Microwave Sintering of Sn doped In_2O_3

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Introduction

Indium Tin Oxide (ITO)

Typical Applications:

- ✧ Resistive elements in integrated circuits
- ✧ Sputtering targets
- ✧ Conductive inks
- ✧ Transparent conductive thin films

- ✧ **Highly dense and pure ITO sputtering targets are crucial for apply uniform and reliable thin film layers to glass and plastic substrates.**

Microwave Sintering

Goal:

Use microwave sintering to **increase density of indium tin oxide sputtering targets**, thereby **increasing purity of ITO coatings.**

Typical Microwave Sintering Advantages :

- ✧ Rapid uniform heating
- ✧ Decreased sintering temperature
- ✧ Improved physical and mechanical properties
- ✧ Unique properties over conventional sintering processing
- ✧ Decreased cost and time of processing

- ✧ Little information is known about the dielectric properties of ITO, especially at elevated temperatures.

Procedure:

- 1.5 g compacts were uniaxially pressed from 4% and 10% Sn doped In_2O_3, then cold isostatically pressed to 20,000 psi.
- Pellets were microwave and conventionally sintered over the temperature range of 1100-1500°C.
  - Conventional: 5 °C/min ramp, 10 min dwell
  - Microwave: 50% for 12 min, 75% for 6, 100% (900 W) for remainder
    - Average ramp rate 40 °C/min, no hold
- Geometric density, conductivity and microstructural comparison was evaluated.
  - Conductivity was measured by 4-point method on cut bars from the compacts.

Microwave and Thermal Package

Figure 1. Microwave sintering was performed in a Research Microwave System Thermwave (A), using the refractory thermal package (B).

