Министерство образования и науки Российской Федерации

федеральное государственное автономное образовательное учреждение

высшего образования

«НАЦИОНАЛЬНЫЙ ИССЛЕДОВАТЕЛЬСКИЙ

ТОМСКИЙ ПОЛИТЕХНИЧЕСКИЙ УНИВЕРСИТЕТ»

Институт физики высоких технологий

Направление подготовки 22.04.01 «Материаловедение и технологии материалов»

Кафедра наноматериалов и нанотехнологий

МАГИСТЕРСКАЯ ДИССЕРТАЦИЯ

Технология керамических мишеней для напыления прозрачных электропроводных покрытий

УДК 666.65:621.793.7

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Томск – 2016

Планируемые результаты обучения
по ООП 22.04.01 «Материаловедение и технологии материалов»,
профиль «Производство изделий из наноструктурных материалов»

Код результата	Результат обучения
Р1	Осуществлять сбор, анализ и обобщение научно-технической информации в области материаловедения и технологии материалов с использованием современных информационно-коммуникационных технологий, глобальных информационных ресурсов
Р2	Работать с патентным законодательством и авторским правом при подготовке документов к патентованию и оформлению ноу-хау
Р3	Выполнять маркетинговые исследования и анализировать технологический процесс как объекта управления, разрабатывать технико-экономическое обоснование инновационных решений в профессиональной деятельности
Р4	Руководить коллективом в сфере своей профессиональной деятельности, толерантно воспринимая социальные, этнические, конфессиональные и культурные различия
Р5	Внедрять в производство технологии получения керамических, металлических материалов и изделий, в том числе наноматериалов, быть готовым к профессиональной эксплуатации современного оборудования и приборов, позволяющих получать и диагностировать материалы и изделия различного назначения.
Р6	Разрабатывать новые и модернизировать существующие технологии получения керамических, металлических материалов и изделий, в том числе наноматериалов
Р7	Внедрять системы управления качеством продукции в области материаловедения,эксплуатировать оборудование,позволяющее диагностировать материалы и изделия из них, в том числе наноматериалы
Р8	Действовать в нестандартных ситуациях, нести социальную и этическую ответственность за принятые решения, выбирать наиболее рациональные способы защиты и порядка в действиях малого коллектива в чрезвычайных ситуациях
Р9	Общаться в устной и письменной формах на государственном языке РФ и иностранном языке для решения задач профессиональной деятельности, подготавливать и представлять презентации планов и результатов собственной и командной деятельности, формировать и отстаивать собственные суждения и научные позиции
Р10	Самостоятельно осваивать новые методы исследования, изменять научный, научно-педагогический и производственный профиль своей профессиональной деятельности
Р11	Применять принципы рационального использования природных ресурсов, основные положения и методы социальные, гуманитарные и экономические подходы при решении профессиональных задач с учётом последствий для общества, экономики и экологии.
Р12	Использовать основные категории и понятия общего и производственного менеджмента в профессиональной деятельности

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Кафедра наноматериалов и нанотехнологий

УТВЕРЖДАЮ:

Зав. Кафедрой

_____ _____ О.Л. Хасанов

(подпись) (дата)

ЗАДАНИЕ

на выполнение выпускной квалификационной работы

В форме:

магистерской диссертации

Студенту:

Группа	ФИО
4бм4и1	Жакмен Маноэль Франсуа Мари

Тема работы:

Технология керамических мишеней для напыления прозрачных электропроводных покрытий

Утверждена приказом директора	No 2599 от 06.04.2016

Срок сдачи студентом выполненной работы:	

ТЕХНИЧЕСКОЕ ЗАДАНИЕ:

Исходные данные к работе	Объектом исследования является технология получения кармических мишеней для напыления прозрачных электропроводных покрытий
Перечень подлежащих исследованию, проектированию и разработке вопросов	Изучение поведения оксида индия-олова при спекании; Изучение процессов консолидации оксида индия-олова в зависимости от режимов грануляции.

Консультанты по разделам выпускной квалификационной работы

Раздел	Консультант
1. State of Art 2. Characterisation technique 3. Experimental Part 4. Sintering Conditions 5. Compaction Behaviour of ITO Powder	Эдгар Сергеевич Двилис
6. Social responsibility	Галина Владимировна Лямина
7. Financial management, resource and resource efficiency	Галина Владимировна Лямина

Дата выдачи задания на выполнение выпускной квалификационной работы по линейному графику	

Задание выдал руководитель:

Должность	ФИО	Ученая степень, звание	Подпись	Дата
С.н.с	Э. С. Двилис	д. ф-м. н.		

Задание принял к исполнению студент:

Группа	ФИО	Подпись	Дата
4бм4и1	Жакмен Маноэль Франсуа Мари		

SUMMARY

TABLE OF FIGURE

TABLE OF TABLE

ABSTRACT

In this study the consolidation of ITO ceramic by uniaxial compaction method have been exterminate. The utilization of granulated powder, powerful ultrasound assistance and collector pressing has been used as improvement of the conventional compaction technique of the raw powder. We work with ITO powder with a particle size average of 50 nm and a chemical composition of 10 wt% of SnO_2 content. The granulation of the powder permits to reduce the compaction coefficient of the raw powder. granulated powder show improvement of the powder compaction and permit to use more high pressures than the with the raw powder. The gain of ITO hand processing is 50 MPa of maximal working pressure (ITO initial maximal pressure is 650 MPa for 86% of density) and an increment of the final density up to 5%. we use also 2% of Polyvinyl Butyral as plasticizer and we obtain a powder with 400 MPa more of working pressure and which lead to 96,1% of density after compaction at 1 GPa. The condition of sintering has also been studied and show that grain growth is limited with high pressure and small sintering time and a good sintering temperature is 1550 °C

INTRODUCTION

The technologies such as photo voltaic, LCD screen, LED, so technologies who use light to produce electricity the inverse electricity to produce light need special kind of electrode Transparent conductive oxide (TCO). TOC material combine the transparency to visible light and electrical conductivity. we will interest our study to the most famous of them the indium tin oxide (ITO).

The production of TCO film is in general produce by RF magnetron spluttering. The production of high quality film depends mostly of quality of the target. The aim of this internship is to obtain a target with a micro and macro structural homogeneity, but also a maximal density.

Target are generally produce by cold or hot isostatic pressing. These techniques are very efficient in term of homogeneity and density. the main problem of this technique is it price. To reduce the price of production of target we want to replace these expansive techniques by uniaxial pressing which is more cheap

during this internship we use different improvement of the conventional uniaxial pressing such as powerful ultrasound assistance and or collector pressing. With have also try to use granulated powder to improve the compaction in comparison of the raw powder

This report is divide in several part, a state of art about subject connected to our work like collector pressing, sintering phenomena, RF magnetron spluttering... after an experimental part, two study have been carried out first one on the sintering behavior and the second one on the utilization of granulated powder

I) STATE OF ART

1) Indium Tin Oxide

Indium tin oxide (ITO) is a material composed by indium oxide In_2O_3 doped with tin oxide SnO_2. This material combines two interesting properties, electric conductivity and if it is in thin layer optical transparency. This combination of properties is the definition of transparent conductive oxide TCO.

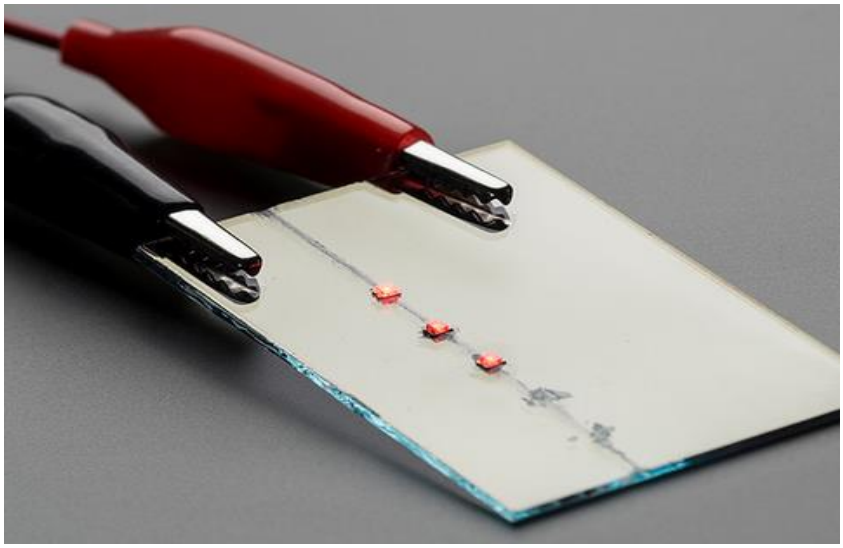


Fig 1 : Picture of glass coated with ITO represent the conductivity and the transparency

The conductivity of ITO depends directly on the percentage of dopant (tin oxide) but in the other hand tin reduce the optic transparency of ITO. In general, the amount of tin oxide is around 10 %. J. Ba et. al. [1] Have investigate the conductivity of ITO regarding their amount of tin oxide dopant, the better value of conductivity 2,6 $S.cm^{-1}$ have been found for 15% of SnO_2.

Actually ITO is the most use TCO in the industries. His great properties and the crucial place of TCO in modern technology place ITO in a strategic position and the price of the indium oxide always increase it is why decreasing the price of the production of manufactured device is important.

2) Radio Frequency Magnetron Spluttering

RF magnetron spluttering is a technique of Physical Vapour Deposition (PVD) this technique consists to evaporate a material and condense it on a substrate. [2]

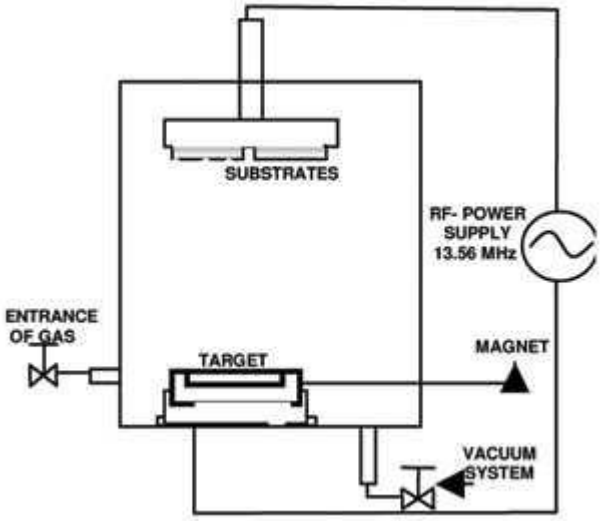


Fig 2 - Sketch of RF Magnetron spluttering

In figure 2 we can see the architecture of the RF magnetron spluttering. The principal is easy we enter argon gas inside the chamber, the target is composed by the material we want to spluttered in our case ITO, and the substrate on the top can be a sapphire, a slice of glass, a silicon wafer... The gas inside the chamber is turn into a plasma thanks to the RF-Power supply the cathode of the system is the target and the anode the substrate. When the gas is turn into plasma the ionised atom is attracted by the target due to coulomb attraction. The bombardment of the target by ionised atom lead to an ejection of target material and these atoms finally condensate on the surface of the substrate this is the principal of the spluttering.

For the magnetron part, the ionised atom of argon and their free electron are localized near the target because of the magnetic field produce by the magnet this lead to two main advantage first it enhances erosion of the target by increasing concentration of ion gas near to it but also it protects the substrate from them.

3) Sintering

The elaboration of ceramics is decomposed in two phase first the compaction of the powder to create a green body and second the sintering. The sintering can be describing macroscopically by the increment of the relative density by the shrinkage of the volume and the strengthening of the object [3]. These phenomena have as driving force the reduction of the system's surface energy. The reactions in cause are generally mass transport [3] but sometime also sublimation condensation [4]. Mathematically it exists two approach to explain the sintering reaction: The Frenkel approach (1945) this approach is to consider the system like the coalescence of viscous spheres, this driven by surface tension The Pines approach (1946) This other approach is to consider the evaporation of the emptiness

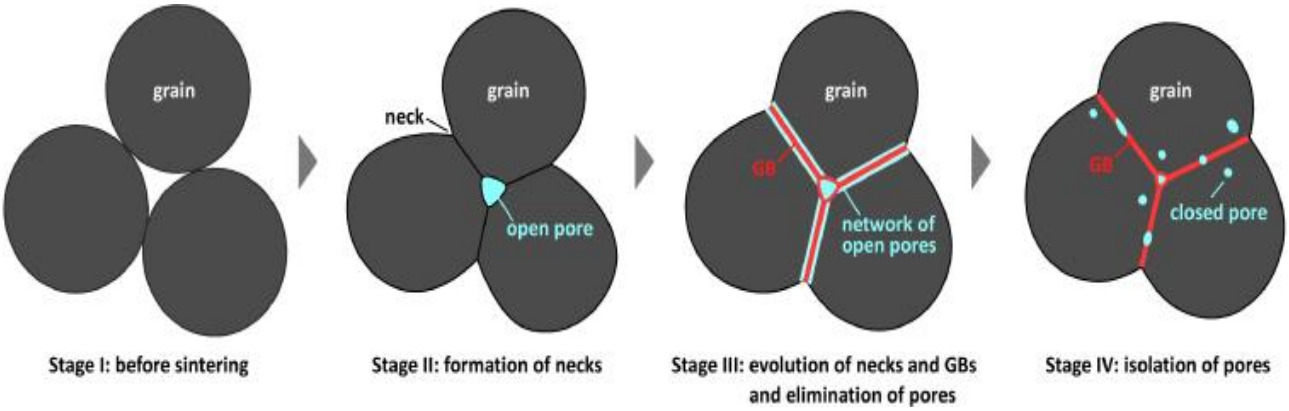


Fig 3- Sketch of the four sintering stages

in figure 3 we can see he four different stage of the sintering. Before sintering if the compaction has been well done, we can expect that the particles are in contact between each other. The second stage of the sintering is the formation of bridge between the particle at this step the solid have a network of open porosity. Third stage is the expansion of these bridge and the diffusion of mater in these particle, this stage coincides with the reduction of the porosity pores. The last stage is the ceramic state with grain join, grain and closed porosity.

