Gallium Oxide Nanostructures for High Temperature Sensors

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Project: DE-FE0007225
Project Period: 10/01/2011 to 09/31/2014
Outline

• Introduction
• Research Objectives
• Experiments
  ► Synthesis
  ► Characterization
• Results and Discussion
  ► Ga₂O₃ Thin Films (Physical Methods)
  ► Ga₂O₃ Nano-Particles and Nano-Wires (Chemical Methods)
• Summary & Future Work
Introduction
T,P Tolerance

High-T

High-P

Ox. and Cr. Resistance

High-O

High-C

Energy Systems
Sensor

Microstructure
Choice of Materials
Poisoning

Shelf Life
Stability

Sensitivity
Selectivity

Contamination

Gallium Oxide (Ga$_2$O$_3$)

- **Fundamental Science – Phases** ($\alpha, \beta, \gamma,$ and $\delta$)
- **Surface Chemical Reactions**
- **Novel Properties – Metal Doping**

**Ga$_2$O$_3$**

- **Ease of Thin Film Formation**
- **Integration into Nano-electronics**

**Chemical Sensors**

- **Wide band gap (>5 eV) semiconductor**
- *High thermal and chemical stability (T$_m$: 1725 °C)*
- *Due to a high melting point and stable structure, it is one of the most suitable materials for high temperature gas sensing.*
Sensing Mechanism

At T>700 °C, defects $\rightarrow$ equilibrium with surrounding atmosphere $\rightarrow$ n type conductivity $\rightarrow$ depends on oxygen partial pressure

$$\sigma = \sum_i P_{O_2} \exp \left( \frac{-E_A}{k_B T} \right)$$

Electrical conductivity
Activation energy
Oxygen partial pressure
Boltzmann constant
Temperature

At T< 700 °C, Ga-oxide exhibits sensitivity to reducing gases (CO, H$_2$)
Objectives and Goals

**Objective 1:** To fabricate high-quality pure and doped Ga$_2$O$_3$-based materials and optimize conditions to produce unique architectures and morphology at the nano scale

**Objective 2:** Derive the structure-property relationships at the nanoscale dimensions and demonstrate enhanced high-temperature oxygen sensing and stability

**Objective 3:** To promote research and education in the area of sensors and controls

**Goal:** Develop the high temperature oxygen sensors (employing Ga$_2$O$_3$-based nanostructures)
Experiments
Materials

Target (for Deposition)
\( \text{Ga}_2\text{O}_3 \)

Substrate(s):
- Si(100)
- Alumina

Powder (for Milling)
\( \text{GaN} \)
Fabrication – Thin Films

♦ RF magnetron sputtering
♦ Deposition Conditions

Fixed:
- Base pressure ~10^{-6} Torr
- Power: 100 W
- Target-Substrate distance: 7 cm
- Sputtering gas: Argon

Variables:
Sample set 1:
Substrate temperature: RT to 800 °C

Sample set 2:
Deposition time or thickness:
Nano- Particles, Wires and Belts

1. Start
   - Measure 0.5 g of planetary ball milled powder

2. Place sample in an alumina crucible. Insert the crucible in a tube furnace

3. Start nitrogen flow into the tube furnace. Flow rate maintained at 3 L/min (using a flow meter) for 10 min.

4. Check for leaks
   - Yes: Stop nitrogen flow & tighten the end caps. Apply vacuum grease to the tubing connections and end caps
   - No: After 10 min. Reduce flow rate to 0.3 L/min. Set furnace temperature to 930 °C

5. Let the furnace run for 12 hours.

6. After 12 hours. Turn off the furnace. Let the crucible cool in a nitrogen atmosphere for another 12 hours.

7. After cooling, stop nitrogen flow. Retrieve crucible. Measure mass of the sample. Note the color of the sample.

8. Perform XRD, SEM & EDS analysis on the retrieved sample.

9. Stop
Characterization
Results and Analysis
Surface Morphology - SEM

$\theta_{\text{dep.}} = 30$ min.
Surface Morphology - SEM

600 °C

800 °C

$t_{\text{dep.}} = 30 \text{ min.}$
Morphology – Thickness

400 °C

500 °C

600 °C

t_{\text{dep.}} = 60 \text{ min.}
$t_{\text{dep.}} = 60 \text{ min.}$
Grain Size

Grain Size

Ts (°C)

Grain Size (nm)
Crystal Structure – GIXRD

Amorphous

Texturing

Crystalline

Texturing
Optical Properties

![Graph showing optical properties with transmittance and absorbance plots.]

\[ t_{dep.} = 30 \text{ min.} \]
Optical Properties

\[ t_{\text{dep.}} = 60 \text{ min.} \]
Optical Properties

Bandgap for RT 30 min

Equation: $y = a + bE$

Adj. R-Square: 0.9999

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(Ev) vs $(\alpha E)^2$
Ball Milling Synthesis

GaN Original Powder

After ball milling (03/03/12)

Nano Particles !!!
Summary

- Ga-oxide thin films, nano-particles and nano-belts were synthesized
- The effect of temperature is remarkable in deciding the structure and morphology of Ga-oxide films
- Preliminary results obtained on the optical properties are encouraging
Future Work
Acknowledgements

- DOE-NETL
- Sampath Samala, Ashwin Kumar and Ernesto Rubio
- Richard Dunst
- EMSL/PNNL, Richland, WA
THANK YOU!