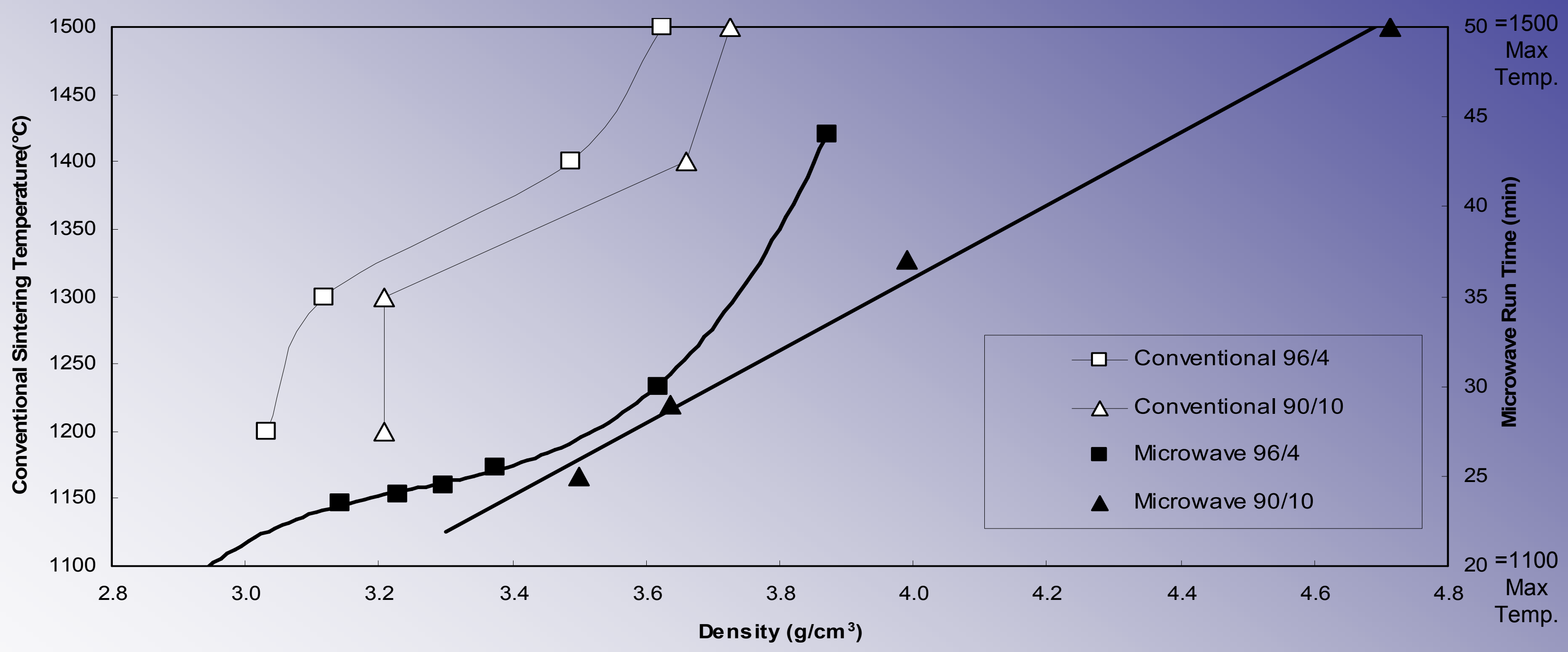
Microwave Sintered Pellets

Figure 2. Various stages of pellet densification. These samples are 10% Sn doped and the sintering temperatures range from 1100-1500 °C. A color change is observed with the densification of indium oxide.

Figure 3. Contrasts the difference between conventionally sintered (A), green (B) and microwave sintered (C) pellets for 4% Sn doped pellets at 1400 °C sintering temperature.

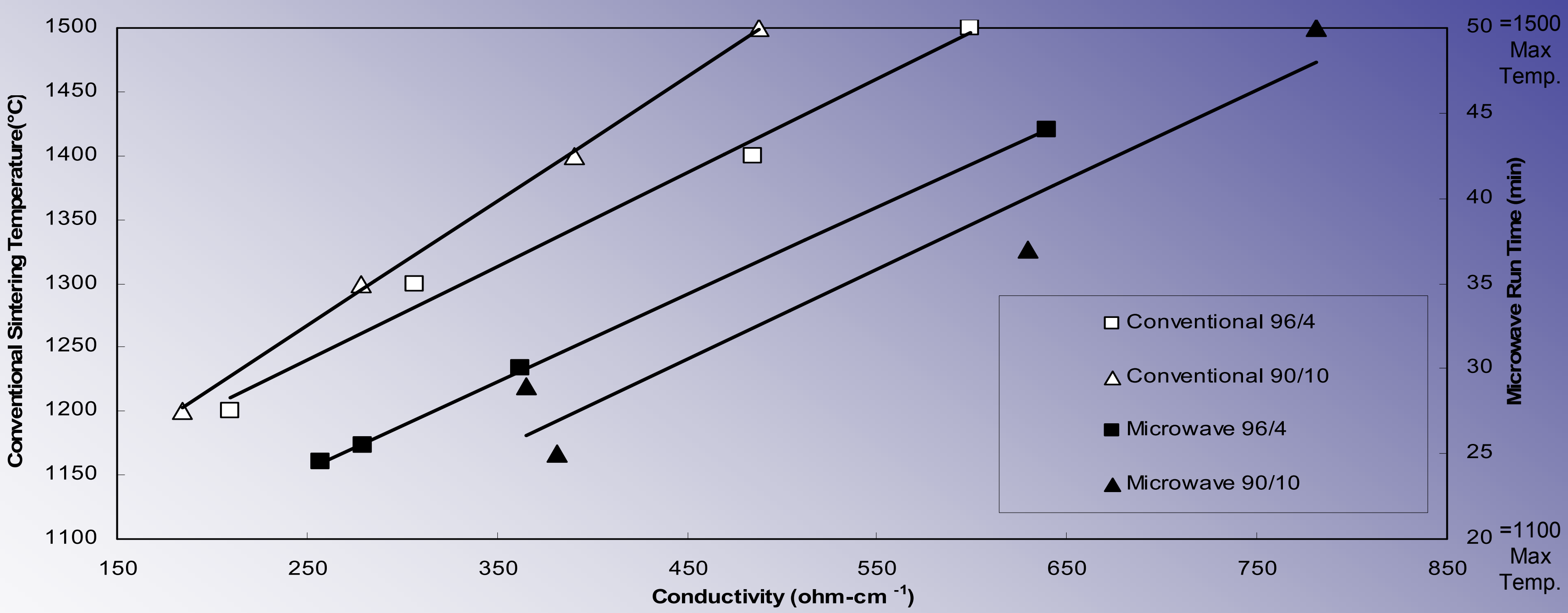
A B C

Density Comparison



✧Microwave sintering produced denser pellets than conventionally sintered samples for similar firing temperature in a fraction of the time