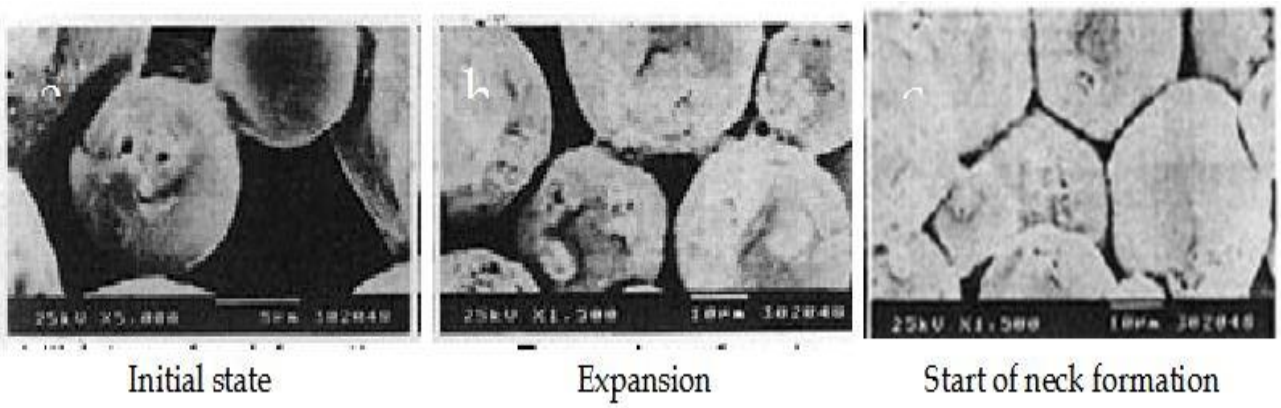


Fig 4 - MEB picture of three different step of the sintering [5]

in figure 4 we can see the MEB picture which are representative of the sintering in a spark plasma sintering (SPS) device.

We see the formation of the bridge in the second step and the modification of the grain shape in the last step.

This SPS device is a particular device and it is not our aim to explain it. The conventional sintering is usually carried out in a furnace with or without controlled atmosphere. The sintering is generally composed by a slow increment of temperature to attain the sintering temperature and after a slow cooling. The sintering programme can be composed by different temperature step for removing binder plasticizer.

4) Isostatic Pressing

Actually to consolidate ITO target the industries use the cold or hot isostatic pressing (CIP/HIP) technology. [6]

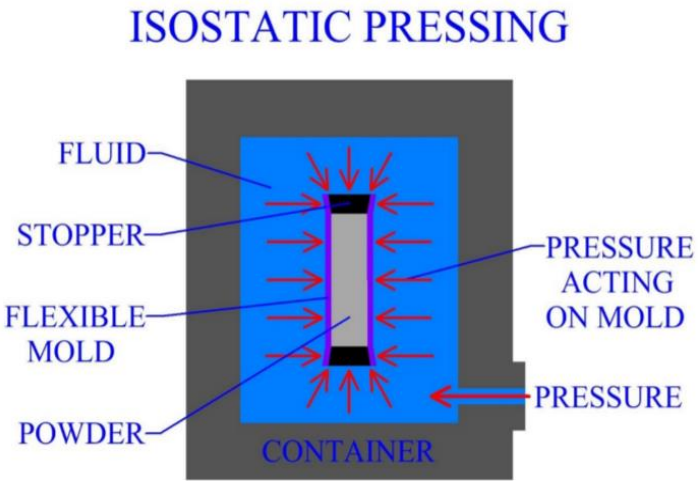


Fig 5 - Isostatic pressing representation

in figure 5 we can see the representation of the isostatic pressing. The CIP technique principal is to fill a flexible mold with powder. The mold is submerged in a fluid this media will be under pressure to compact the mold with a perfect homogeneity. the mold can be made in polymer in or in flexible metal. The material of the mold depend mostly on the interaction between it and the powder and or the fluid, the pressure applied on it and the temperature of the fluid in case of HIP.

The main advantage of this technique is the great homogeneity of the compaction. The pressure is applied in all the surface of the mold by the fluid which can be a gas or a liquid depending of which pressure condition we want to use. the deformation of the mold is theoretically the same on every point of this surface so the only inhomogeneity which remain will be in the hearth of the green body. In practice the corner of the mold do not have the same compaction pressure so a few inhomogeneity remains on these part. Taking account these little detail this compaction technique stays the most suitable for compaction of powder hard with big elastic after effect such as ITO.

In the other hand this technique presents many issue for its industrial application. First of all, this technique does not allow the engineer to use complicated shape [7], otherwise the compaction will be non-homogeneous. This ascertainment is not a problem for our research because a target does not need any particular shape.

But the most important problem is the price of this method. The single utilisation mold, which can be made in expensive material, and which need a very precise shape without defect is expensive and increase price of the experiment. If the flexible mold contain defect, during the compaction inhomogeneity of the green body or tearing of the mold which lead to loose of the powder can happen.

Second point is the device, because of the architecture of it its price is way more important than a conventional uniaxial press. This device is designed to applied temperature, and very high pressure on the fluid, this fluid need to be contained in sealed and pressure resistance container.

Due to the expensiveness of this technique the aim of our research is to use conventional compaction technique to reduce the production price of ITO target.

[6] C. Liu et al success to produce target of ITO with an CIP at 300 MPa, after sintering at 1550 °C for 8 hours they reach a relative density of 99,6%.

5) Uniaxial Conventional Dye

During this intern-ship we used different compaction technologies as improvement of uniaxial pressing. In ceramic technologies the simplest press is the uniaxial static press. [7]

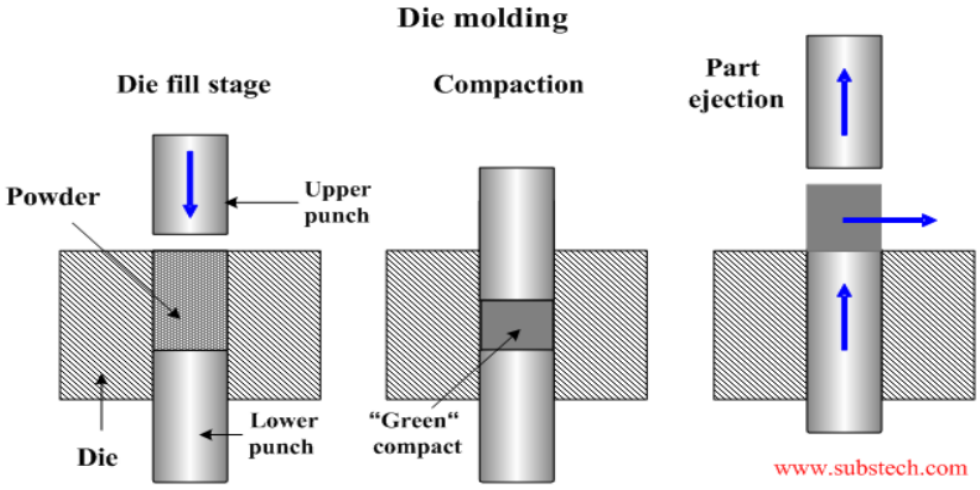


Fig 6 - Tree stage of conventional press

In figure 6 we can see that the PU is composed by a dye and two punch in uniaxial press the pressure is applied only on the upper punch. The compaction is composed by tree steps: first the dye is filled by the powder and the punch are put in place. Second the pressure is applied on the up punch and the powder is compacted to become a green body. Third the green body is ejected from the dye.

During a conventional compaction the maximal value of wall friction force and the corresponding maximal value of densification degree of a green body are find in the region of conjugation of the movable punch and the mold. Because of loss of the compaction force for overcoming the wall friction forces inside of the green body the minimal densification is located at the most distant part from the moving punch. The distributions of the wall friction forces and the density in the height of powder body are asymmetric and non-uniform. [8]

6) Collector Pressing Dye

To solved the problem of non-uniformity of the green body in uniaxial pressing, a modification of the dye can be applied.

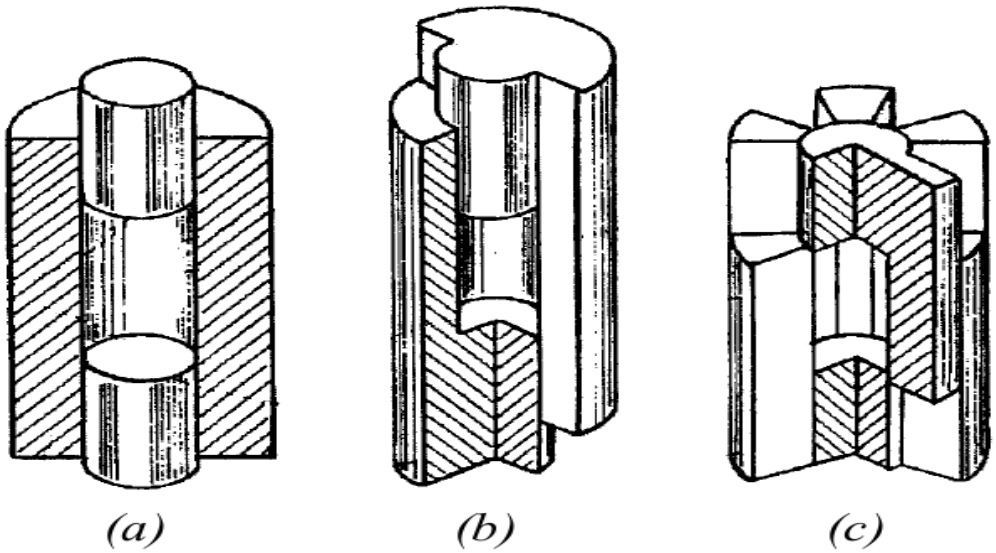


Fig 7 - Representation of uniaxial press (a) and collector pressing dye with 2 passive compose (b) and six passive component (c) [9]

The collector pressing is a specific modification of the conventional uniaxial pressing. The dye of collector pressing can be describe like it showed in figure 7. In collector pressing both punch moves jointly with (for example) two (b) or six (c) passive components of the lateral surface disposed in the opposite geometries one up in front one down.

The distributions of the wall friction forces and the density of a powder body are centrally symmetrical, and the average density in the horizontal cross section is invariable along the green compact height. But the non-uniformity of density distribution remains in the horizontal layers of a compact. The increment of the quantity of alternate oppositely moved parts of the passive shape-forming surface results in minimization of the non-uniformity of density distribution in the horizontal cross sections of a powder body. [10]

7) Powerful Ultrasound Assistance

beside using lubricant and or plasticizers to reduce dye wall friction [11] another technique consist in using an intensive mechanical ultrasonic vibration applied to the mold of the dye. Thanks to this ultrasonic wave the particles will periodically detach from the dye wall. Taking account that the friction forces operate only when these two surfaces are in contact the dye wall particle friction is clearly reduced. Thus, the dye-wall friction coefficient will decrease in proportion to the ratio of time periods of the contact and detachment [12].

a) Description of the Device

The compaction of dry powder with PUA is carried out in a dye include in resonance-size wave-guide. The dye can be a conventional uniaxial dye or a collector pressing dye this dye can be use until 1,4 GPa. [13]

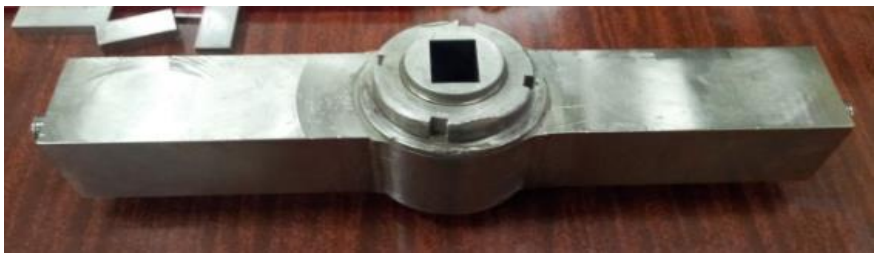


Fig 8 - Picture of the wave-guide with the rectangular collector pressing dye.

The ultrasonic waves are produce by two magnetostrictive (MS) transducer who produce the vibration by apply magnetic field to a MS material.

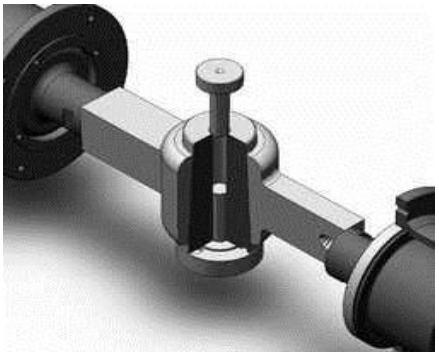


Fig 9 - Picture of the two magnetostrictive transducer and a schematic representation of the PUA device

b) PUA action during compaction

The pressing is carried out at room temperature [13], we can take in account that the dissipation of the ultrasonic wave in the dye produce heat (We measure 60 °C after one experiment). Standing wave are induced in the dye to get the maximal amplitude in the region of powder compacting. At the initial stage of compaction, the powder has low apparent density so a high sound attenuation so the wave affect mostly the dye wall and reduce the dye wall friction. When we increase the compaction pressure the inter-particle bonds become stronger and numerous and increase sound propagation through the powder. Due to vibration interaction of particles and agglomerates the ultrasonic action also decreases the inter-particle friction forces [12].

The dye has fixed resonance frequency, when we apply a quasi-resonance condition we can compress nano powder uniformly. That condition signify that the ultrasound wave amplitude are proportional to nanoparticle or agglomerate size. Like that at the beginning of the compaction the packing of the powder remains uniform and the furtherer part of the compaction have the minimal gradient of mechanical stresses.

A second advantage of the ultrasound action on the powder is the deagglomeration of the nanoparticle during pressing and the mechanic-activation of the particle which reduce the sintering temperature and by the same occasion the grain growth of the ceramic [15].

8) Improvement of Uniaxial Pressing

Some comparative study implying these different technologies have been done in previous study.

