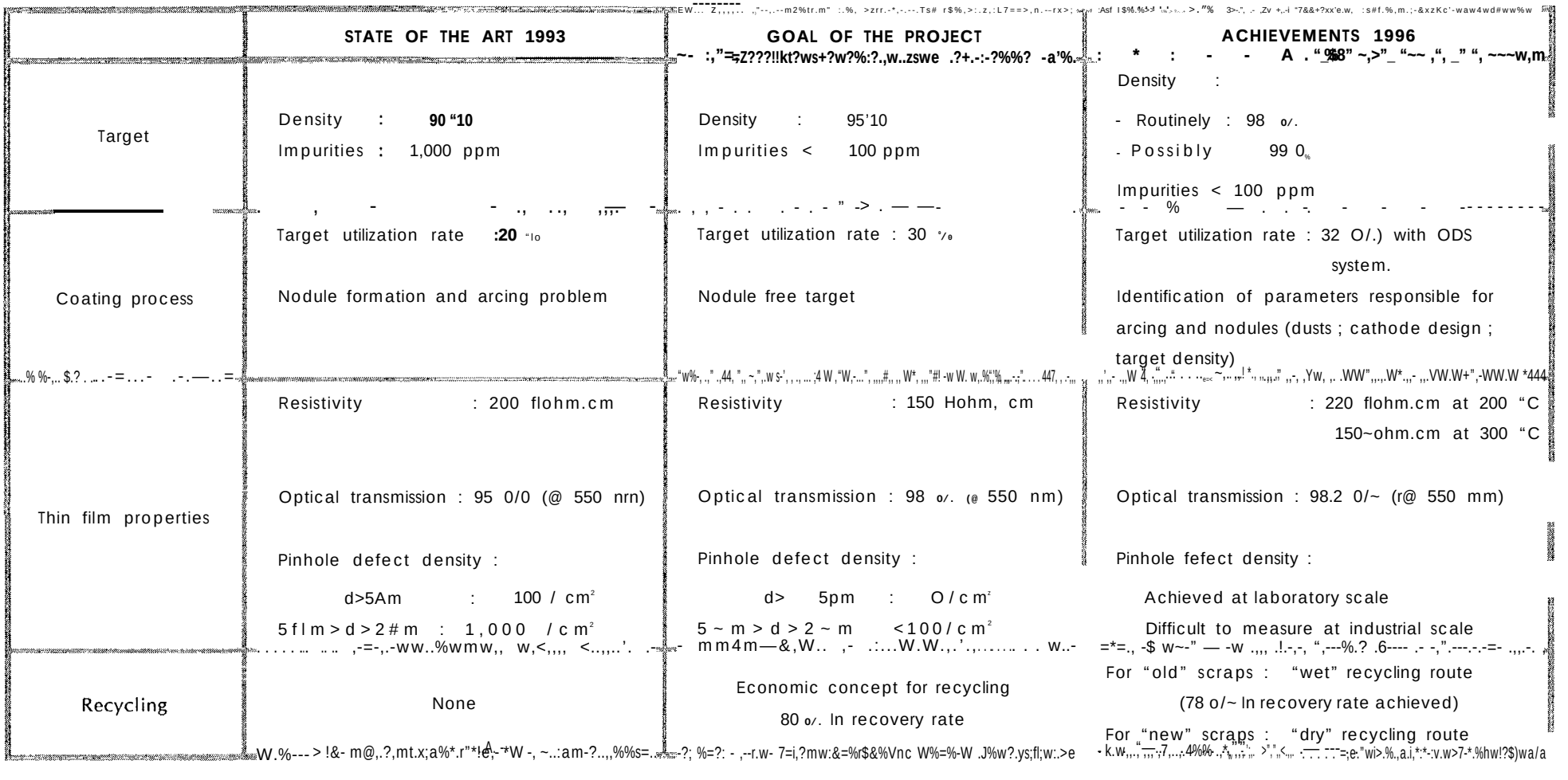
- ***Better management of worldwide resources*** brought by the utilization of a recycling method which is already operational in a METALEUROP'S plant.

This research program was, however, very interesting on a technical point of view and all the partners METALEUROP RECHERCHE, DEGUSSA AG, LEYBOLD AG and St GOBAIN RECHERCHE would like to thank the European Community **for** the help provided during this 3 years project.

6.- ACKNOWLEDGMENTS

Acknowledgment is made to the European Community for the financial support provided, within the framework of the Brite Euram contract BRE2-CT92-0298 and to its coordinating officer Dr N. Hartley.

FIGURE 10:



	STATE OF THE ART 1993	GOAL OF THE PROJECT	ACHIEVEMENTS 1996
Target	Density : 90 “10 Impurities : 1,000 ppm	Density : 95’10 Impurities < 100 ppm	Density : - Routinely : 98 o/. - Possibly 99 0‰ Impurities < 100 ppm
Coating process	Target utilization rate :20 “lo Nodule formation and arcing problem	Target utilization rate : 30 ‘/e Nodule free target	Target utilization rate : 32 O/.) with ODS system. Identification of parameters responsible for arcing and nodules (dusts ; cathode design ; target density)
Thin film properties	Resistivity : 200 flohm.cm Optical transmission : 95 0/0 (@ 550 nrn) Pinhole defect density : d>5Am : 100 / cm² 5flm>d>2#m : 1,000 / cm²	Resistivity : 150 Hohm, cm Optical transmission : 98 o/. (@ 550 nm) Pinhole defect density : d> 5pm : O / cm² 5 ~ m > d > 2 ~ m <100/cm²	Resistivity : 220 flohm.cm at 200 “C 150~ohm.cm at 300 “C Optical transmission : 98.2 0/~ (r@ 550 mm) Pinhole fefect density : Achieved at laboratory scale Difficult to measure at industrial scale
Recycling	None	Economic concept for recycling 80 o/. In recovery rate	For “old” scraps : “wet” recycling route (78 o/~ In recovery rate achieved) For “new” scraps : “dry” recycling route