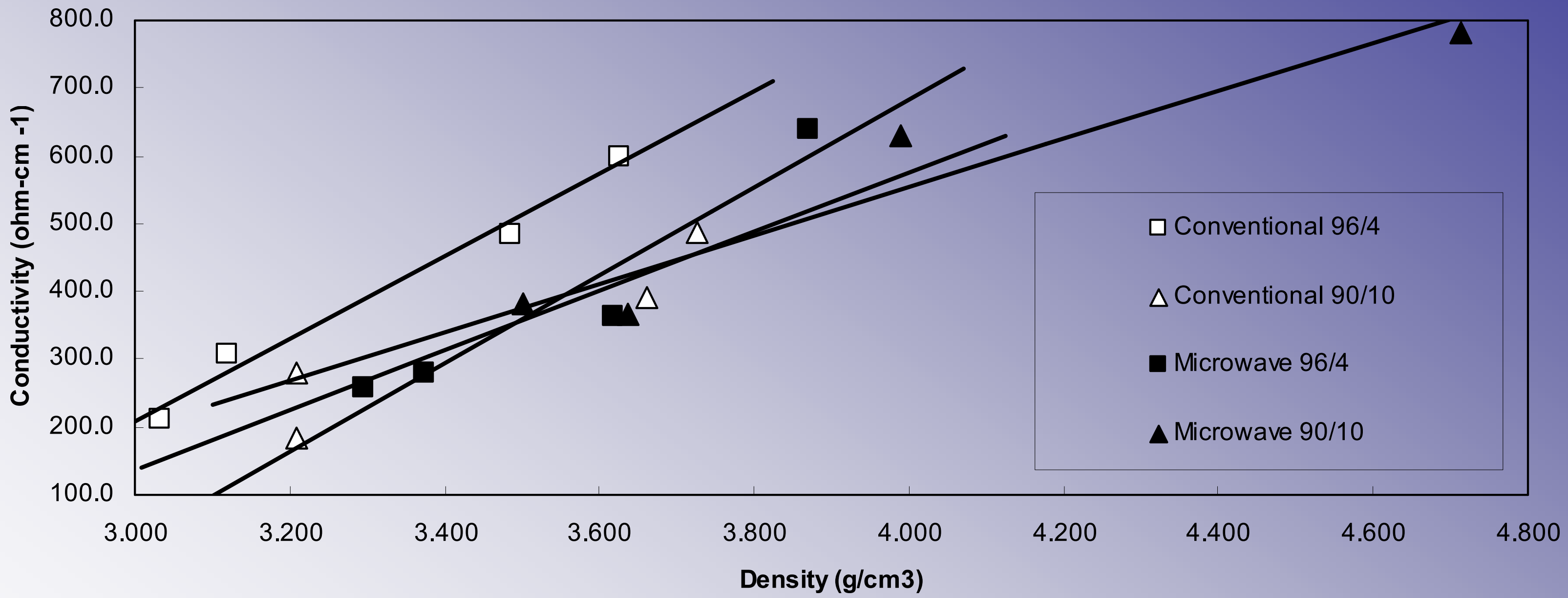
Conductivity Comparison



✧ITO conductivity increases with sintering temperature

✧Microwave sintered compacts display a higher conductivity than conventionally sintered for similar sintering temperature.

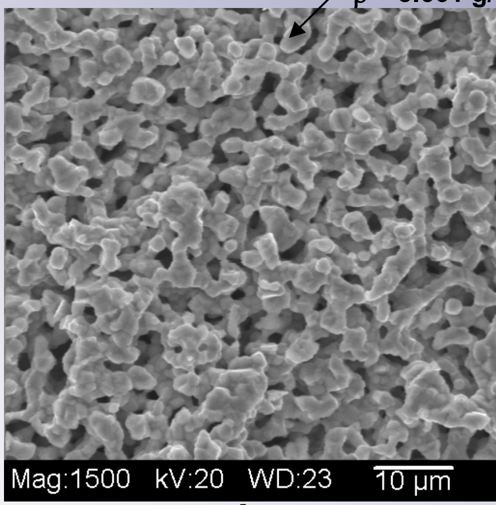
Conductivity and Density



✧Conventionally sintered compacts have a higher conductivity than microwave sintered samples for constant density.

