

**MSNW, INC.  
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**Low Cost Ceramic/Ceramic Matrix Composites for  
Lightweight Space Based Mirrors**

**Dual Use Science & Technology Program  
Technology Investment Agreement  
F33615-02-2-5207**

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## **OBJECTIVE**

**Develop and demonstrate ultra-lightweight bonded sphere constructions of low CTE glass material to serve as low-density mirror substrates.**

## **SPECIFIC GOALS FOR THE LOW EXPANSION SUBSTRATES:**

- Low cost materials and fabrication processes**
- 15 to 30% theoretical density**
- Near-zero CTE  $\pm 95^{\circ}\text{C}$**
- Scalable to large apertures**

## **ALTERNATIVE TO TRADITIONAL GLASS MIRRORS**

- **Bonded Low Expansion Glass Microsphere Approach**
  - **Produced via sol-gel which decreases both raw material and manufacturing costs**
  - **Inherently lightweight**
  - **Near-net shape forming**
  - **Inherently homogeneous**

## **TECHNICAL APPROACH**

- **Phase 1 – Funded**

- Task 1 – Develop Low Expansion Glass Spheres**

- Task 2 – Low CTE Bonding Material Development**

- Task 3 – Initial Physical/Mechanical Property Evaluation of Bonded Arrays**

## **TECHNICAL APPROACH (CONT)**

- **Option Phase 2 – Requires Funding from Other Sources & Guidance From Component Producers**

**Task 4 – Porous/Hollow Sphere Development for Reduced Weight**

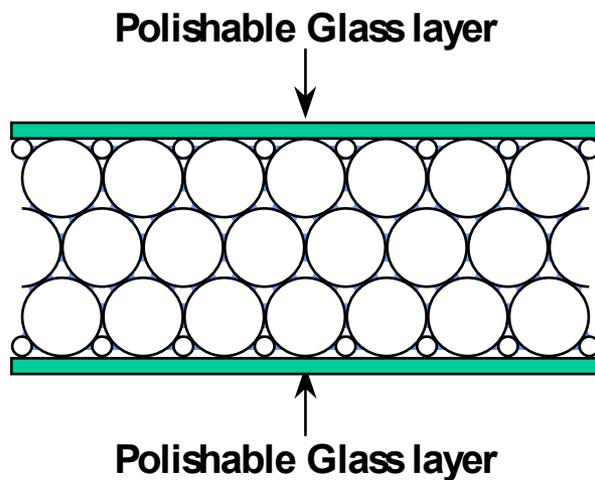
**Task 5 – Develop Polishable Glass Surface Layer**

