

A stylized sunburst graphic composed of numerous light gray, wedge-shaped segments radiating from a central point, set against a solid black background. The segments are arranged in a circular pattern, creating a sunburst effect.

# Developing and Assisting Next Generation Window and Absorber Solar Materials.

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A Subsidiary of  
Brush Engineered Materials

# Materials Development

- Cadmium Sulfide
  - Traditional CdS Powders
  - Need for Microcrystalline CdS Powders
- Copper Indium Gallium (di)Selenide
  - Producing Stable, Reacted CIGS Powders
  - Producing Stable, Pressed CIGS Targets
  - Producing Stable, Scalable CuGa Alloys for CIGS

# Cadmium Sulfide

- Basis for Powder Production as pigment
  - Heating acidic cadmium salt solutions in hydrogen sulfide gas.
  - Temperature of solution and reaction rate effected structure and color.
  - Produced a fine, low density powder.
  - Color ranged from classic brilliant yellow to dark red.



Classic Cubic CdS Yellow.



Hexagonal CdS very red.

# “Cadmium Yellow” Raw Material

- Early Evaporation Material
  - Large scale reaction and filtering capacity made cheap raw material attractive for testing.
  - Low density powders hot pressed into medium density tiles (60%)
  - Tiles were sized to granules (0.8mm to 1.5mm) for evaporation trials.
  - Cold press and sinter routes not feasible due to contamination and moisture concerns.

# Need for New CdS Material

- Evaporation materials did not allow proper n-type film preparation.
  - Loosely bound compound had ambiguous decomposition behavior.
- PVD targets from this material had limited density by any load/press means.
  - Target tiles would crack in-plane or during bonding.
  - Bright yellow appearance was misleading and often behaved like sulfur rich compound.

# New Microcrystalline CdS

- New reaction closer to solid state reactions with special attention to formation temperature.
- Specialized sizing and sieving techniques developed for evaporation or PVD standards.
- Produced a fine, high density microcrystalline powder.
- Color consistently dark yellow/orange.



New Microcrystalline CdS

# CdS PVD Tiles by Hot Pressing

- Refined loading and hot pressing approaches minimized moisture, increased (>90%) density and improved base tile characteristics.



As Pressed CdS PVD Tile (w/redux)



After Machining, CdS PVD Tile

# CdS for Solar Modules

- -325 mesh, -200 mesh and customer powder sizes are available for evaporation or pressing projects.
- Hot pressed, high density PVD tiles are available for n-type CdS deposition.
- Hot pressed PVD tiles are more machinable and lower moisture/oxygen than wet chemical based options.
- CdS remains very sensitive to deposition technique but RF, pDC and evaporation techniques have compared favorably to CBD films.



# Copper Indium Gallium (di) Selenide

1)  $\text{Cu} + \text{In} + \text{Ga} > \{\text{Cu: In: Ga Intermetallic}\} \gg \text{H}_2\text{Se (or Se)}$   
yields  $\text{Cu(In,Ga)Se}_2$  or,  
2)  $\text{Cu}_2\text{Se} + (\text{In,Ga})_2\text{Se}_3$  yields  $\text{Cu(In,Ga)Se}_2$  and finally,  
3)  $\text{Cu} + \text{In} + \text{Ga} + \text{Se}$  yields  $\text{Cu(In,Ga)Se}_2$ .

\* Dr. Noufi, R. - High Efficiency CdTe and CIGS Thin Film Solar Cells: Highlights of the Technologies Challenges, IEEE 4th World Conference on PV Energy Conversion. Presentation. May 7-12, 2006. Waikoloa, Hawaii.

- It is generally recognized that the above list represents the most common pathways for creating a useful and stable CIGS layer in CIGS based solar modules.
- Until recently, very few options outside of single crystal, Bridgman Grown crystalline powders have been available.
- Similarly, alloys rich in Copper dominate, and the use of Ga or In in the alloys has been slow to develop.