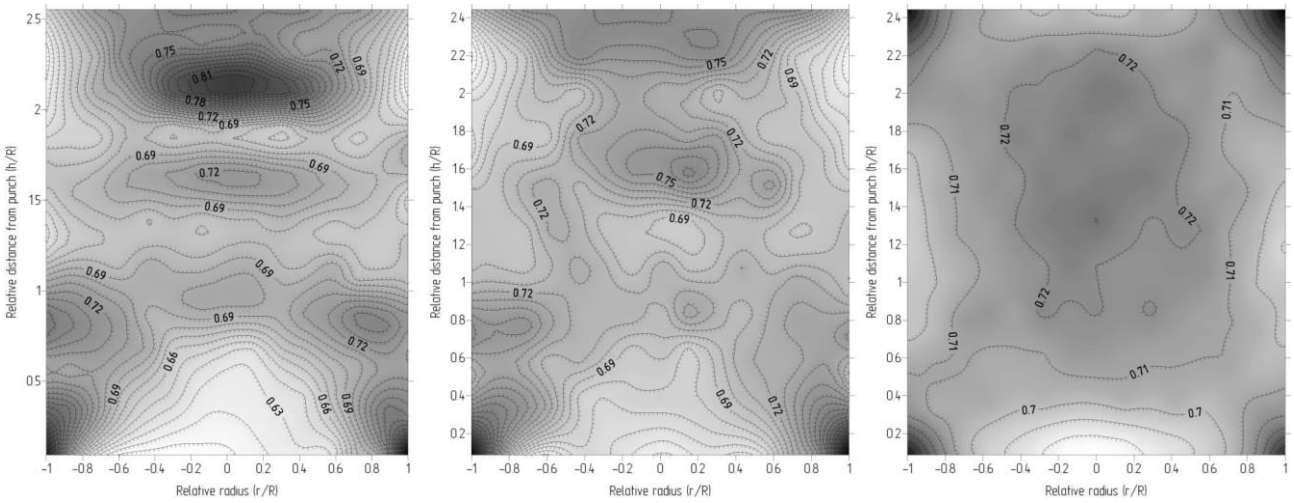


Fig 10 - Density distribution in the green body of BaTiWO nanopowder compacted by UP (a), PUA (b) and CP (c). [8]

We can see in figure 10 that PUA action permit to reduce inhomogeneity of the green body but only locally the axial inhomogeneity remain the top is more compact than the bottom like in uniaxial conventional press. In the other hand the action of collector pressing is even more impressive, even if the green body is slightly axially inhomogeneous we can see that this technique is way more efficient than PU.

Figure 10 is not totally representative of the real homogeneity of the green body because these picture are an average of the density distribution.

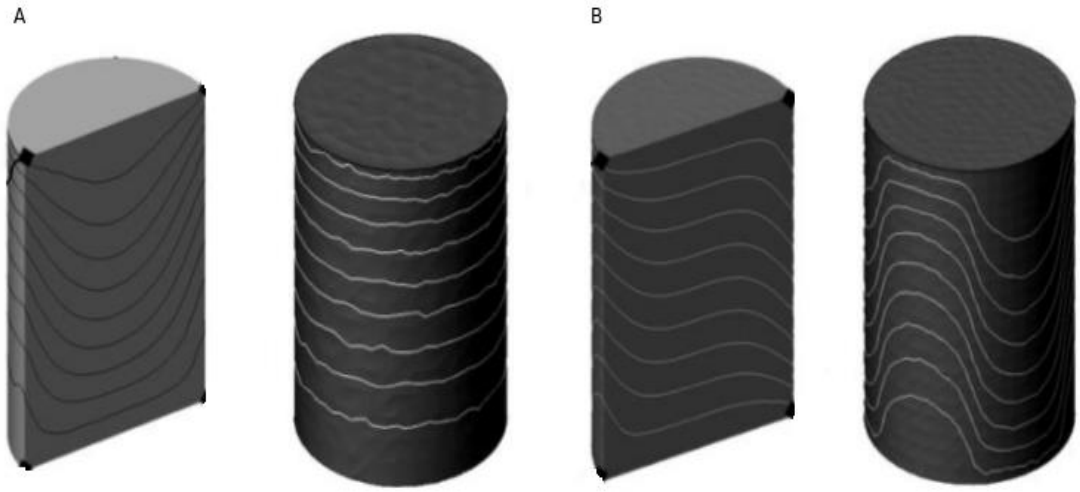


Fig 11 – representation the displacement of layer in cylindrical green compact from simulation of compaction with PU (A), and CP (B) [10]

In figure11 we can see the inhomogeneity of the compression in conventional UP, in collector pressing with six passive part. This representation has been verified experimentally by compression of zirconium oxide add with several layer of copper as colourant. Like we can see in figure 11 the deformation of the green body depends of how much passive part the collector pressing have. So there we see that collector pressing keep inhomogeneity in radius of the green body.

II) CHARACTERISATION TECHNIQUE

1) Scanning Electron Microscopy

during this work we used different method of analysis to obtain different information such as size/shape of particle, grain shape, chemical composition, speed of sound in the solid, particle size repartition...

SEM: JSM-7500 FA (JEOL)

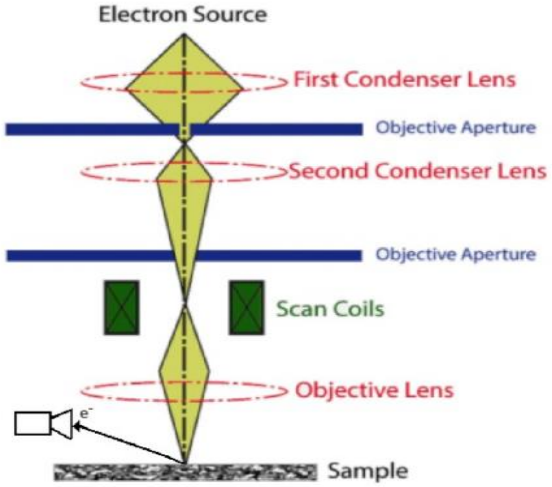


Fig 12 - SEM architectural representation

Electrons are produced at the top of the column by a heated tungsten wire or by a field emission gun (FEG), accelerated down and passed through a combination of electromagnetic lenses and apertures to produce a focused beam of electrons which hits the surface of the sample. Samples are placed in a secondary chamber, all the part of the microscope are under good vacuum. This vacuum is essential, firstly to limit the oxidation of the electron's source but also to avoid collision between electrons and air molecules (these collisions can deviate the beam).

The position of the electron beam on the sample is controlled by scan coils situated above the objective lens. These coils enable the beam to scan over the surface, they drive the beam in applying different magnetic fields. The beam scanned an area and obtain is topography thanks to the secondary electron who are "reflected" by the surface.

2) X-Ray Diffraction

XRD 7000S (Shimadzu) with Cu Kα target

The X-Ray Diffraction is a technique of chemical analysis this technique work only on crystalline mater and not for amorphous one.

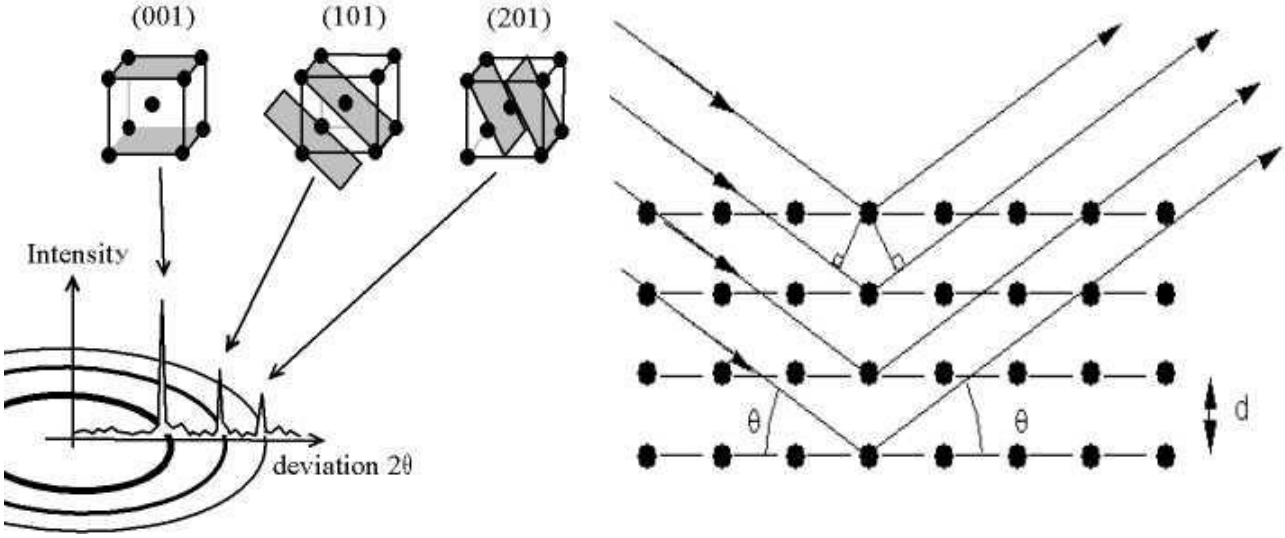


Fig 13 - Representation of the crystallographic plan and the reflection of the X ray beam on it.

The principal of this technique is to see the diffraction of an X-ray beam after it through a crystal. A crystal is composed by different crystallographic plan spaced by different fraction of the lattice parameter of the material. The X-ray beam hit the crystallographic plan with an angle of incidence θ so the beam is reflected by these plan with an angle of diffraction of 2θ.

This technique gives to the searcher several raw information in his spectrum. First the spectrum is defined by the lattice parameter of the material and his crystallographic network, thanks to it we can obtain the chemical composition of the material and his state of stresses. A spectrum incomplete can bring information about the orientation of a crystal. Second point if we analyse a melting of different material we can have a quantitative analysis of it thanks to the amplitude of each spectrum.

3) Particle Size Analyser by Laser Diffraction

SALD 7101 (Shimadzu)

The size analyser by laser diffraction is a device for quantitative analysis.
When a laser beam passes through a sample of dispersed particle the device measures the angular variation of the intensity of the scattered light. Large particle scatter light at small angle relative to the laser beam and small particle at higher angle. After analyse of the data and taking in account the Mie theories, particle size is represented by the diameter of a spherical particle with the same volume than the original particle.

Our SALD device have a laser calibrate at 375 nm and the device is able to make a size repartition of particle with size between 10 nm to 50 μm.

4) speed of sound measurement

We performed the analysis of speed sound thanks to an ultrasound thickness gauge. This device use ultrasound to estimate the thickness of a material. We modified it utilisation to our needs. After a calibration to 5900 m/s using 8 cm steel plaque we carried analysis on our green body sample. We know the thickness of our sample so by multiplying speed of sound in steel by the rapport of real thickness one the measured one we obtain the speed of sound in the green body

III) EXPERIMENTAL PART

In this part we will describe the material and device at our disposition, but also the experimental condition.

1) Compaction device

for the compaction of our powder we have at our disposition a uniaxial press. Precision hydraulic press IP-500M having loading force up to 500 kN and system of control pf current values of loading and displacement of the punches to plot the densification curves of powders and to press the powder samples (Fig 14).

Fig 14 - Precision hydraulic press IP-500M

with have also at our disposition tree dye. A conventional uniaxial dye cylindrical made in steel with a diameter of 14 mm (Fig 15)

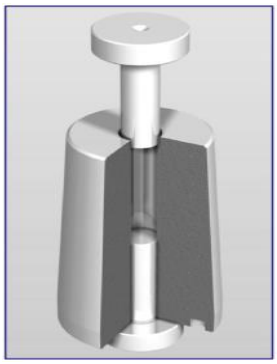


Fig 15 - Conventional uniaxial dye

We have also at our disposition two kind of collector pressing a spiral collector pressing (fig 16 (c)) and a rectangular collector pressing (Fig 8, 17).

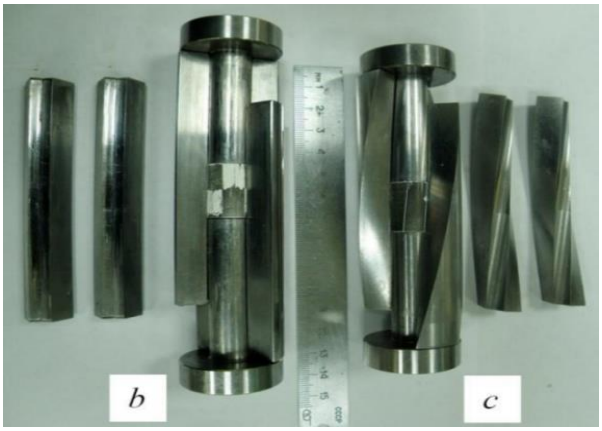


Fig 16 - Picture of straight collector pressing (b) spiral collector pressing (c) [10]

In figure 16 we can see one the left the straight collector pressing and on the right the spiral collector pressing which can be use until 0.8 GPa. The collector pressing in spiral give even more good result than the straight one because the spiral type of collector method was developed for dry powder pressing with a torsion.

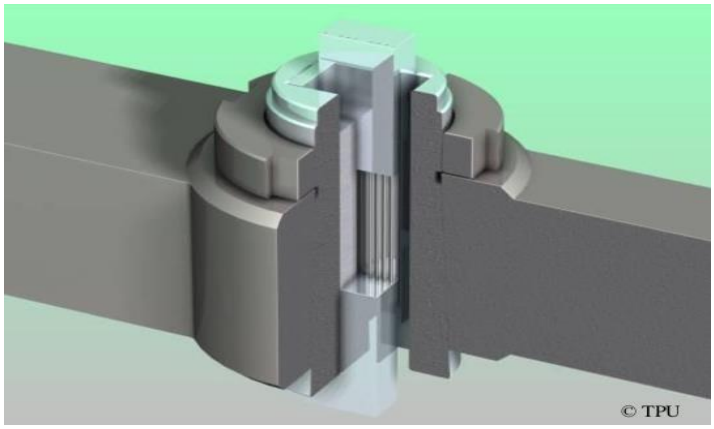


Fig 17 - Rectangular collector pressing.