**Task 6 – Evaluate Formability of Low Expansion Glass Substrate Cores**

**Task 7 – Process Scale-Up, Design and Fabrication of a 0.5 meter Mirror Substrate**

## SCHEMATIC

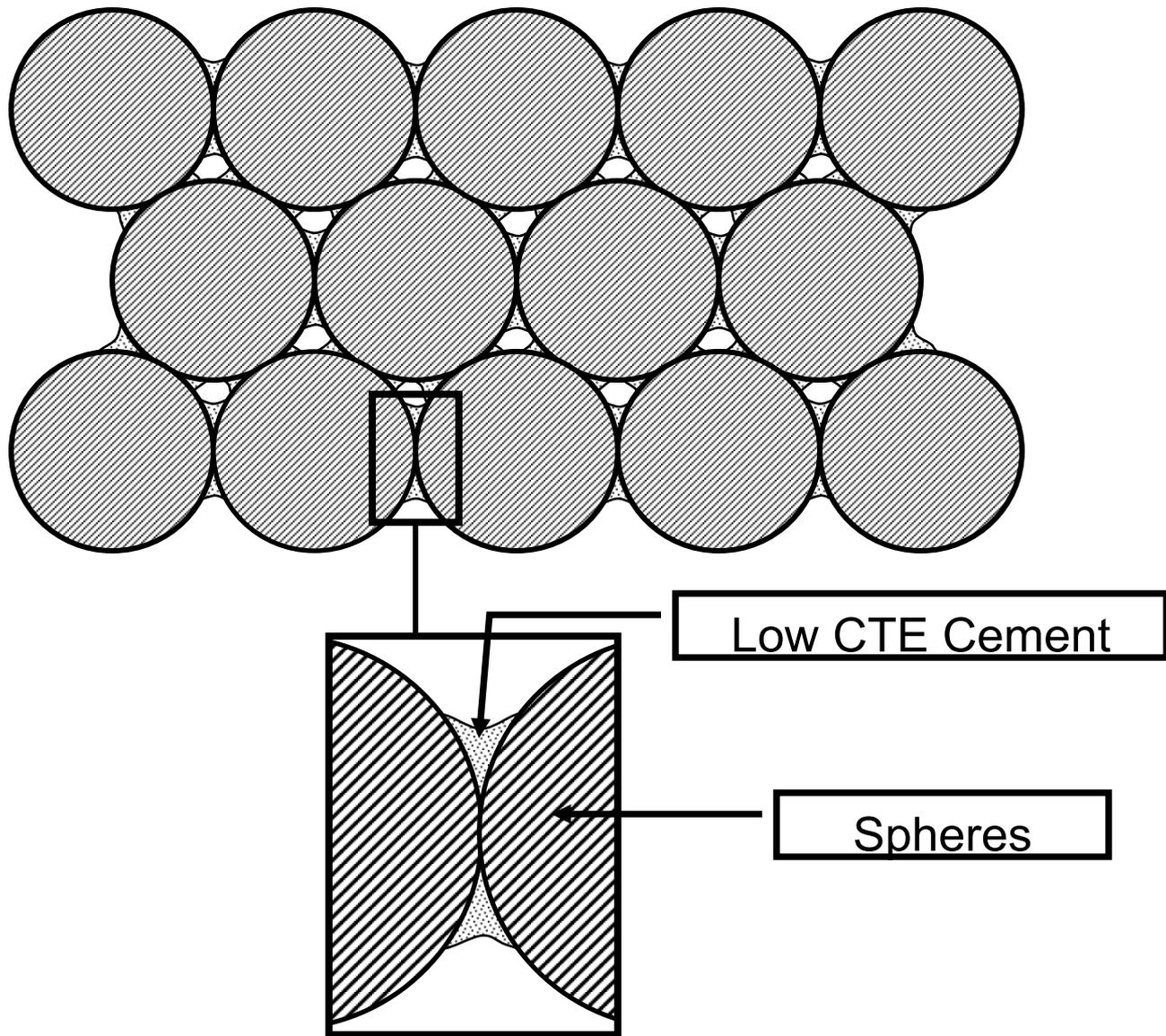
### MSNW, INC. LIGHTWEIGHT, LOW CTE GLASS MIRROR SUBSTRATE



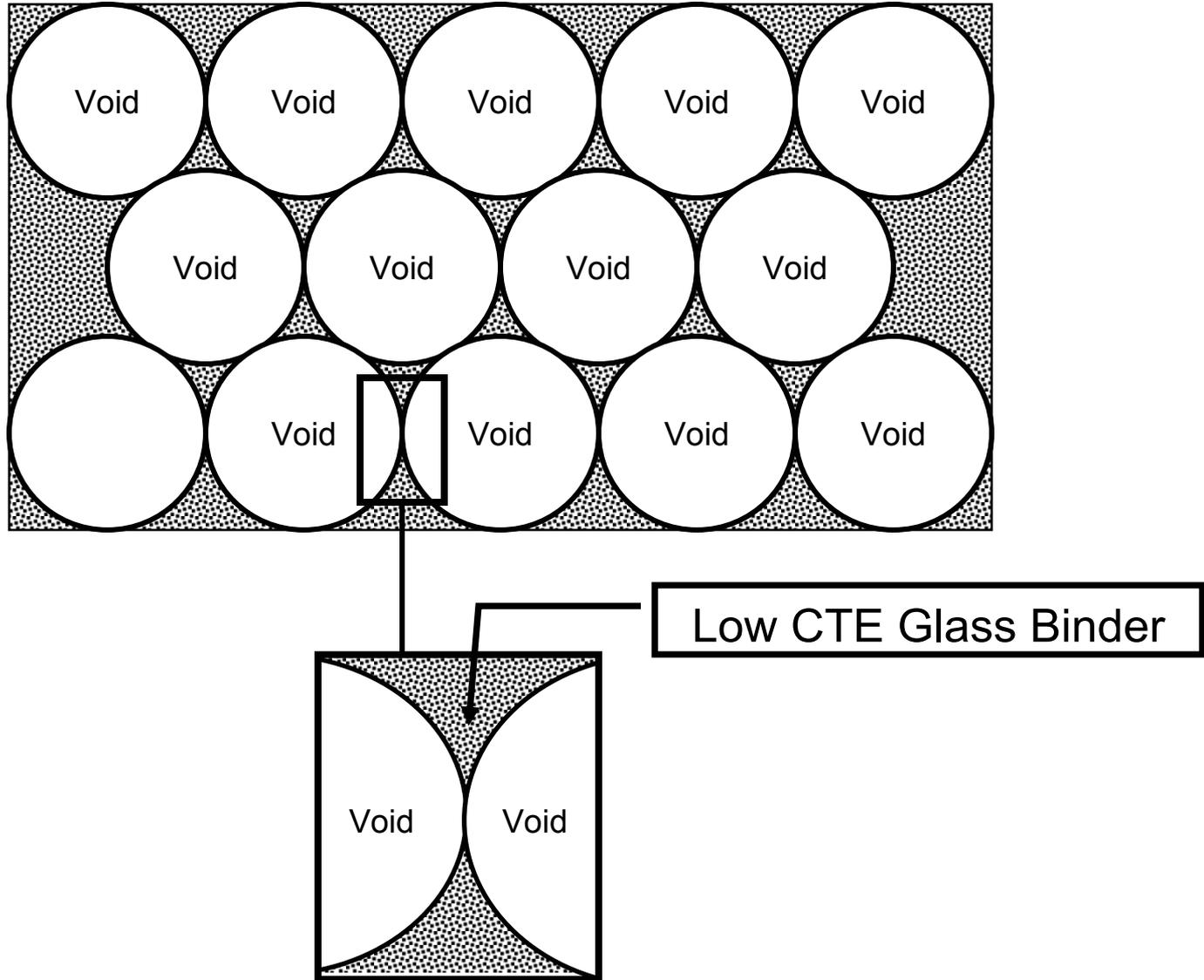
## Progress

- **Currently Studying Two Overall Approaches**
  - 1) **MSNW and/or commercial low CTE glass microspheres bonded with a low CTE binder (either an MSNW glass binder or a modified cement) that is a different composition from the spheres**
  - 2) **Commercial microballoons bonded with either MSNW's low CTE glass binder or a modified cement (spherical voids dispersed in a low expansion glass binder)**

**LOW COST CERAMIC/CERAMIC MATRIX COMPOSITES FOR LIGHTWEIGHT SPACE BASED MIRRORS**  
Initial Concept – Bonded Low Expansion Glass Spheres (Solid, Porous, or Hollow)