# Need for Stable CIGS Powder

- An evaporation CIGS would have to be stable, homogeneous and non reactive.
- As a PVD target, the powder would have to be dense, stable, hard and free from intergranular fracture in sizing and transport.
- The powder would have to be freely flowing in bulk and dilute phase transport for die filling or feeding.

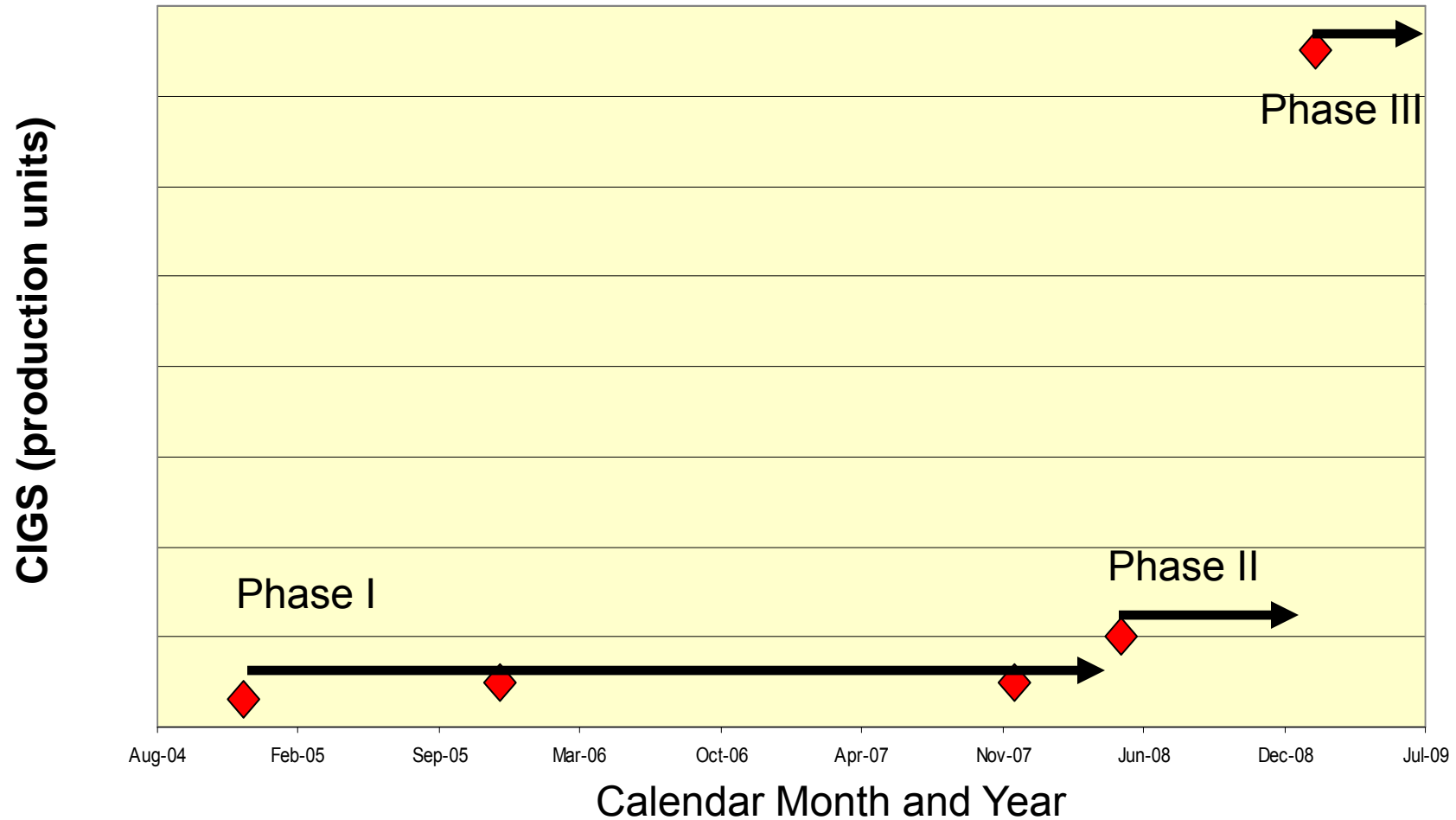
# CIGS Reacted Powder

- Heat of reaction must be maintained to favor primary quaternary selenide.
- Rate of reaction limited to prevent gas generation and vessel interactions.
- Unlike Bridgman Ingots, chemical and material character is homogeneous.



