ITO nanoparticles for Transparent Conductor Coatings

Nowadays, a wide range of high technology applications such as smartphones, tablets, solar panels or touchscreens uses thin conductive oxides based in indium-tin oxide. In this way, one of the applications developed in the project based on the importance of this metal in the market was focused in ITO.

Based on indium was one of the focus for recycling from FPDs, in addition to work in the recycling of this metal during the project, one of the targets was produce ITO nanoparticles and develop applications based on ITO but working with nanoparticles in order to assess their potential in current technologies of TCOs production and potential advantages.

In addition to the production of the nanoparticles, different approximations were followed in order to develop applications, here it is summarised the results obtained during the project. Additional information is provided in the following sections quantifying the results obtained:

- **ITO nanoparticles were synthesised by Lurederra and TECNAN in different production scales by Flame Spray Pyrolysis technology**. The results of the characterisations showed that these nanoparticles are according to requirements for final application. In addition, **these results were replicated when using simulated recycled materials**, validating the process for using secondary materials as raw materials of the process.
- Transparent coatings were produced directly from dispersion of nanoparticles and spraying reaching transparent coatings with more than 90 % of transparency.
- **ITO nanoparticles were used to produce ceramic targets** which were suitable for developing sputtering processes. Although still additional improvements are needed, the results in the project showed that nanoparticles could need lower pressures and lower thermal treatments to achieve similar densities to targets produced with conventional powders.
- Within the project, **thin conductive films have been obtained from nano-based ITO targets by Plasmaquest** partner using their proprietary HiTUS (high target utilisation sputtering) plasma deposition system, **showing promising results in terms of resistivity and transparency (~95%) when compared with coating obtained from commercial targets.** Additional improvements in ITO targets could result indeed in better results in coatings performance.

<u>CHARACTERISATION OF ITO NANOPOWDER</u>

- Blue powder

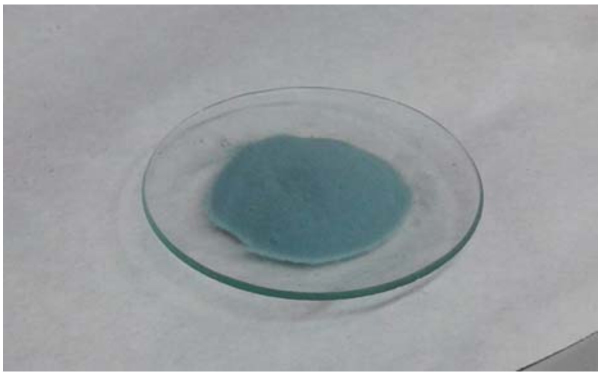

Figure 1. ITO blue powder produced from surrogate recycled materials

- Specific Surface Area of 71.98 m^2/g.

- Cubic phase.