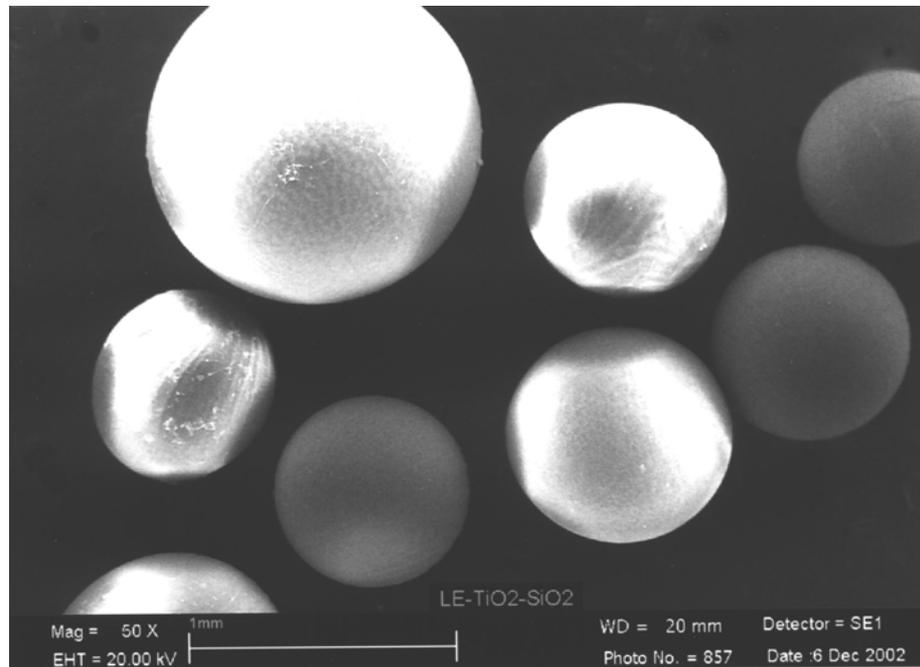
**LOW COST CERAMIC/CERAMIC MATRIX COMPOSITES FOR LIGHTWEIGHT SPACE BASED MIRRORS**  
**Alternate Concept – Thin-Walled Microballoons Bonded With Low Expansion Glass**



## Task 1 Progress

- **Low CTE Glass Sphere Development**
  - **MSNW, Inc. Has Developed a Sol Formulation That Produces a 7.5 w/o TiO<sub>2</sub> – 92.5 w/o SiO<sub>2</sub> Glass with Near-Zero Expansion.**
    - TEOS, ethanol, water, nitric acid, and titanium ethoxide Ti(OC<sub>2</sub>H<sub>5</sub>)<sub>4</sub>
  - **Transparent, Low CTE TiO<sub>2</sub> – SiO<sub>2</sub> Spheres Were Successfully Produced**
    - Utilized MSNW, Inc.'s sol-gel drop tower to form spheres
    - 500°C for 4 hrs heat treatment
  - **Porous TiO<sub>2</sub> – SiO<sub>2</sub> Spheres Were Also Produced**
    - Added fugitive carbon to the sol
    - Product material weak to date
  - **Commercial Microballoons Evaluated**
    - Aluminosilicate Microballoons
    - SiO<sub>2</sub> Microballoons

MSNW, Inc.  $\text{TiO}_2 - \text{SiO}_2$  Glass Microspheres



## Task 2 Progress

- **Binder Development**
  - **Four areas of binder development**
    - 1. An MSNW-developed, low CTE glass binder made via sol gel processing followed by a 500°C heat treatment
    - 2. An MSNW-developed, low CTE glass binder made via sol gel processing followed by a 1450°C heat treatment
    - 3. An MSNW-modified, low CTE commercial cement
    - 4. MSNW No. 1 Binder, modified alkali metal silicate
  - **Low CTE filler powders have also been evaluated as binder additives**
    - **MSNW low CTE glass frit**
      - Same composition as  $\text{TiO}_2 - \text{SiO}_2$  spheres
    - **Zirconium tungstate**