Standard CIGS powder

# CIGS Reaction and Scale-up



# Composition, Scale and Process

- Phase I. Mostly Catalog and Small Targets
  - Develop reactions, QA techniques and build composition confidence limits.
- Phase II. Scale up finished goods and Test Concepts for Scaling PVD tile raw materials.
  - Requires specific development for die loading and composition variance.
- Phase III. Optimize Reaction and Scale to meet cost and performance criteria.
  - Powders for direct use may differ from PVD tile materials.

# Phase I & II Milestones

- XRD, ICP, Spectrograph Standards.

Average Composition of Stable/Identifiable CIGS at%

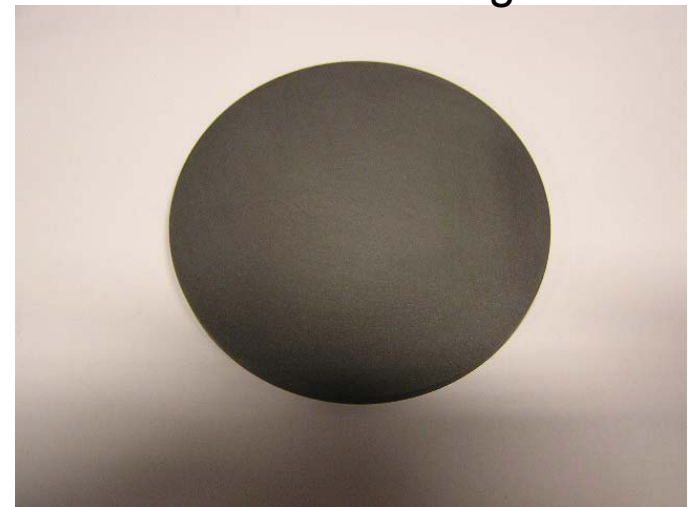
Cu 20 – 25      In 15 – 21

Ga 6 – 9      Se 50

- Powder yield increased from R&D to Production Quantities.
- Pressing and Machining Process Standardized.