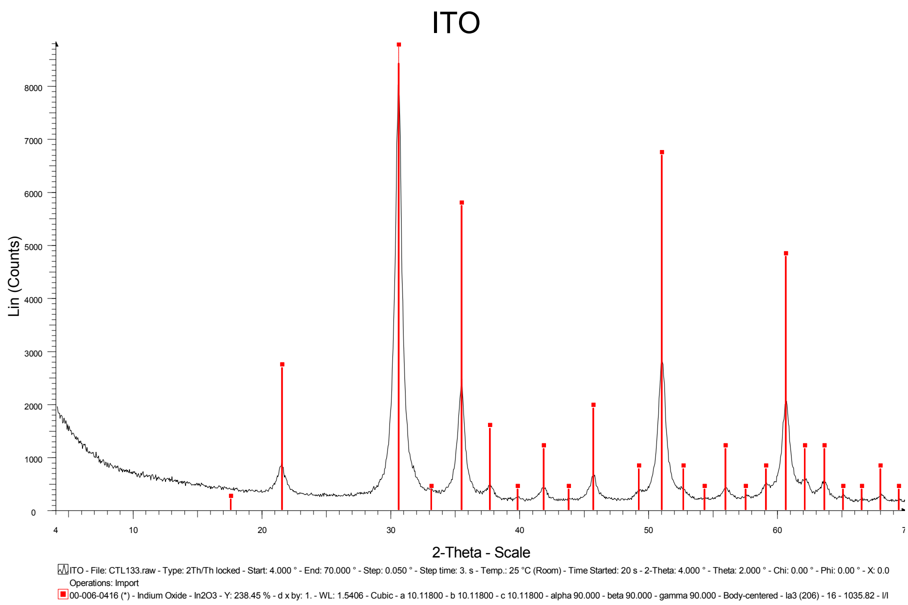

Figure 2. XRD Diffractograme of ITO

- TEM microscopy

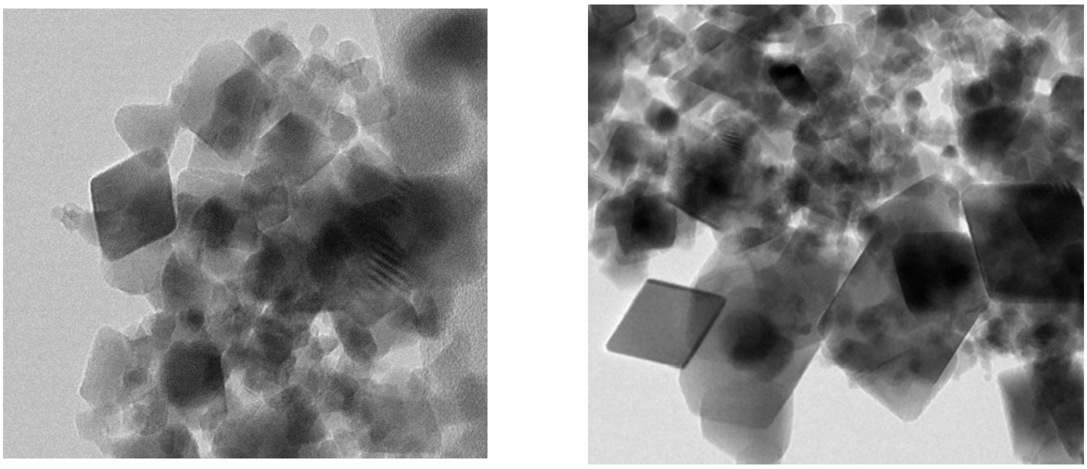

Figure 3. TEM images of ITO

- Stable dispersion of ITO nanoparticles (15% in isopropanol) for thin film spraying with average size of 29 nm.

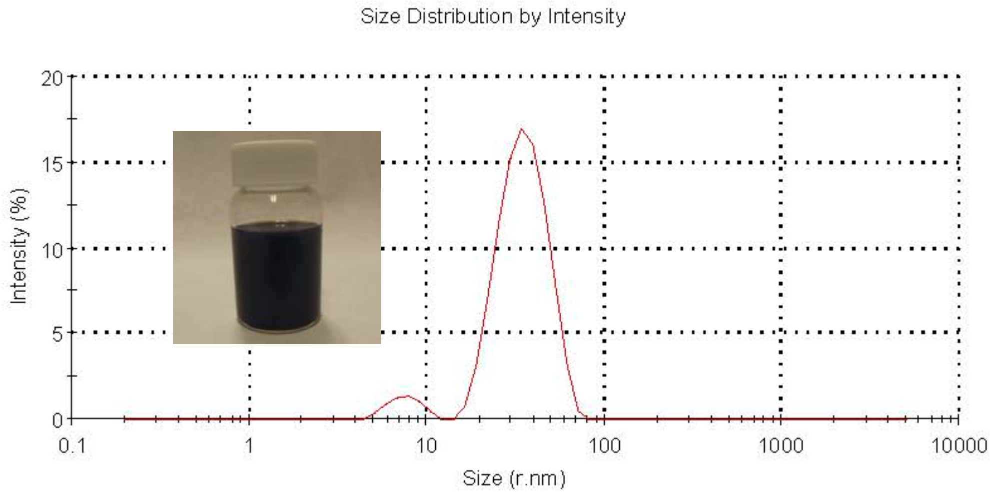

Figure 4. Size distribution of ITO nanoparticles dispersion

PRODUCTION OF ITO TARGETS

TCOs films can be obtained through different technologies; however, the technology most commonly used with this end is sputtering. To use this technique, ceramic targets have to be produced through a process based in powder compression and sintering.