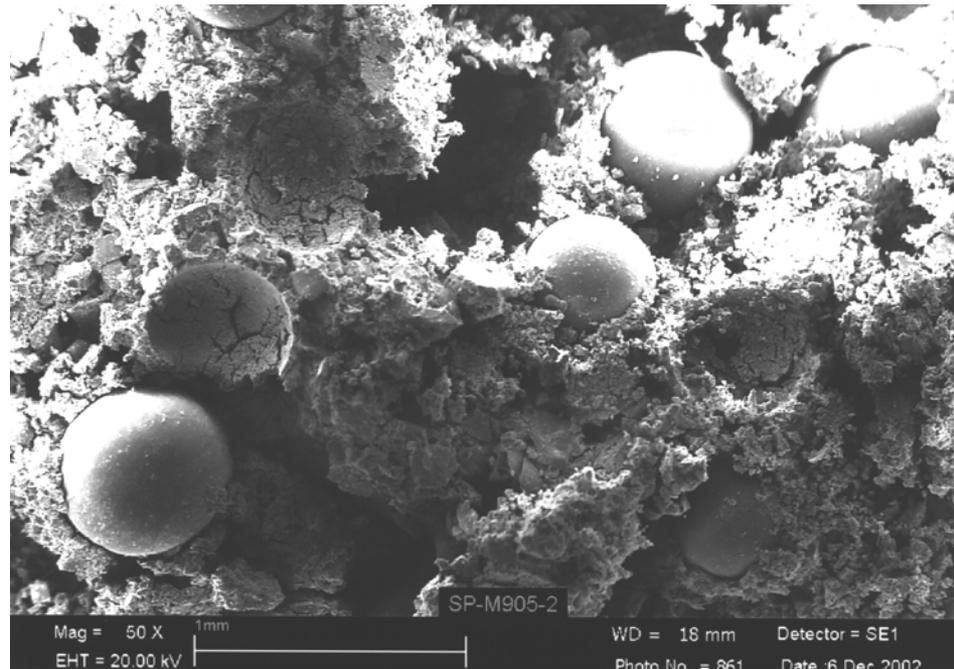
## Task 2 Progress – Binders 1 and 2

- **MSNW-developed, low CTE glass binder made via sol gel processing followed by a 500°C heat treatment**
  - Same sol utilized for Task 1
  - TEOS, ethanol, water, nitric acid, and titanium ethoxide  $\text{Ti}(\text{OC}_2\text{H}_5)_4$
  - Makes great spheres
  - Unsuccessful as a binder (high shrinkage, cracked, weak samples)
- **MSNW-developed, low CTE glass binder made via sol gel processing followed by a 1450°C heat treatment**
  - Commercial silica sol combined with titanium n-butoxide  $\text{Ti}(\text{OC}_4\text{H}_9)_4$
  - Intermediate stage consists of titania and silica particles; these are heated to 1450°C to form a binder by diffusion
  - 7.5 w/o  $\text{TiO}_2$  – 92.5 w/o  $\text{SiO}_2$  binder with near-zero expansion
  - Unsuccessful combined with MSNW spheres
    - Binder did not wet the spheres; samples were weak
  - Successful combined with aluminosilicate spheres
    - Binder wets the microballoons; samples are strong