Figure 17 show us the rectangular collector pressing dye, this collector pressing is composed by two passive part located on the big surface of the green body.

All of our dye has been joined with the ultrasonic wave-guide by a shrinkage fit. Like this it is possible for us if we mount the two magntostricteur on it, to make some sample with powerful ultrasound assistance.

2) Sintering and relative density

The sintering is carried out in a conventional furnace without control of the atmosphere (normal atmosphere). this furnace can be used until 3000 °C with a precision of 1°C and a thermocouple precise at 0,1 °C. the walls of the furnace are composed by porous aluminium oxide which is have a good thermal isolation first of all due to the porosity of it and also its refractive properties.

Sintering condition: in the next part of this report will talk about our research about improvement of sintering condition. there we will explain our usual sintering condition they have been use to sintering our sample during the study of ITO granulation powder. Sample has been sintered during two hours at 1550 °C with 100 °C/hour for the warming and 200 °C/hour for the cooling. Because of the ITO penetration inside the alumina, we put them on a support of ITO which is a big old sample of ITO with a diameter of 50 mm.

Relative density measurement: the density of sample has been taken by measurement of the green body or ceramic dimensions and weight. The weight has been taken by a high accuracy weighing (accuracy 0,001g). Measurement of length and width or diameter (depend of sample shape) by a calliper. the thickness of the sample has been take on five different points by a thickness gauge.
After these measurements the theoretical density of ITO (Sn 10%) which is ρ_{ITO}=7,12g.cm^{-3} have been divide by the density of the sample to obtain this relative density.

3) Powders Description

The ITO powder has been supplied by Changsha Santech Materials Co., Ltd, China (http://www.santechchem.cn/contents/9/86.html).

In this study we will work with an ITO powder under different aspect of granulation and for some experiment with addition of binder or lubricant.

Producer product information: ITO (indium tin oxide): In_2O_3 90 wt.%; SnO_2 10 wt.%; particle size 20-70 nm; specific surface (BET) 20-40 m^2/g; content of ITO >99,99%; theoretical density 7,12 g/cm^3

To judge of the quality of the powder we have carried some analysis in the laboratory

a) Chemical Composition

ITO properties is influence by the amount of tin oxide; we can find study of ITO with material which contain an amount of 4 to 12 wt% of tin oxide [16] [17].

we have use XRD technique to calculate the amount of tin and indium oxide in our powder:

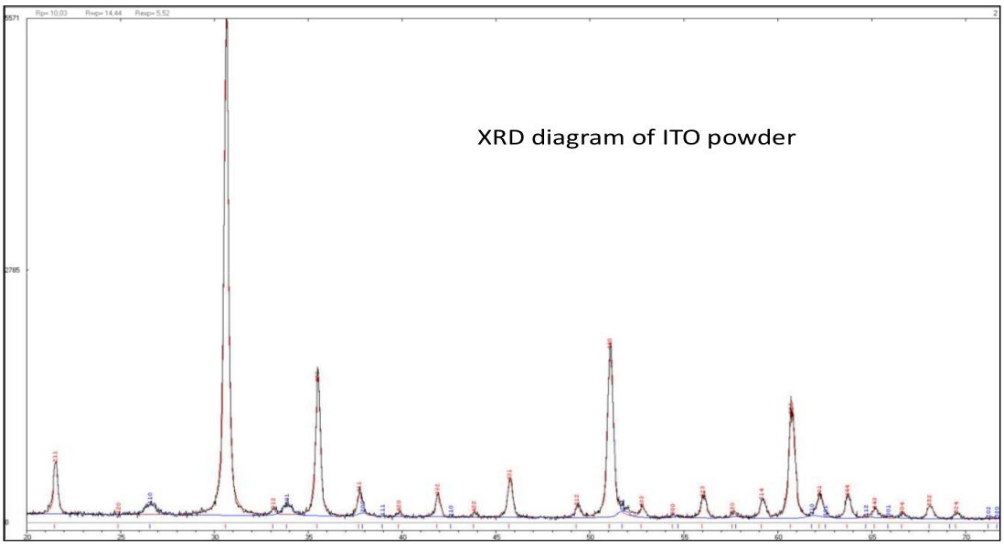


Fig 18 - XRD diagram of ITO initial powder In_2O_3 red SnO_2 blue

In figure 18 we can see that our powder is first constitute by 2 different powder tin oxide and indium oxide. After treatment of the diagram the proportion of tin oxide have been evaluated around 10 wt%.

b) Particle Size

ITO initial powder have a certain polydispersity:

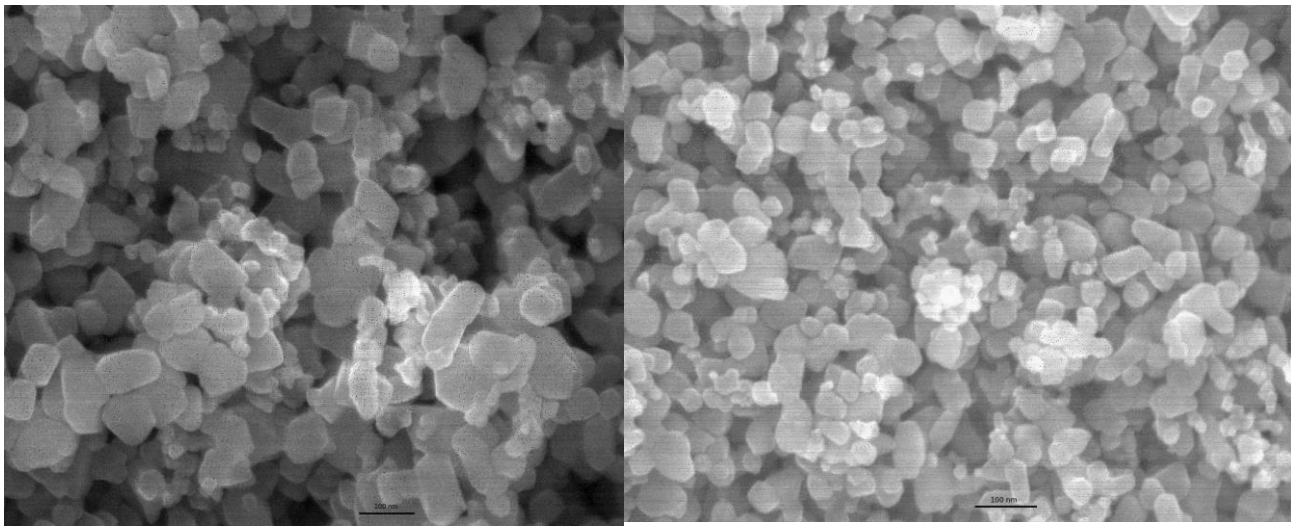


Fig 19 - SEM images of ITO powder.

Figure 19 show two SEM picture with a representative population of particle and in the light of several other picture we have determined that particle size is between 20 to 150 nm with an average of 50 nm.

To obtain a quantitative repartition of our particle we have use a Particle Size Analyser by Laser Diffraction (SALD).

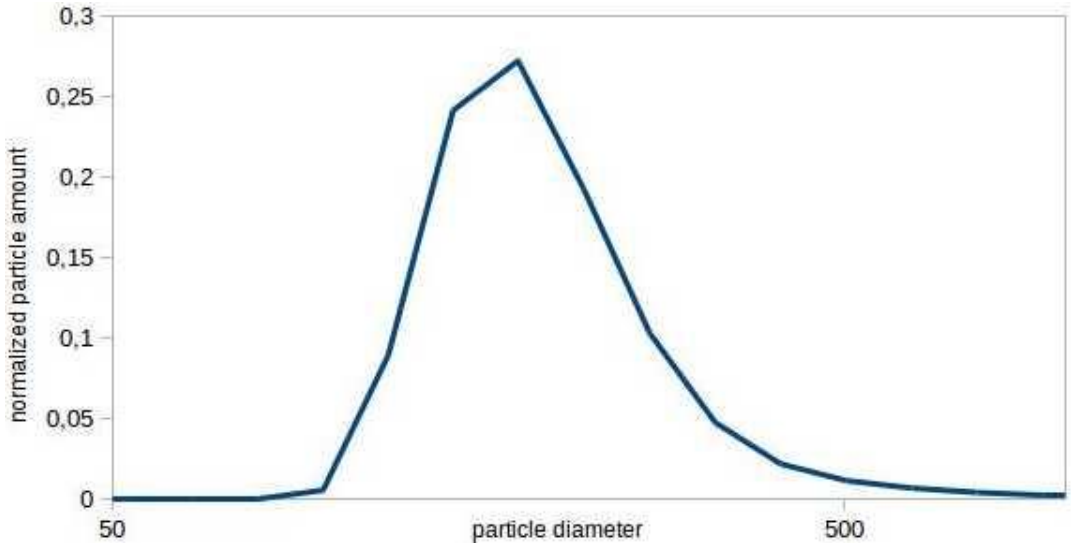


Fig 20 - SALD particle size distribution of ITO initial after 20 minute of ultrasound

Figure 20 show the size reparation of our ITO powder we can see that after 20 minute of ultrasound we keep aggregate of 100 to 500 nm. Previous analysis shows that without ultrasound treatment aggregate are from 100 to 5000 nm.

c) Granulation description

In this study we have work with several different state of this powder:

- ITO initial (non granulated)

- ITO CG (commercial granulation) ITO 1 to ITO 4 provide by the customer already granulated with ITO 1 the biggest pellets around 500 μm and ITO 4 the smallest pellets 4 μm.

- ITO HP (hand processing) ITO granulated by milling of green body of ITO initial compacted at 800 MPa with an agate mortar and pestle

- ITO PVB produce by addition of polyvinyl butyrate in isopropanol solution to ITO initial. 5 minutes of ultrasound then agitation during 2 hours, drying at 80 °C one night, compaction at 800 MPa, milling and sieving of the powder to obtain pellets

The main problem of the utilisation of a powder such as ITO initial is its low powder density and so its compaction coefficient (volume of powder divide by volume of green body). More this coefficient is high more we need a big free volume in the dye to put the powder.

Its why the utilisation of granulated powder is interesting.

Table 1 - Powder density and compaction coefficient of powder

ITO	initial	CG 1	CG 2	CG 3	CG 4	HP 800	PVB
Powder density	0,854	1,726	1,598	1,299	1,613	1,824	1,674
Compaction Coefficient 500 MPa	4,1	2,1	2,3	2,8	2,4	2,1	-

In Table 1 we see that the compaction coefficient of these commercial granulated powder are better than the ITO initial one.

IV) SINTERING CONDITIONS

1)Temperature

To obtain good sintering condition with the material of the laboratory a study of ITO initial sintering with different compaction technologies, temperature, compaction pressure and time of sintering has been carried out.

ITO green compacts with Ø14 mm have been sintered during 4 hours at 1450°C, 1500°C and 1550°C with heating rate of 150 K/hour (Table 2).

Table 2 - Powder density and compaction coefficient of powder

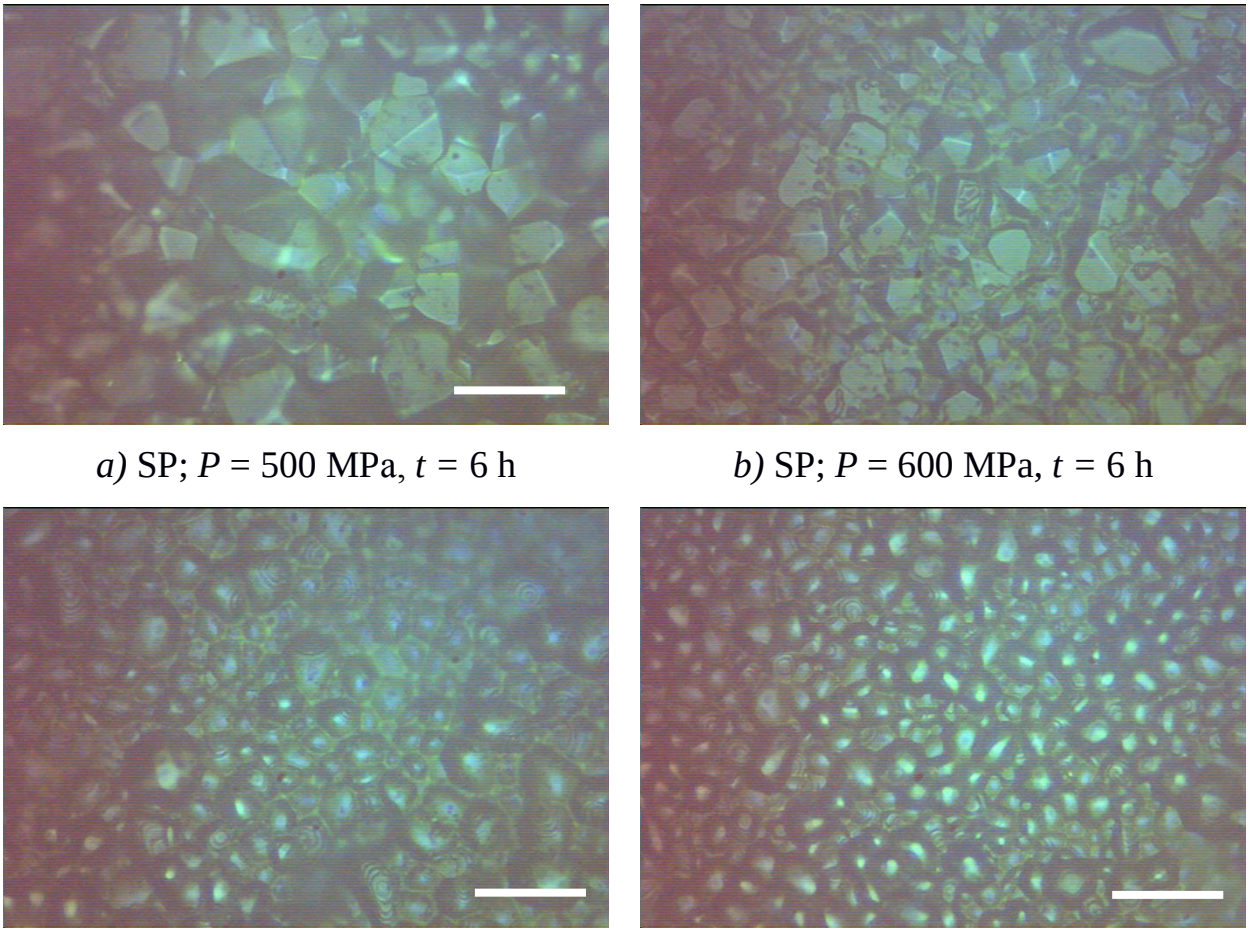
techniques	UP			UP+PUA		CP
T, °C / P, MPa	1450	1500	1550	1450	1550	1550
200	76,9 %	N/A	N/A	76,6 %	N/A	N/A
400	88,3 %	N/A	N/A	87 %	N/A	N/A
500	92,6 %	94,3 %	95,3%	89,5 %	N/A	N/A
600	b	b	b	92,3 %	96,9 %	96,8 %
700	b	b	b	b	b	N/A

The mass loss does not exceed 2%, b – sample cracked after ejection from the dye

The table 2 show that after a compaction at 500 MPa in uniaxial pressing the density of the sintered ceramic increase with the temperature from 92,6 % to 95,3 % for respectively 1450 °C to 1550 °C. for upper temperature the mass loss of the ceramic is more important than 2 %. Concerning CP and PUA pressed at 600 MPa the tendency is the same 1550 °C give the better result and at upper temperature the mass loss is more than 2%

2) Time and pressure of compaction effect

We have studied grain and pore micro-structure of sintered ITO by optical microscopy of ITO samples surface.



a) SP; P = 500 MPa, t = 6 h

b) SP; P = 600 MPa, t = 6 h

c) SP; P = 600 MPa, t = 4 h

d) SP; P = 700 MPa, t = 4 h

Fig 21 – Micro structure of polished ITO, sintered at 1550 °C for different pime after uniaxial pressing with different pressure.