Within RECYVAL-NANO it was employed ITO nanoparticles to prepare targets because, as it is well known, the surface of nanoparticles is highly reactive due to the surface area to volume ratio. This is due to the reason that most of the atoms that composes the nanoparticle are on the surface, which makes them more reactive. As a clear consequence of this, the sintering temperatures of these particles could be lower than the conventional ones and can be sintered over shorter time scales, reducing at the same time both ITO target manufacturing process and energy consumption.

ITO targets were produced during the project by means of cold pressing using a simple hydraulic press. The results indicated that densities of ITO green compacts targets of 4.75 g/cm3 were achieved with pressures of only 390 kg/cm2 where used values of pressure for compaction are in the range of 1000 to 3000 kg/cm2. Sintering of targets in the project under air atmospheres and below 1400 ºC achieved values of densities of 6.40 g/cm3 (89 % of the target).

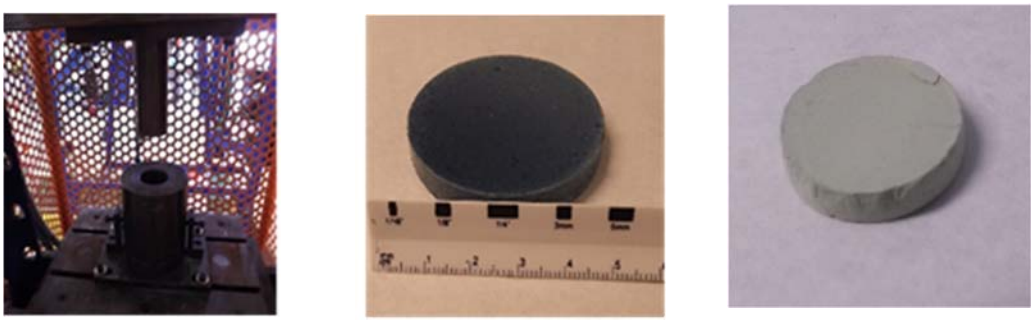

Figure 5. Images of ITO target preparation

PRODUCTION OF ITO THIN FILMS WITH ITO NANO-BASED TARGETS

Sputtering of the ITO targets for the production of thin film coatings was developed in RECYVAL-NANO by the partner Plasmaquest using their proprietary HiTUS (high target utilisation sputtering) plasma deposition system. In this system, the plasma is generated independently of the target, this affords the benefit of being able to separately control the plasma density (ion current) and the target bias (Ar ion impact energy). Therefore, a low target bias can be applied for sputtering, which is beneficial in minimising damage to delicate targets.

Within the project, optimisation of the deposition process was made in order to be able to produce coatings from commercial sintered ITO targets and nano-based non-sintered targets produced. Although the non-sintered targets showed a slow deposition rate in comparison with commercial sintered targets, the values of resistivity obtained were only slightly higher than commercial ones. **The coatings produced could be classed as transparent conductive oxides, showing promising results in terms of resistivity and transparency (~95%) when compared with coating obtained from commercial targets.**

Target	Resistivity (Ω.cm)
Non-sintered target	$7x10^{-4}$
Commercial target	$4.5x10^{-4}$

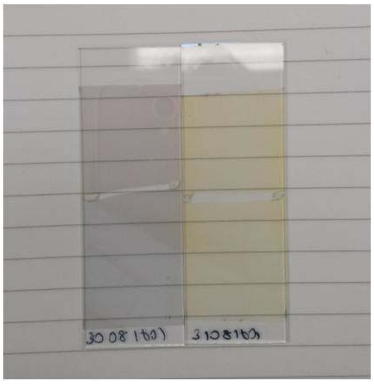

Figure 6. ITO coatings on glass from the non-sintered target (left) and a commercial target (right).

In view of the results obtained during the project, some potential aspects could be further investigated, on the one hand, if exist the possibility of minimise the temperature in ITO targets production process and obtain thin conductive oxide films with good properties and deposition rate due to the fact of employ nanoparticles. And on the other hand, to see how far in reducing the values of resistivity could be achieved when using nano-based ITO targets decreasing to a value in the low $x10^{-4}$ Ω.cm region.

INTEREST ON EXPLOITATION

The Recyval consortium is interested in making available to third parties these technologies developed under the project in several ways:

a) Sale of Know How: The relevant Recyval partners make process technology available under licence

b) Joint research: The relevant Recyval partners will contribute Recyval Foreground to new research projects on terms to be agreed.

Third parties interested in any of the above should in first instance contact to:

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giving information on their areas of interest