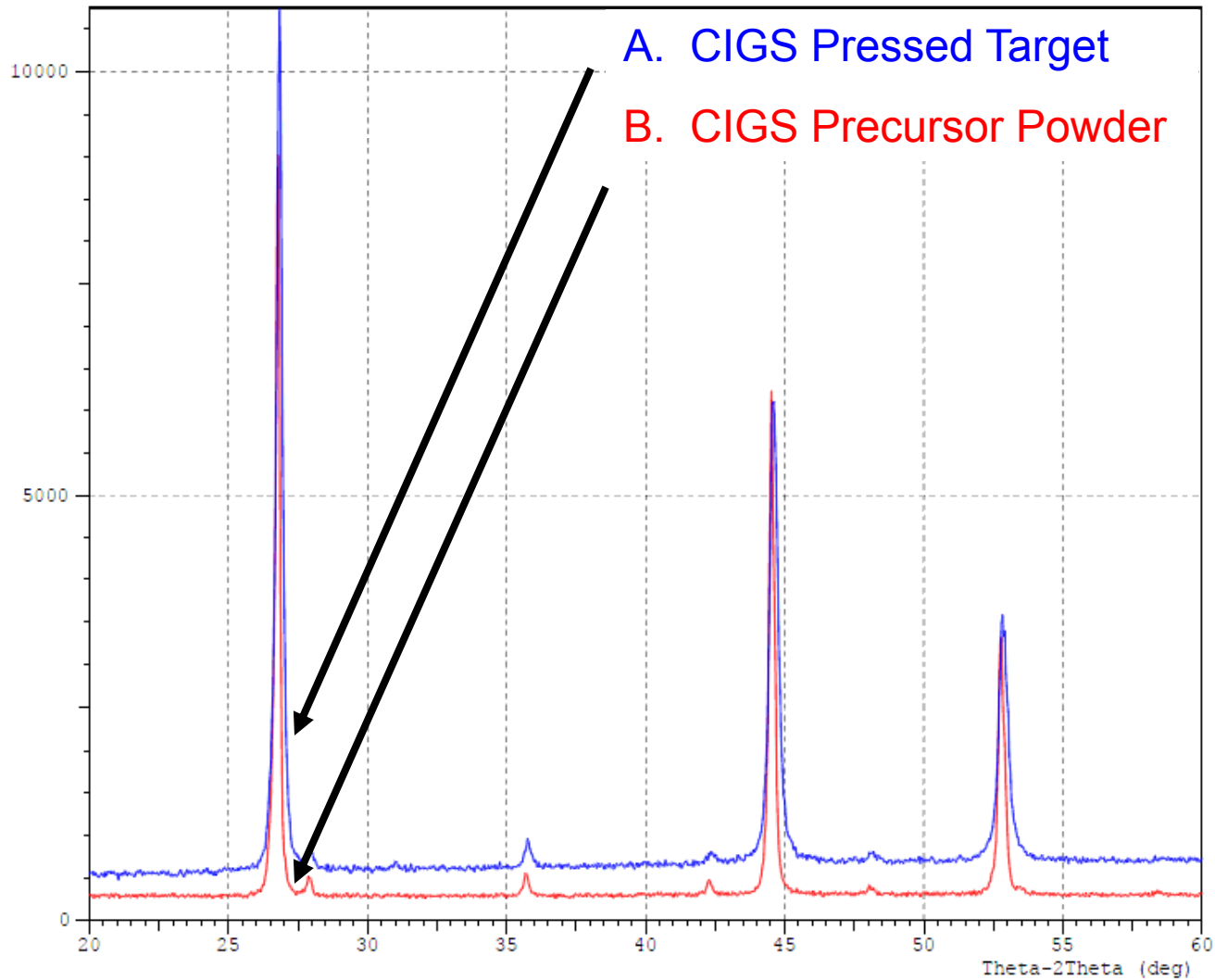


As Pressed CIGS Target

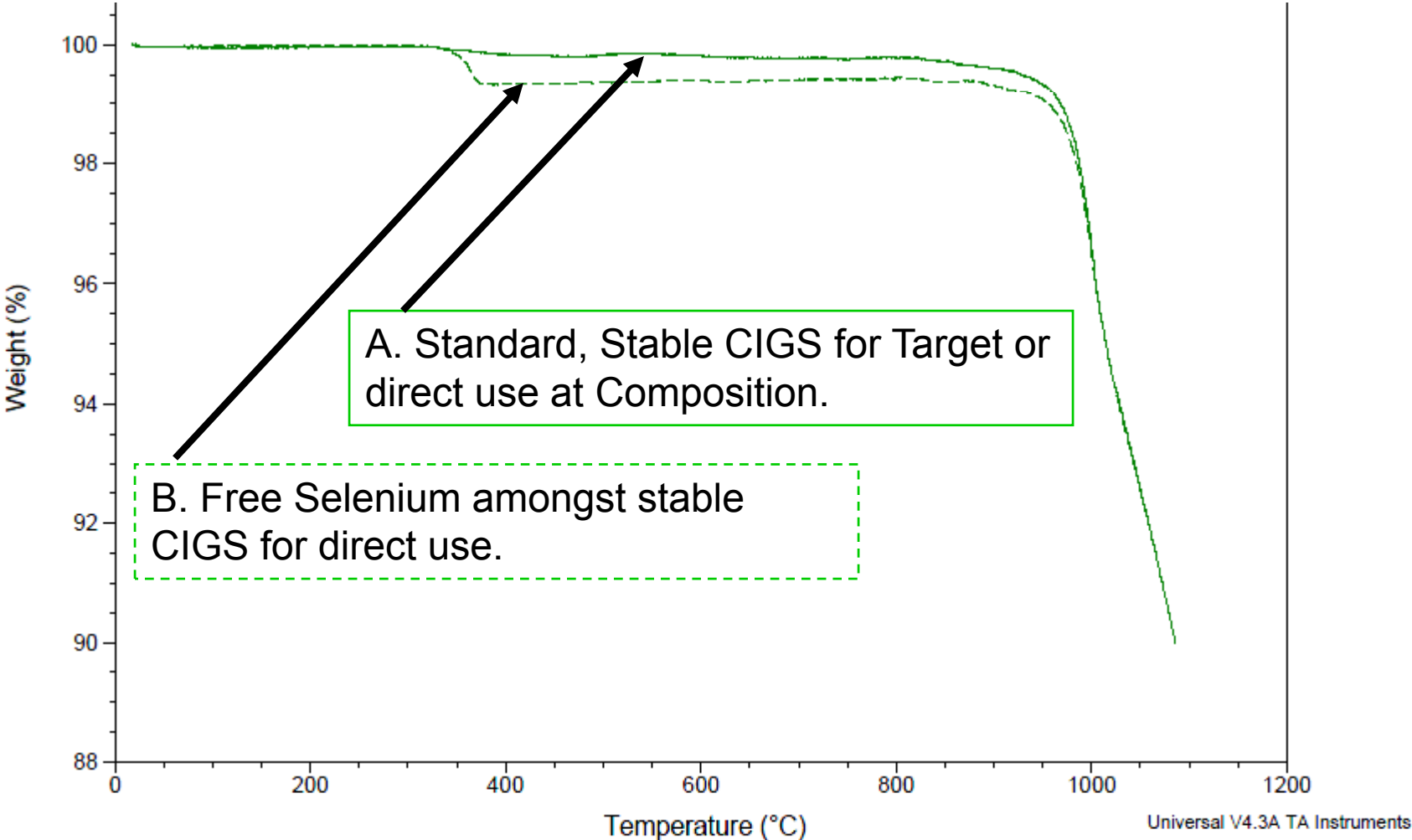


Machined CIGS Target

# XRD Overlay, CIGS Powder and Target



# Example, Thermogravimetric Analysis of Two CIGS Compositions





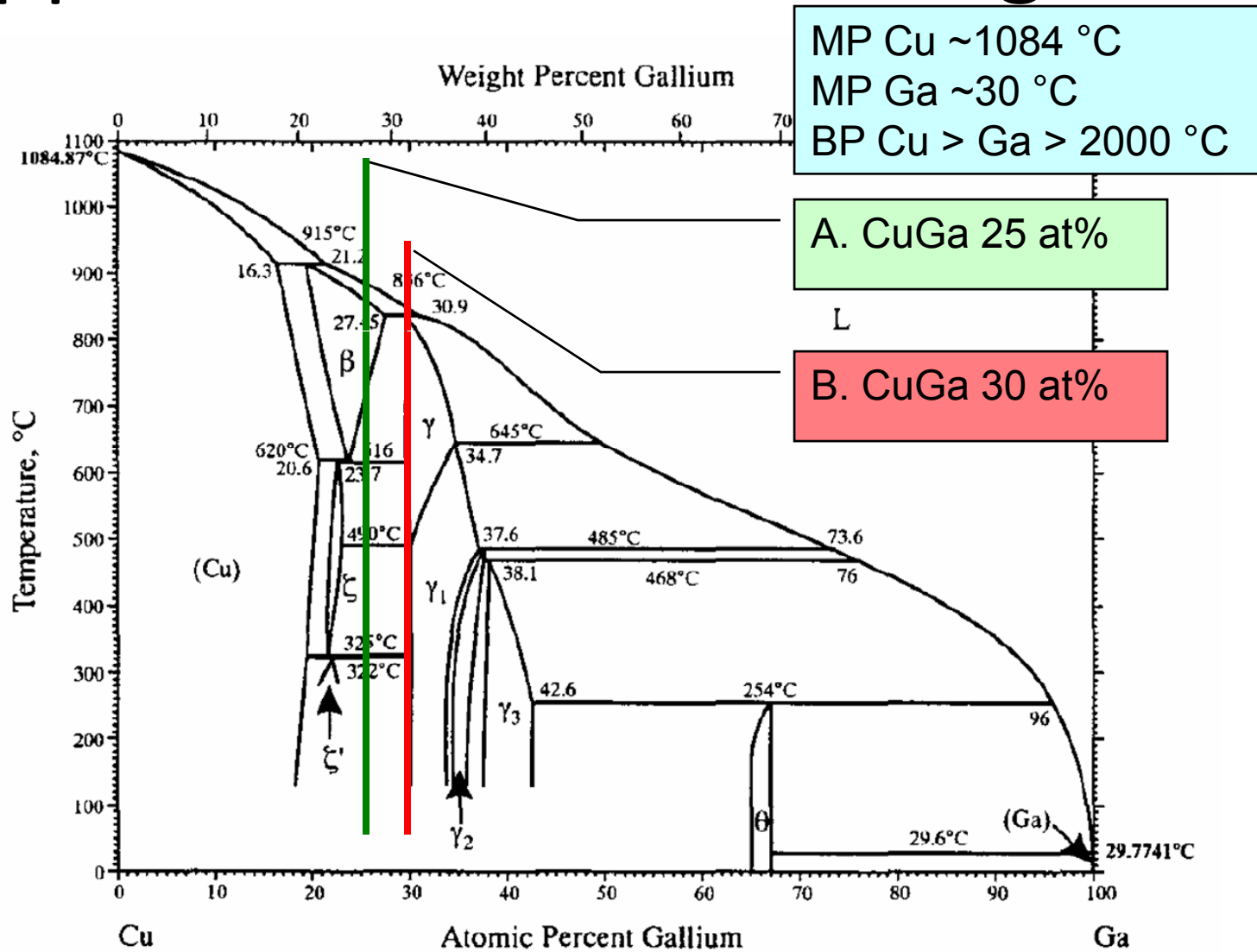
# CIGS Compound for Solar Modules

- -325 mesh, -200 mesh and customer powder sizes are available for evaporation or pressing projects.
- Hot pressed, high density PVD tiles are available for p-type CIGS deposition.
- Hot pressed PVD tiles can be arranged on large backing plates with conventional bonding.
- Standard Compositions have pre and post pressing XRD verification and characterization.
- CIGS remains very sensitive to deposition technique but RF, pDC results show excellent Se incorporation.