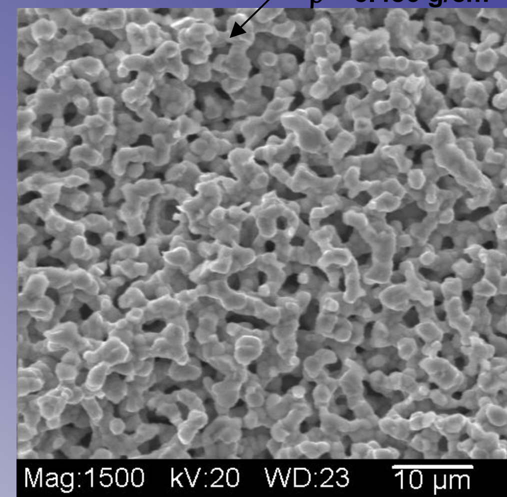
SEM

ρ = 3.661 g/cm3



A

ρ = 3.486 g/cm3



B

✧ Secondary electron mode SEM images of fractured surface morphology for representative:

Conventional Sintered ITO

(A) 90/10 $\rho = 3.661$ g/cm^3

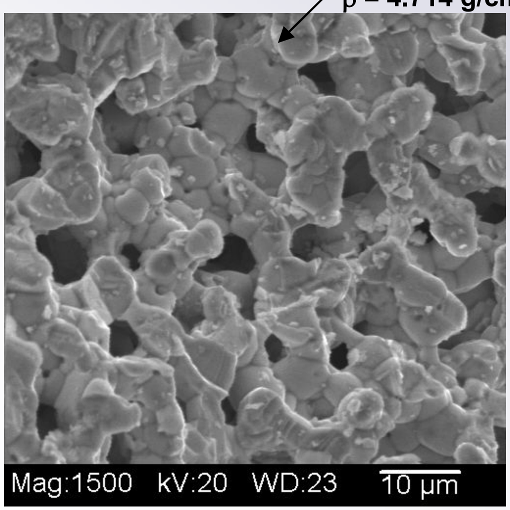
(B) 96/4 $\rho = 3.486$ g/cm^3

Microwave Sintered ITO

(C) 90/10 $\rho = 4.714$ g/cm^3

(D) 96/4 $\rho = 3.617$ g/cm^3

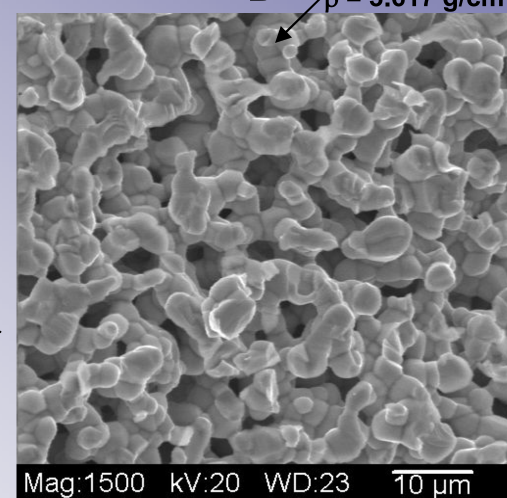
ρ = 4.714 g/cm3



C

← Larger grain size →

ρ = 3.617 g/cm3



D

Conclusion:

- Microwave sintering produced the most dense and most conductive compacts for both the 90/10 and 96/4 ITO compacts

- Conventional sintering produced more conductive compacts at a similar density

- Microwave sintering of ITO can be optimized to:
  - Increase density
  - Decrease sintering time
  - Decrease energy consumption

Future Work:

Increase Density of ITO Compacts

- ✧ Increase green density
  - ✧ Improve processing
- ✧ Use powder with smaller particle size
- ✧ Varying the sintering conditions
  - ✧ increasing dwell time

✧ Scale-up Work

- ✧ Sintering larger masses
- ✧ Microwave sintering at higher rates with more power