The Figure 21 shows the influence of pressure of compaction and sintering Time on grain growth. We can see that when we sintered during 6 hours (a and b) the grain growth is more important than for 4 hour (c and d). And when we applied more pressure during compaction we reduce grain growth (a vs b and c vs d).

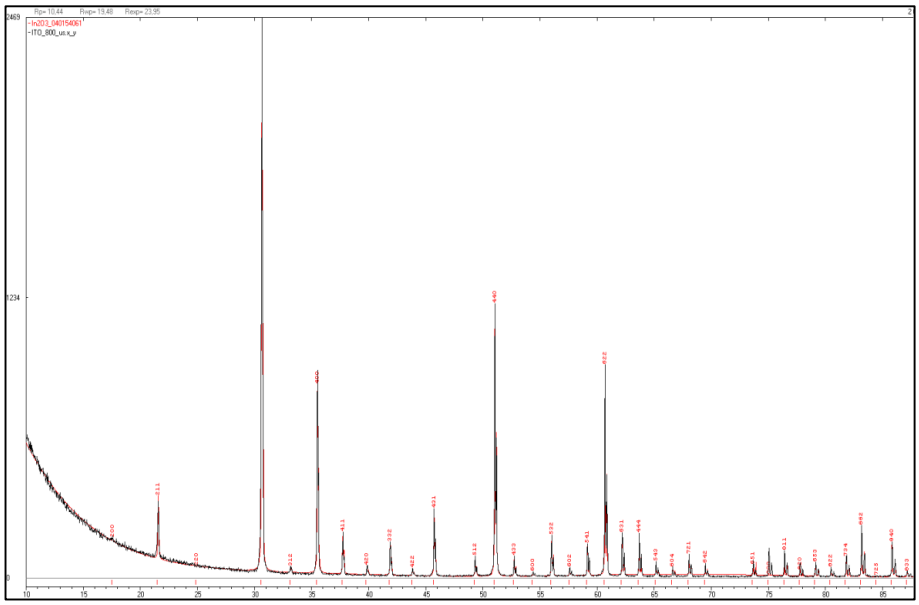


Fig - 22 XRD diagram of ITO ceramic after sintering

To now if we have produce ITO material after sintering we have use XRD technic. The figure 22 show us that the ceramic after sintering contain only one phase, which is indium oxide. The doping of indium oxide by tin oxide just change the existing spectrum of indium oxide by modifying a little the lattice parameter. So we have produce ITO.

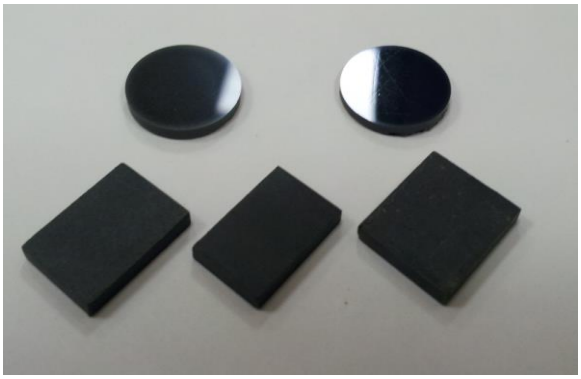


Fig 23 - Photo of different ITO sample after sintering and polishing

The ITO powder is green but after sintering (Fig 23) we can see that the colour is close to the black this is due to the big homogeneity of these sample

The results of this study are close to what we can find in literature:

T. O. L. Sunde et. al. [4] have investigate the sintering mechanism for pure In_2O_3 and ITO Sn 5%. first they see that the sintering temperature of ITO powder increase with the amount of tin oxide. Second they show that up to 1450 °C the sintering is carried by evaporation-condensation mechanism so due to the high partial pressure of tin sub-oxide SnO_2 we can observe mass loss especially when we increase sintering temperature.

S. M. Kim et al [16][17] have study the sintering of ITO powder with different amount of tin dopant 0, 4, 8 and 12 wt% in function of temperature. And they find that the most important shrinkage of the ceramic is at 1550 °C but the temperature where the shrinkage increase mostly depend on the amount of tin so more we have tin more the shrinkage temperature is high. They also show that the grain growth is less important after one hours than after sixteen hours

V) COMPACTION BEHAVIOUR OF GRANULATED POWDER

This part of our study have as aim to find the better compaction parameter to obtain a high dense ceramic.
In this study we have used cylindrical UP (uniaxial press) of 14 mm of diameter, rectangular collector pressing which make sample with 2,2 mm thickness, 14,6 mm of large, spiral collector pressing of 14 mm diameter with 6 passive part. But most of the study have been realized with the rectangular collector pressing.

Find the better condition of compaction is dived on several sub studies. The powder granulation we will test different granulated powders with ITO initial as a referential. The technique of compaction we will use normal uniaxial pressing UP, collector pressing CP, and Powerful Ultrasound Assistance PUA with coupled with Collector Pressing. In this study we will first define what use the compaction behaviour of these powder to compare them, but we also will try to reach highest pressure because as we see in the sintering part of our work pressure reduces grain growth and obviously permit to obtain more compact green body

The sintering process of all the sample has been the same with an exception for sample which contain binder. Sample has been sintered during two hours at 1550 °C with 100 °C/hour for the warming and 200 °C/hour for the cooling. We choose to use two hours because it is enough time to obtain a good sintering of the ceramic but it is also a gain of energies and time for the laboratory. When the optimal condition of pressing will be find, the laboratory will search for the optimal sintering condition. For example, it has been proved that the utilisation of PUA produce a surface activation and reduce the sintering temperature [15].

1) ITO initial

ITO initial have been compacted by uniaxial pressing at 550 MPa using uniaxial press to obtain its compaction curves. The method of cyclic loading-unloading every 50 MPa has been applied taking into account elastic deformation of punch (Hookean deformation); elastic and inelastic (irreversible) components of deformation of powder.

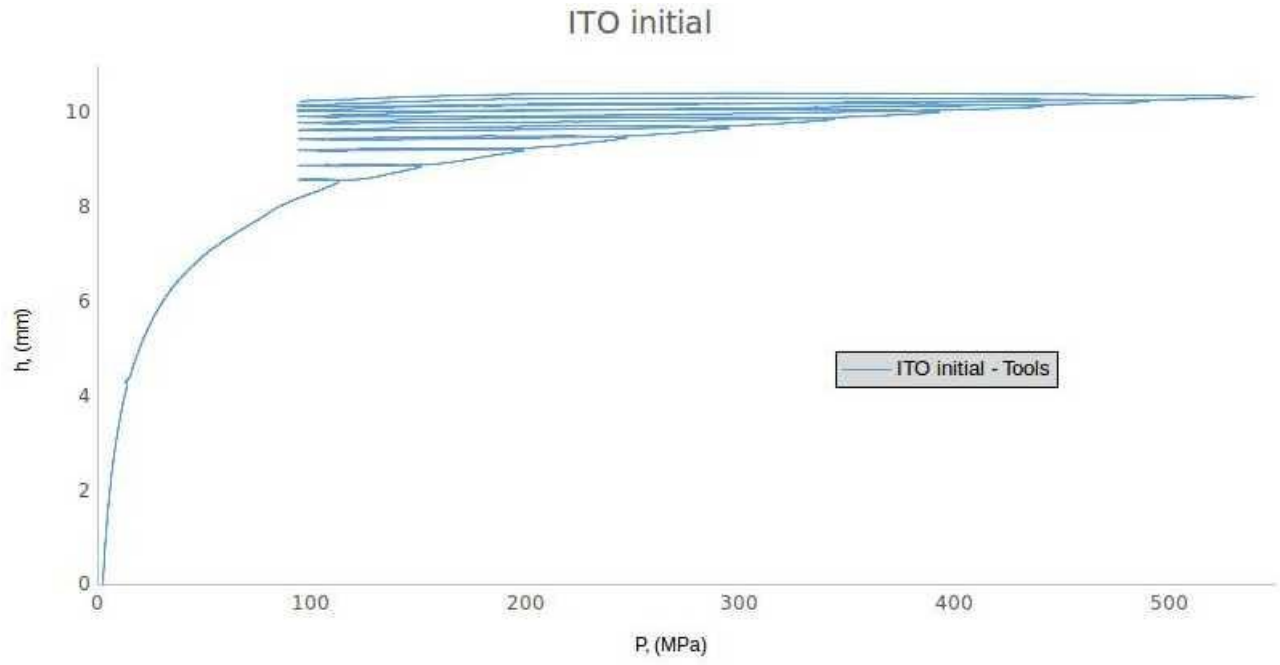


Fig 24 - Displacement of the punch in function of pressure for ITO initial with the method of cyclic loading-unloading every 50 MPa

From figure 24 we can see the compaction of ITO initial (the tools deformation have already been suppressed). figure 24 allow us to extract two important curve: the elastic after effect of ITO in function of pressure, and the compaction curve of ITO.

a) Compaction curve of ITO initial

The compaction curve of ITO initial has been extract from fig 25 by taking the relative density after the unload of each load unload cycle, like that we take in account the elastic after effect of the powder.

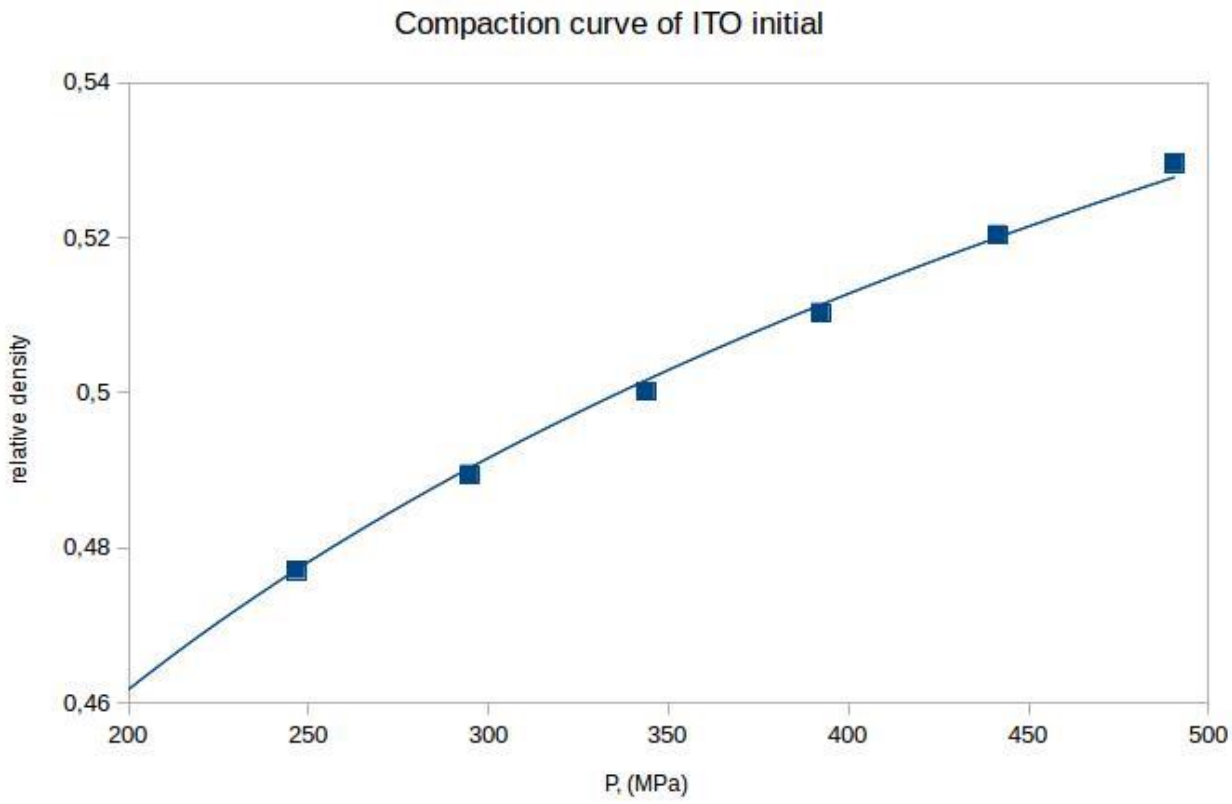


Fig 25- Compaction curve of ITO initial

Like we explain it before the aim of this work is to increase the densification and the homogeneity of our ITO ceramic its why we use improvement of the uniaxial pressing technologies such as collector pressing or powerful ultrasound assistance or both. This curve permits us to obtain the coefficient of the compaction equation:

$$\rho = b \cdot ln\left(\frac{Pw}{Pcri}\right) + 1 \qquad (1)$$

B dimension less coefficient, P_w work pressure, P_{cri} critical pressure, ρ density relative

Table 3 – Coefficient b and critical pressure of ITO initial

ITO	b Coefficient	Critical pressure
intitial	0,074	299 GPa

The critical pressure is the theoretical pressure where our green body have 100% of relative density. We can see that for ITO this coefficient is very high.

b) Elastic after effect of ITO

The elastic after effect is calculate by the difference between the relative density of the green body at the beginning of one load unload cycle and the relative density after the unload (at 100 MPa).