# Phase III, Powder Capacity and Alloys for Multi-Step CIGS PVD

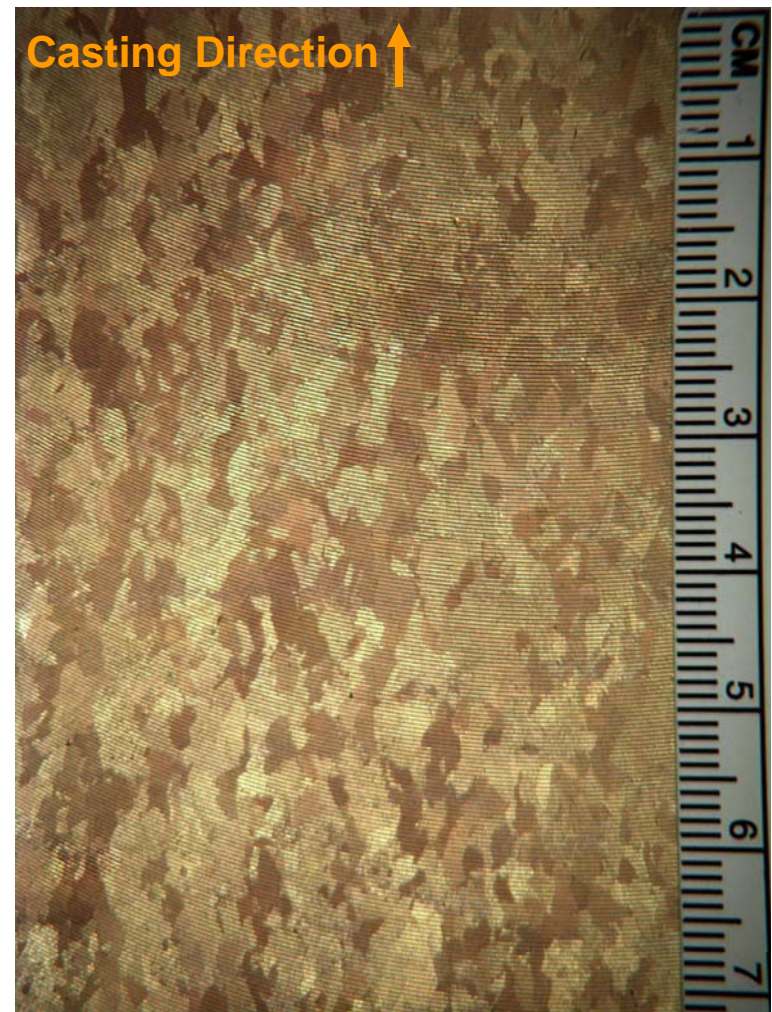
- Work proceeds further increase production units and lower cost of reacted CIGS.
- Work continues to develop and scale CuGa alloys for large PVD platforms.
- Proprietary and Batch Casting Technology was employed for different target thicknesses and compositions.
- All casting technology must retain desired workability and Gallium content throughout.

# Copper – Gallium Phase Diagram



# Proprietary Cast CuGa Alloys

- CuGa 25 at% developed at 8mm thick by 200mm wide by “x” long.
- Casting is done in N<sub>2</sub> purge.
- Uniform 1-3mm grain orientation & size.
- Fixed cooling rate at Atmosphere.
- Proper Cubic / Hexagonal phase relationship maintains machinability.



Proprietary Cast CuGa 25 at%

# Cast and Hot Rolled CuGa Alloys

- CuGa 25 at% developed as VIM cast and hot rolled to up to 24mm tiles.
- Custom cooling profile controls cubic and hexagonal phase concentrations.
- Hot top is easily cut off.
- Hot rolling produces fine 0.5mm uniform grains throughout.
- Machinability is excellent.



Cast CuGa 25 at% @ 25x

# CIGS Alloys for Solar Modules

- Proprietary Cast CuGa25 8mm x 200mm x “specified” m @ 1-3mm grain size is available for large PVD tools.
- Cast and rolled CuGa25 16 -24mm @ 0.5mm grain size tiles are available for large PVD tools.
- Novel hybrid technique for the production of CuGa30 8mm polycrystalline tiles is available for large PVD tools.
- Novel technique for at% Gallium 30-50% are under development.
- All CuGa tiles can be conventionally bonded.

# Solar Materials Development

- You have seen a novel Cadmium Sulfide Developed from initial chemistry to pressing.
- You have seen how Copper Indium Gallium (di)Selenide powders and Targets have been developed for a series of technologies.
- You have seen traditional casting and metallurgy bring new casting products for multi-step CIGS PVD applications.

# Thank you for your Attention!

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## Subsidiaries

CERAC, incorporated ▪ Thin Film Technology ▪  
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