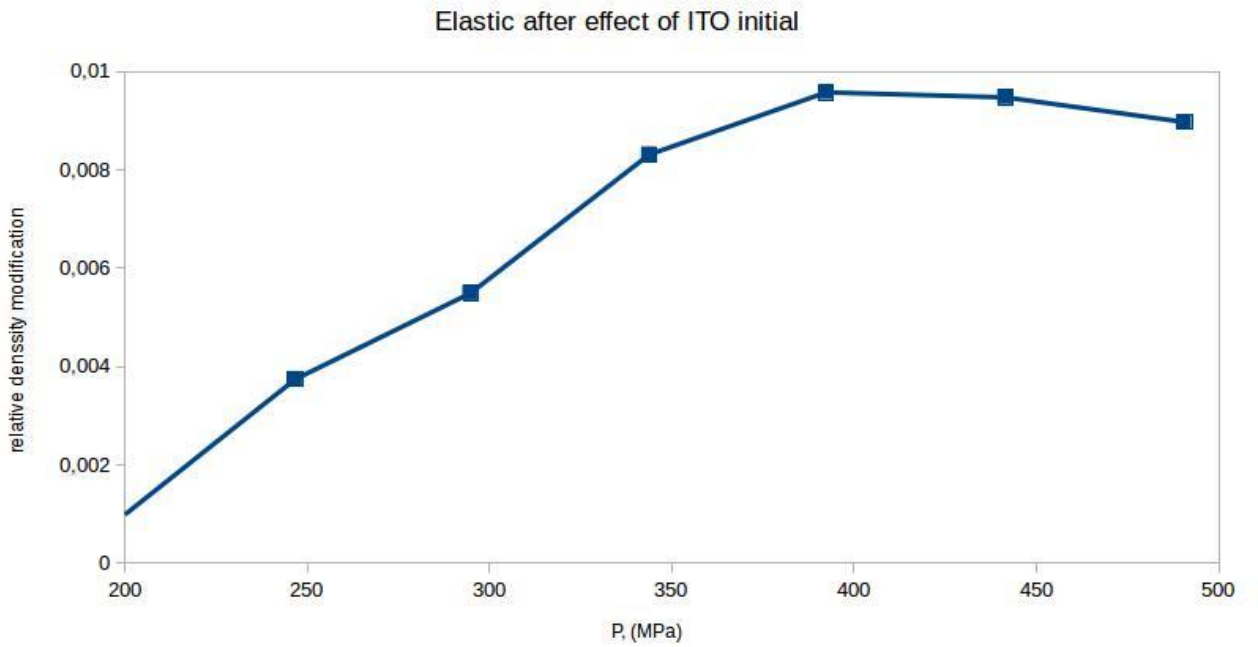


Fig 26 - Value of the elastic aftereffect of ITO in relative density in function of compaction pressure

In Figure 26 we can see that the ITO powder have big elastic aftereffect, approximately one per cent of relative density when we stop to applied pressure. This big elastic after effect is a problem to work at high pressure because it creates tension in the green body and these tension can lead to crack.

c) Comparison between UP and CP

ITO initial have been compacted by uniaxial pressing at 500 MPa using collector pressing and at 300 MPa using uniaxial press. The compaction curves of these greens body have been taken during the experiment.

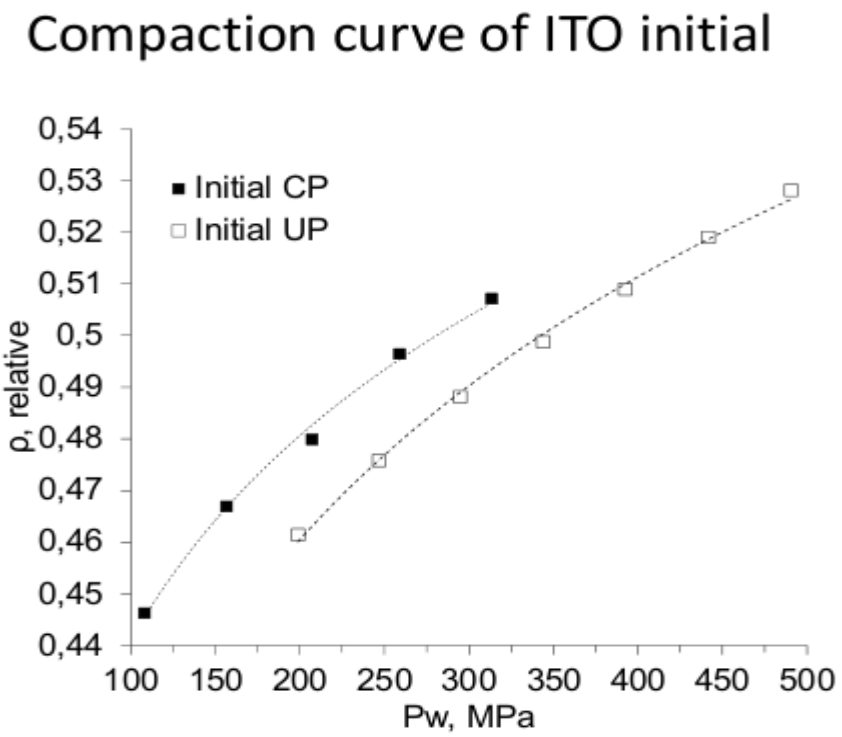


Fig 27 - Comparison between collector pressing and uniaxial pressing compaction curve of ITO initial

In figure 27 we can see that the collector pressing technique give a better compaction when we work at low pressure but after calculation the critical pressure of CP is 516 GPa so two time more important than UP critical pressure but we work at pressure which do not exceed 1 GPa so CP is better than UP. Otherwise the collector pressing allow us to work at more high pressure because of the reduction of the elastic after effect stresses during the ejection of the green body.

due to the very high compaction coefficient of ITO initial we have try different way of granulation of the powder first of all to decrease this compaction coefficient but also to see if we can obtain a powder with a better compaction behaviour that ITO initial.

2) ITO CG 1 to 4

a) ITO CG compaction and sintering

Our supplier has send the ITO initial powder but also 4 ITO powder granulated. These granulated powder have the same characteristic than ITO initial. The supplier does not give us the condition of granulation of these powder he explain us that they have been produced by dry granulation technique.

These four powders have been named regarding the size of their pellets, with ITO 1 the thickest and ITO 4 the finest.

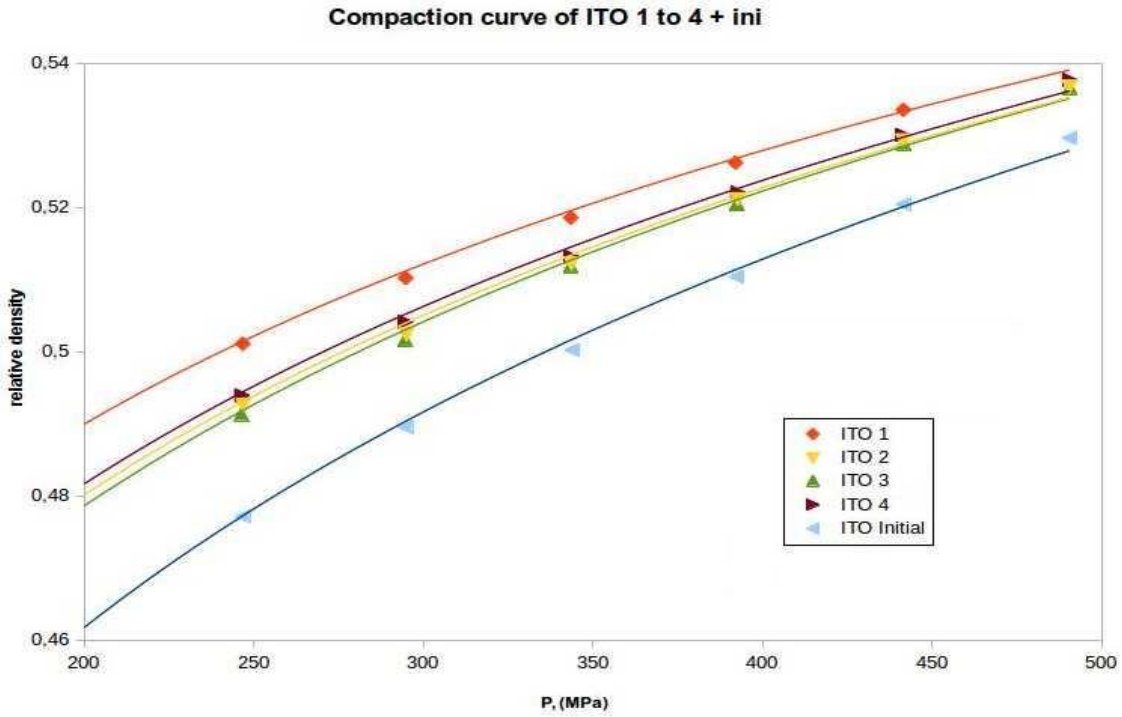


Fig 28 - Compaction curve of ITO 1 to ITO 4 and ITO 1 curve realized in UP using 500 MPa

Table 4 - Coefficient b of equation 1 and critical pressure of ITO CG and initial

ITO	b Coefficient	Critical pressure
1	0,055	2243 GPa
2	0,061	949 GPa
3	0,063	793 GPa
4	0,061	1024 GPa
initial	0,074	299 GPa

In the figure 28 we can see that at low pressure the compaction of ITO 2, 3 and 4 are more or less the same. ITO 1 have the better compaction in this scale of pressure and ITO initial give the less compaction.

In the table 4 we can see that the critical pressure of granulated powder is way more important than the critical pressure of ITO 1.

Even if the compaction seems better using these granulated powder we cannot judge it without sintering. To reduce the number of sample we used only ITO 1, 3 and initial. ITO 2 and 4 looks mot necessary because ITO 3 have the same compaction result and a smaller critical pressure.

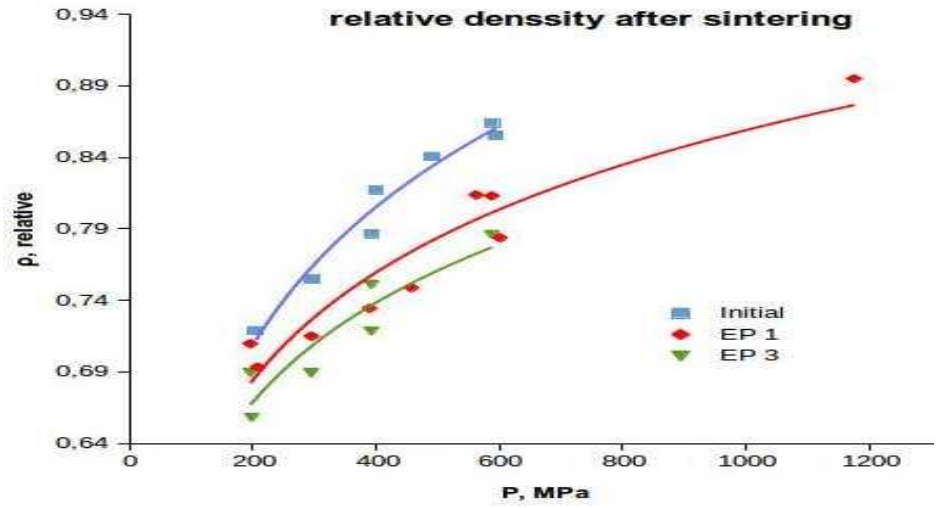


Fig 29 - Relative density after sintering in function of compaction pressure

figure 29 show us that after sintering ITO initial give more compact ceramic than granulated powders this is probably due to the non-homogeneity of the compaction

b) ITO CG speed of sound

To illustrate the inhomogeneity of the green body, we have taken some measurement of ultrasound's speed in the green body.

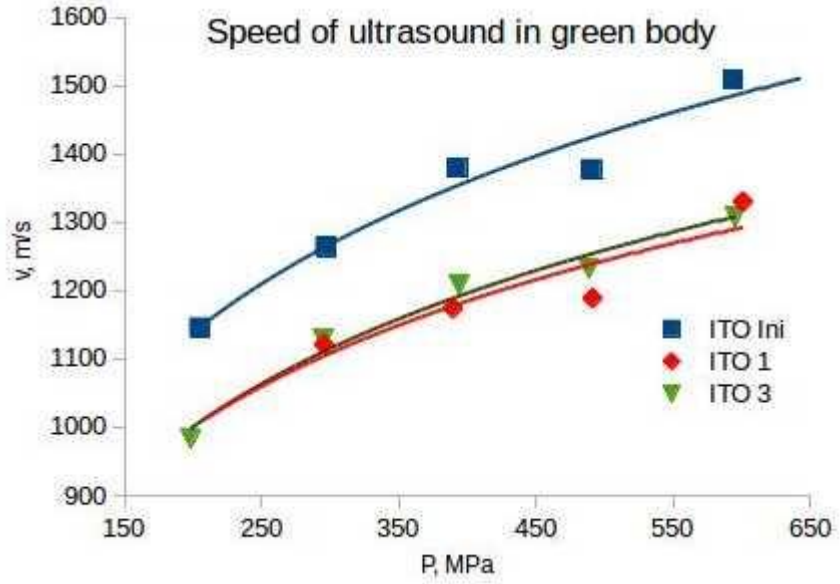


Fig 30 - Speed of sound in function of pressure of compaction in green body of ITO 1, 3 and initial

The figure 30 show us the speed of ultrasound in green body in function of the compaction pressure. The speed of sound in green body is function of speed in the bulk material, the coordination number of these particle and the homogeneity of the green body. Between these 3 powder the bulk speed is the same. In green body the coordination number depend mostly on the relative density of the green body and the size of the particle it is the average number of particle in contact with a particle. If we take a look to Fig 30 the average density and theoretically the coordination number of ITO initial is less important than for granulated powder. In figure 29 can see that ITO initial have a better sound's speed than granulated powder. So we can expect that the homogeneity of ITO initial is very good and this good parameter eclipse the little ad result in coordination number and sintering prove it.

3) ITO initial HP

For this part of our study we have make our own granulation. We have compressed ITO initial at 800 MPa and after we have milled the green body by an agate mortar and pestle. This new powder has a lower compaction coefficient.

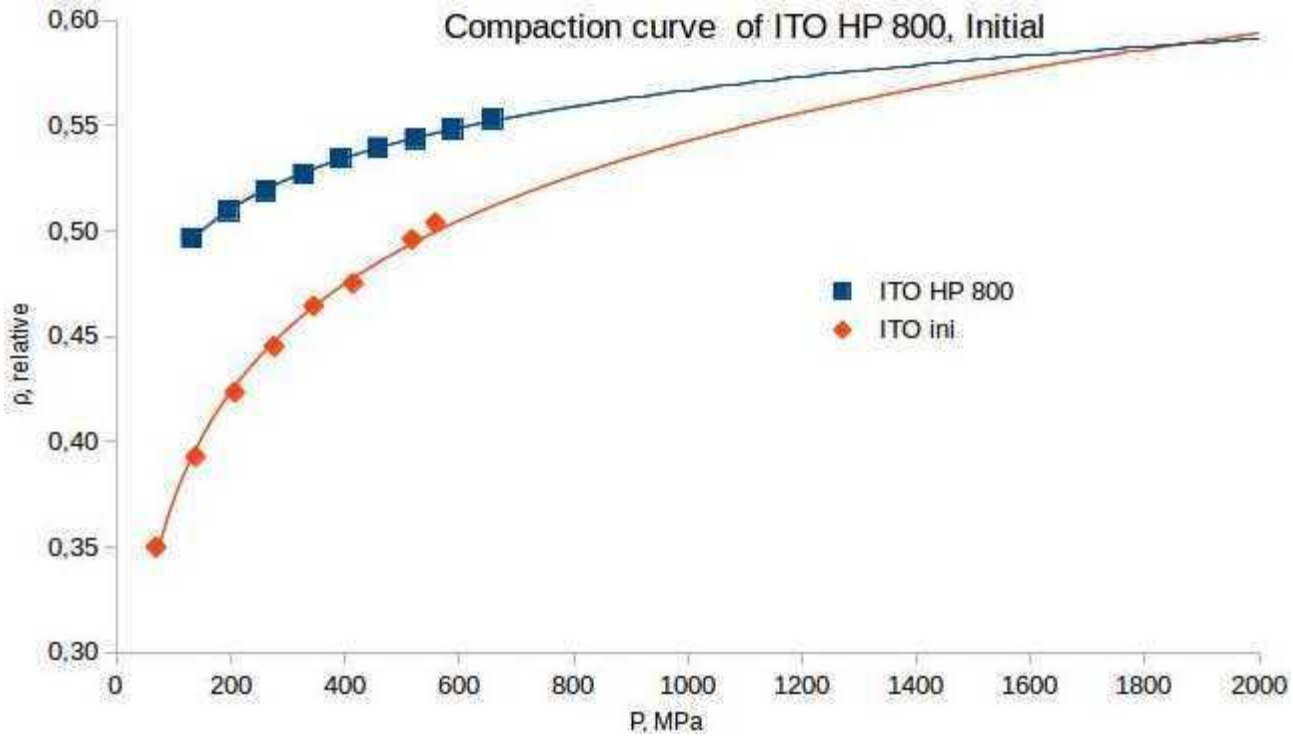


Fig 31 - Compaction curve of ITO initial and ITO HP 800 realized by collector pressing technique

In the figure 31 we can see the compaction curve of ITO HP 800 and ITO initial. As we can see on these curve the compaction of ITO HP is more efficient than ITO initial unless 1,9 GPa. In this press form the compaction curve of ITO initial lead to a critical pressure of 487 GPa and for ITO HP 800 the critical pressure is 2306 GPa. So like for ITO CG this powder seems promising during the compaction.

Like for the ITO CG we used sintering to check the homogeneity of the compaction.

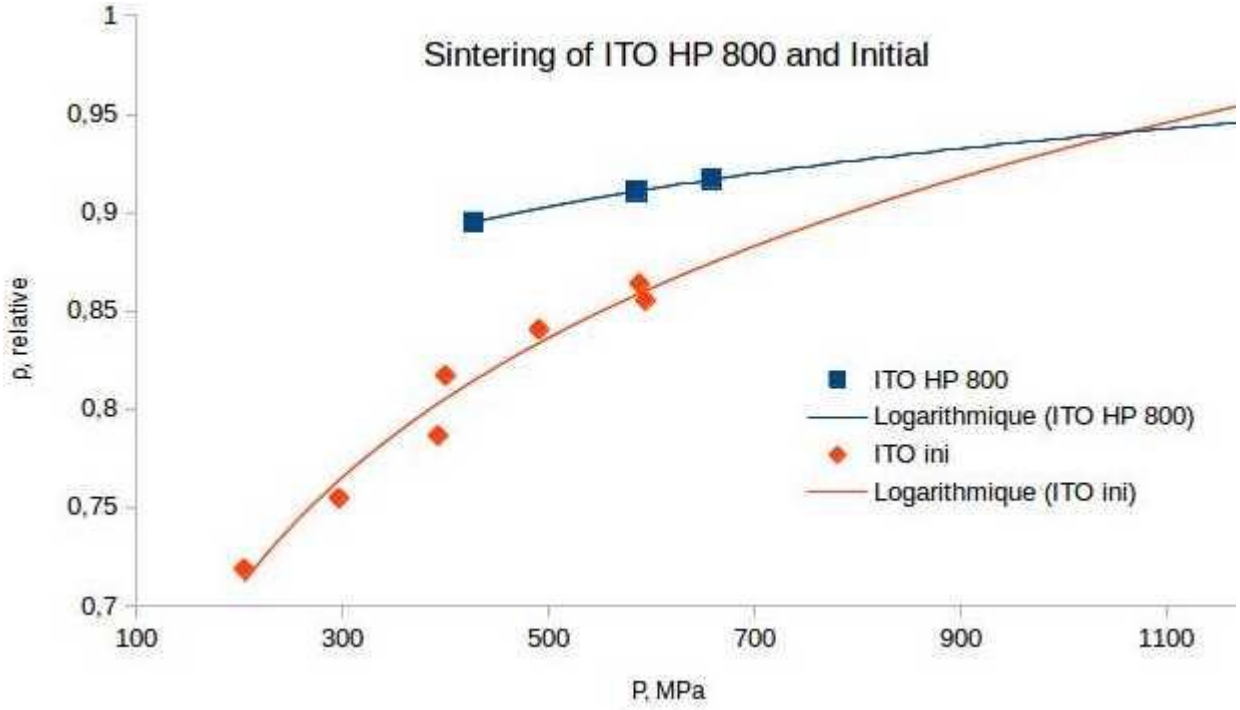


Fig 32 - Sintering result of ITO initial and ITO HP 800

In figure 32 we can see that the ITO HP give us better result than ITO initial. With ITO HP 800 we almost reach 91,6 % of relative density when it has been pressed at 650 MPa. The ITO sample crack after 600 MPa, and at this pressure we can make sample of relative density of 91,0 % and 86,4 % for respectively ITO HP 800 and ITO initial so 3,5 % less compact.

In the other hand if we follow the extrapolation of the trend line, at 1,05 GPa the ITO initial powder become better than ITO HP, if we find a way to increase the pressure of compaction of ITO initial this powder will be better than ITO HP.

4) ITO PVB

After all these experiment we does on dry compaction the main problem seems that we cannot use high pressure. In this short part of our study we tried to use plasticizer to increase the compaction of the powder and reduce the stress in the green body during its expulsion from the dye.

a) ITO PVB elaboration and compaction

We have elaborate the powder with PVB of good purity dissolute in isopropanol. After adding the powder, we applied 5 minutes of ultrasound to the melting then agitation during 2 hours, drying at 80 °C one night. After drying the powder, 4,5 g of it has been compressed at 800 MPa in a spiral collector pressing press form which is big enough to contain it. This compaction in this specific press form was an important step because first the powder has a very bad powder density; second it sticks on the steel of the press form. Filling our little collector pressing with the original powder was too difficult to make proper sample of 1 g. After compression we have been very astonish to obtain a very strong sample without any crack and weakness. Break this sample to milled it have been hard.

After milling and sieving the powder to avoid the big pellets we have make some sample:

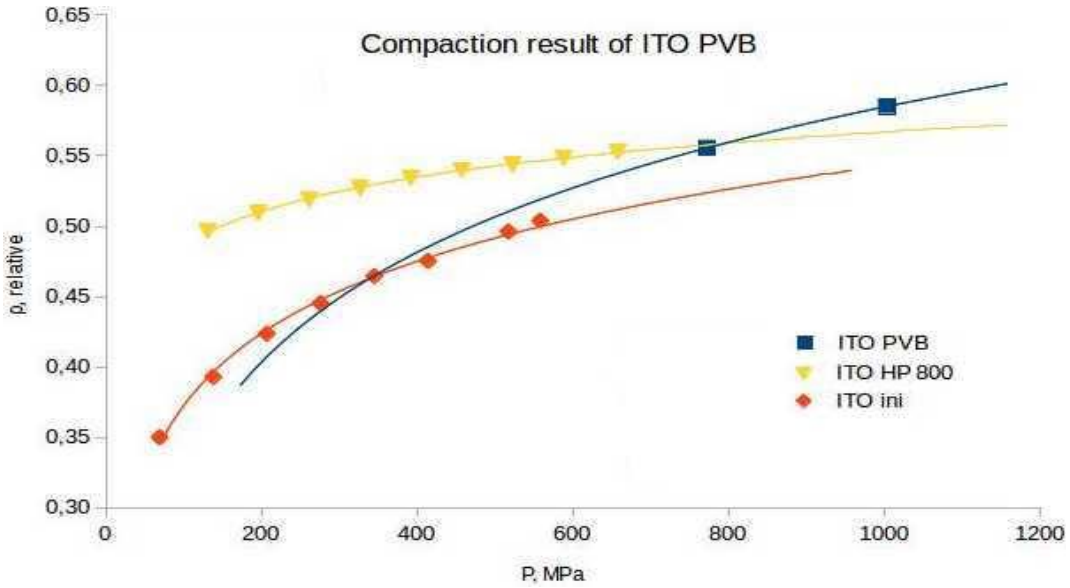


Fig 33 - Compaction curve of ITO initial and HP 800 and two sample of ITO PVB

In the figure 33 we can see the compaction curve of ITO initial and ITO HP 800 which are our two best powder. In blue we see the two sample of ITO PVB we made, first of all we can see that the compaction looks way more efficient and second we can make sample at high pressure like 1 GPa.

b) ITO PVB debinding

Another part of the consolidation of binded green body is its sintering with debinding steps [18]. To debinded our ITO sample we follow the literature data. J.Y. Yun et al. [19] Have study the debinding of polyvinyl butyral with different concentration on $Pb(Mg_{1/3}Nb_{2/3})O_3$-$PbTiO_3$ ceramic consolidation. They have study three case of sintering: sintering without debinding; debinding during sintering operation; and debinding, cooling, and sintering.

At the end this research show that the better process is the third one the micro structure of the ceramic is way better. Concerning the amount of carbon contained by the ceramic plasticized by 3 wt% of PVB, for the debinding during sintering the amount of carbon is 0,11 wt% and for debinding cooling sintering 0,019 wt% of carbon.

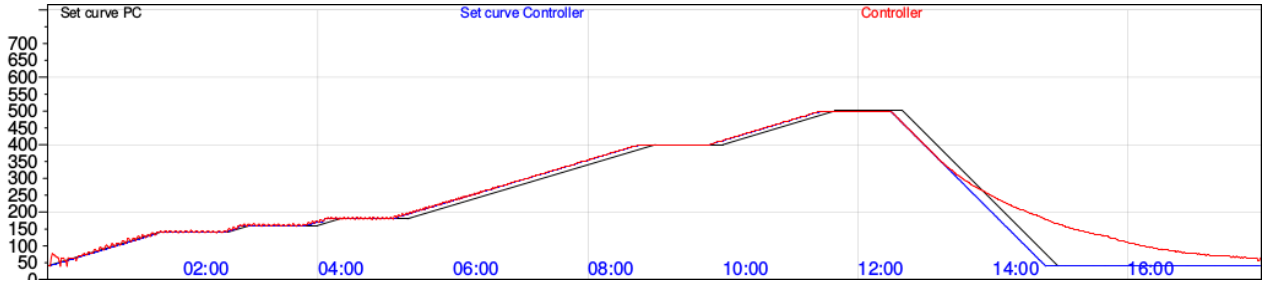


Fig 34 - Debinding thermal program with temperature in function of time

As you see in figure 34 we have decided to use the debinding cooling sintering process. This graph shows us the debinding operation of ITO PVB. This debinding is composed by five step of 1 hour at 140, 160, 180, 400, 500 °C during the increasing of temperature we use 60 °C/hour and for cooling 200 °C/hour. In the study of J.Y. Yun et al. [19] the step has been maintained during 5 hours but the thickness of hour sample is approximately 2,15 mm and in the study the sample are 5 time more big so we assumed that one hour will be enough.

c) ITO PVB sintering

The first sample have been first debinded and after sintered, like that we have taken its dimension and weight. The mass loss of the sample after this debinding step have been proximately 2,11 % so we have well burn the binder.

after this the sintering have been done following the usual condition; 1550 °C during two hours, 100 °C/hour for heating, 200 °C/hour for cooling.

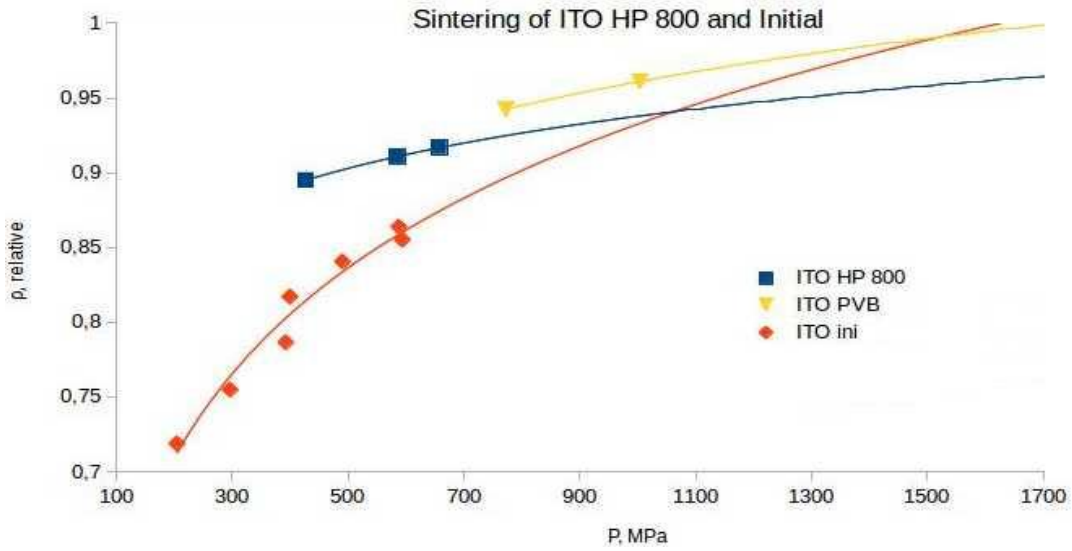


Fig 35 - Sintering of ITO initial, HP 800 and ITO PVB

In figure 35 we can see the sintering result of ITO HP 800, initial and PVB. In this graph we can see that the ITO PVB powder give us also very good result after sintering even if after 1550 MPa the extrapolation show that ITO initial become better. At 1550 MPa the density of the ceramic will be almost 99%. more than this ITO initial have a poor compaction coefficient and green body crack after 600 MPa, ITO PVB have good compaction coefficient and can be used at 1 GPa and maybe more.

Finally, this study of granulated powder show use that ITO granulated can give us better result than ITO initial. In the other hand if we find a way to press this powder at high pressure the extrapolation of the trend lines says that around 1,5 GPa this powder will be the better one. For now, our better powder is ITO PVB but due to the presence of the binder even after debinding we probably keep trace of carbon. The laboratory does not have appropriate device to check the quantity of carbon trace. XRD do not have enough resolution, trace of carbon signal will be confound with the background noise. For EDX analyses the amount of oxygen in ITO will literally hide the carbon trace because of the proximity of their Kα bands.

5) Future collector pressing dye and wave guide

In this part we will present you a new project of the laboratory regarding this previous study of granulated powder; the main problem of the consolidation of the ceramic is the work pressure. If we want to produce ceramic of good density it is essential to work at high pressure at lead more than one GPa. The main problem of the compaction of ITO is the inhomogeneity after compaction. The utilisation of collector pressing or PUA have a positive action on it they allow use to work at more high pressure.

Thus the laboratory has decided to invest in a new dye who combine with a good efficiency collector pressing and PUA:

Fig 36 - Representation of the new dye and wave guide for the combination of collector pressing and PUA

In figure 36 you can see the future dye and its future wave guide the collector pressing part is approximately in the same geometry than our old rectangular collector pressing with two passive part. An amelioration can be see one the left picture the pic on the bottom are there to limit the movement of the punch because he will be fixed on the passive part.

The main point of this new device remain in the utilisation of collector pressing with ultrasound.

TASK FOR THE SECTION

“Social responsibility”

Student:

Group	Name
4БМ4И1	**Jacquemin Manoel francois marie**

Institute	Level of study	department	direction
High technologie	Master degrees	**Nanomaterial and nanotechnology**	150100 material science and techno0logy of material

Task for “social responsibility”
Explain the global context of your research Include the global issue which motive these research Exposed the global benefit for the industry, the word or the humanity, that your work will theatrically produce

Date of issue of the task	**-**

Consultant issued this task:

position	name	Academic degree	signature	date
Docent	**Lyamina G.V.**	**Ph.D. of chemistry**		

Student which has taken the task

group	name	signature	date
4БМ4И1	**Jacquemin Manoel francois marie**		

VI) SOCIAL RESPONSIBILITY

Indium is a metal of the p group with a chemical behaviour close to aluminium and gallium. Indium metal is rare we find it in very small quantity in zinc mine. Since the end of the XX centuries the utilisation of this material have increase exponentially every year. The apparition of new technologies such as LCD screen, photovoltaic cells, touch screen, LED (optic fibber use InP) ... and the wide utilisation of them explain the infatuation of its utilisation.

The price of indium metal has seen an enormous increment between 2001 and 2010.

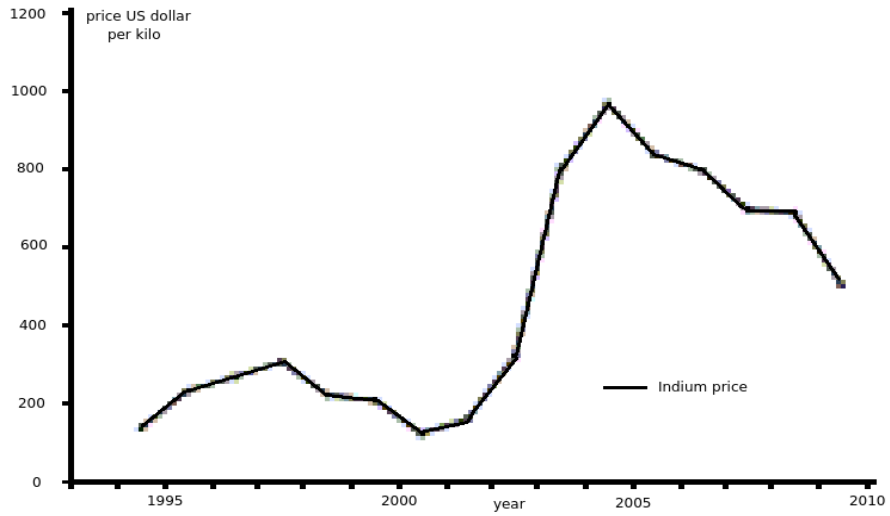


Fig 37 - Price of indium in US dollar per kilo evolution in 15 years [20]

In figure 37 we can see that the indium price increase from 100 to 1000 dollars between 2001 and 2005. The multiplication by ten of the price is mostly due to the apparition of the new technologies and the depletion of the word global extracted stock. After 2005 the beginning of the global crisis leads to a decrement of the price of indium. This is due to the slowdown of new technologies production compare to a period with no crisis. In 2010 the price was around 600 dollars. Actually, indium is even more cheap, the price in june 2016 is 240 dollars but last year he was on average two time more important with a maximal of 760 dollars and a minimum of 140 dollars [21]. Price is instable.

Even if these last years the price of metallic indium have decrease the planetary stock of indium is the root of the problem. in 2009 the word consumption of indium was 1 210 tonnes for the year [22] and the word production was around 600 tonnes. The rest is supplied by the recycling industries but the process is expensive and polluting. The global stock of indium has been estimate around 11 000 tonnes in 2009 so its estimate that in 20 years the indium resources will be finished.

In this context the price of indium in the long term will increase. Our research has for aim to replace the isostatic pressing technique which is expensive by the conventional uniaxial press (with some improvement) which is way more cheap. This replacement will allow the industrial to produce target for RF magnetron spluttering with a production cost inferior.

So our research will reduce the price of manufactured product made with ITO. Like that technology which use ITO will stay affordable more time before the price and the rarity of indium make these products too expensive for normal customer.

TASK FOR THE SECTION

“Financial management, resources and resources efficiency”

Student:

Group	Name
4БМ4И1	**Jacquemin Manoel francois marie**

Institute	Level of study	department	direction
High technologie	Master degrees	**Nanomaterial and nanotechnology**	150100 material science and techno0logy of material

Task for “financial management, resources and resources efficiency”
Make a comparative study of the material used in this research. Taking account of all characteristic of the material which can interest the industrial who want to put in production this project

Date of issue of the task	**-**

Consultant issued this task:

position	name	Academic degree	signature	date
Docent	**Lyamina G.V.**	**Ph.D. of chemistry**		

Student which has taken the task

group	name	signature	date
4БМ4И1	**Jacquemin Manoel francois marie**		

VII) FINANCIAL MANAGEMENT, RESOURCE AND RESOURCE EFFICIENCY

For this economical part we have compare the price of ITO powder in different supplier website. Our study on powder take in account the price and the purity of indium oxide powder tin oxide powder and the already prepared indium tin oxide powder.

Table 5 - Comparative table with price purity and size for SnO_2 In_2O_3 and ITO powders on sale by two different supplier

material	In_2O_3			SnO_2			ITO (10% SnO_2)		
supplier	Price ($/gr)	purity	Size	Price ($/gr)	purity	Size	Price ($/gr)	purity	Size
Sigma Aldrich	21,8	99,998	< 1 µm	17,7	99,99	< 1 µm			
Sigma Aldrich	11,4	99,99	< 1 µm				31,36	99,99	Nano <50 nm
Sigma Aldrich	18	99,9	Nano <100 nm	14,8	99,9	Nano <100 nm			
Alpha Aesar	12,7	99,5	Nano <50 nm	7,9	99,5	Nano <100 nm	11,4	99,5	Nano 17-28 nm
Alpha Aesar	10,9	99,99	325 nm	22,8	99,996	22 nm	16,8	99,99	325-500 nm
Alpha Aesar	9,4	99,9	325 nm						

In the table 5 we can see several different price for our powder. First of all, we can notice that sigma Aldrich is two time more expensive than alpha Aesar. We can see also that the purity of the powder is an important factor of price for example Sigma Aldrich sell an In_2O_3 powder with 99,998% of purity two time more expensive than 99,99%. the size of the powder has also a certain influence on the price. Alpha aesar sell an In_2O_3 powder with a nano-size (under 50 nm) but a bad purity 99,5% more than a powder of 325 nm with a purity of 99,9%.

Theses ascertainment are the same for the SnO_2 powder, Alpha Aesar sells this powder with a purity of 99,5% and a size under 100 nm three time less than a powder with 99,996% of purity and an average size of 22 nm.

Last observation that we can make is that the ITO powder is generally more expensive than the two different powder after a melting of In_2O_3 and SnO_2 at respectively 90 and 10 wt%. This mean that if the customer is ready to make the mix by his own, powder can be more cheap. To make the mix laboratory need some extract device which will increase the initial investment.

The price of the powder depends mostly on the customers need, if they need ultra-pure powder or just regular one the price increase so much. For our application the nano-sized of the powder is important because is permit use to use ultrasound with more efficiency and second point the sintering is more efficient with NPs powder due to their big surface energy. So the utilisation of nano calibrate powder increase again the price of our ITO powder,

The limit of this study is the fact that we carried out this analyse on the price of the powder packed in 5 to 25 g if this analyse lead to an industrial application of the technologies, the mass of powder will approach more the kilo than little weight as that. Generally, these amount of expensive material are sell by wholesaler and they are used to negotiate the price of the material regarding the asked quantity.

CONCLUSION

During this internship several parameters of the consolidation of ITO powder has been studied.

1 the sintering temperature we find that the temperature which lead to the better shrinkage without modified to mush the chemical composition of the ceramic is 1550 °C.

2 The influence of the compaction pressure and the sintering time on the grain growth have also been studied. To avoid as much as we can grain growth we need to compact powder with the highest pressure we can. Second 4 hour of sintered lead to less grain growth than 6 hours.

3 During the study of ITO sintering we also study which is the highest pressure we can use for compaction of the raw powder with our three different technologies. When conventional uniaxial pressing does not work after 500 MPa we can reach 600 MPa using Powerful Ultrasound Assistance or collector pressing technique.

The second part of the study is about powder granulation. We try to improve the characteristic of the ceramic by replacing the raw powder by granulated powder.

4 We find good compaction result when we use ITO hand processing at 650 MPa we obtain 91,7% of density which is almost 5% better than with raw powder. And with this powder we can work at 700 MPa so 50 MPa better than ITO initial.

5 When we use the powder with plasticizer we obtain the better result of the study. First of all, thank to this powder we can work at 1 GPa and probably more. At this condition of pressure after sintering the PVB ceramic give us sample with 96,1% of density this result is the better we obtain in our study. First because the compaction of other powder at such pressure lead to sample with cracks which are not exploitable, and if we follow the trend curve of these other powder at 1 GPa the PVB powder still give us the better result.

This conclusion lead us to some objective to finish these research they are possible way for attain our goal:

6 To obtain ITO ceramic of 99% of density our estimation thanks to trend line of powder density, is to use ITO PVB or ITO initial at 1,5 GPa. The problem is we need to find some way to increase the compaction pressure without crack of the green body. In our study we show that the PUA and collector pressing have a positive action on this parameter. The future dye of the laboratopry will maybe increase again this positive action but for now the better increment of compaction pressure come from the utilisation of plasticizer.

7 Another point is that all green body depending on the material and the compaction technologies have different optimal sintering condition. If we find these condition, we can increase the density of the ceramic using the same compaction pressure. For it we need to find the good condition (powder type, press technologies, pressure) of compaction. When a good green body is find we need to carried out a study of sintering specifically focus on this type of sample.

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