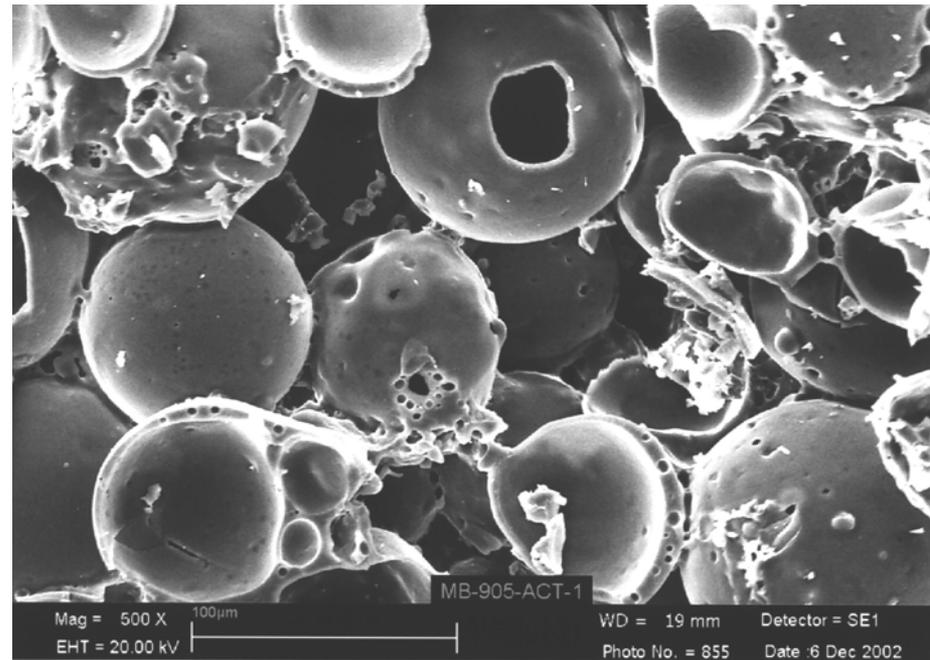
## Task 2 Progress – Binder 3

- **Silica – based cement and modifications**
  - CTE is low ( $0.3 \times 10^{-6}/^{\circ}\text{F}$ ); Flexural strength is 2100 psi
- **Three cement variations were combined with spheres**
  - Silica-Based Cement
  - Silica-Based Cement plus 25 w/o MSNW  $\text{TiO}_2 - \text{SiO}_2$  frit
  - Silica-Based Cement Activator
    - Contains alkali metal silicate - promotes wetting
- **First two variations did not wet either of the spheres; samples were weak**
- **Activator combined with microballoons**
  - Heat treated at  $1100^{\circ}\text{C}$
  - Activator wets the microballoons; sample is relatively strong

**Fracture Surface**  
**MSNW  $\text{TiO}_2$  –  $\text{SiO}_2$  Microspheres**  
**Bonded With Modified Silica-Based Cement.**  
**Note Absence of Wetting of the Microspheres by the Binder.**



**Fracture Surface**  
**Activator-Bonded Microballoons**  
**Binder 3 – 1100°C Heat Treatment**  
**Note Wetting of the Microballoons by the Binder**



## **Task 2 Progress – Binder 4**

- **MSNW No. 1 Binder Combined with Microballoons**
  - **Binder is a modified alkali metal silicate similar to previous “activator”**
  - **Heat treated at 900°C**
- **At This Point, Several Promising Binder/Sphere and Binder/Microballoon Combinations Had Been Manufactured and Characterized Utilizing SEM/X-ray**
- **CTE Testing Was Initiated to Screen the Materials**

### **Task 3 Progress – Initial Physical/Mechanical Property Evaluation of Bonded Arrays**

- **MSNW, Inc. Designed and Built A Laboratory CTE Screening Apparatus**
  - All promising bonded sphere combinations were tested
  - RT to 100°C
  - Multiple samples were tested of each composition
- **Using This and Other Data, the Following Downselect Criteria Were Applied**
  - Low CTE ( $10^{-6}$  in/in/°C or lower) screening data
  - Low Density (0.5 g/cc or lower)
  - Low Processing Temperature (1100°C max)
- **Two Systems Satisfied The Downselect Criteria**
  - Microballoons bonded with Activator
  - Microballoons bonded with MSNW No. 1 binder

## **Task 3 Progress – Initial Physical/Mechanical Property Evaluation of Bonded Arrays (Cont)**

- **Downselected Systems Sent to ATK/COI for Further CTE Testing**
  - **Successful (gritless) water jet cutting was performed by ATK/COI to produce required CTE sample geometry**
    - 6 in. by 1.75 in. by 0.1 in.
  - **Two samples of each system were tested from -100°C to +100°C**
- **MSNW, Inc. Screening Data Was Compared to ATK/COI Data**
  - **Aluminosilicate microballoons bonded with MSNW No. 1 binder**
    - $10^{-6}$  range for both data sets
  - **Aluminosilicate microballoons bonded with 905 activator**
    - Factor of 10 difference (ATK/COI data was  $10^{-6}$  ; MSNW data was  $10^{-7}$ )
  - **Will acquire third party data**
- **Newest Screening Data**
  - **20% additions of zirconium tungstate promising ( $10^{-7}$ )**

## Task 3 Progress – CTE Data for Aluminosilicate Microballoons Bonded With Activator

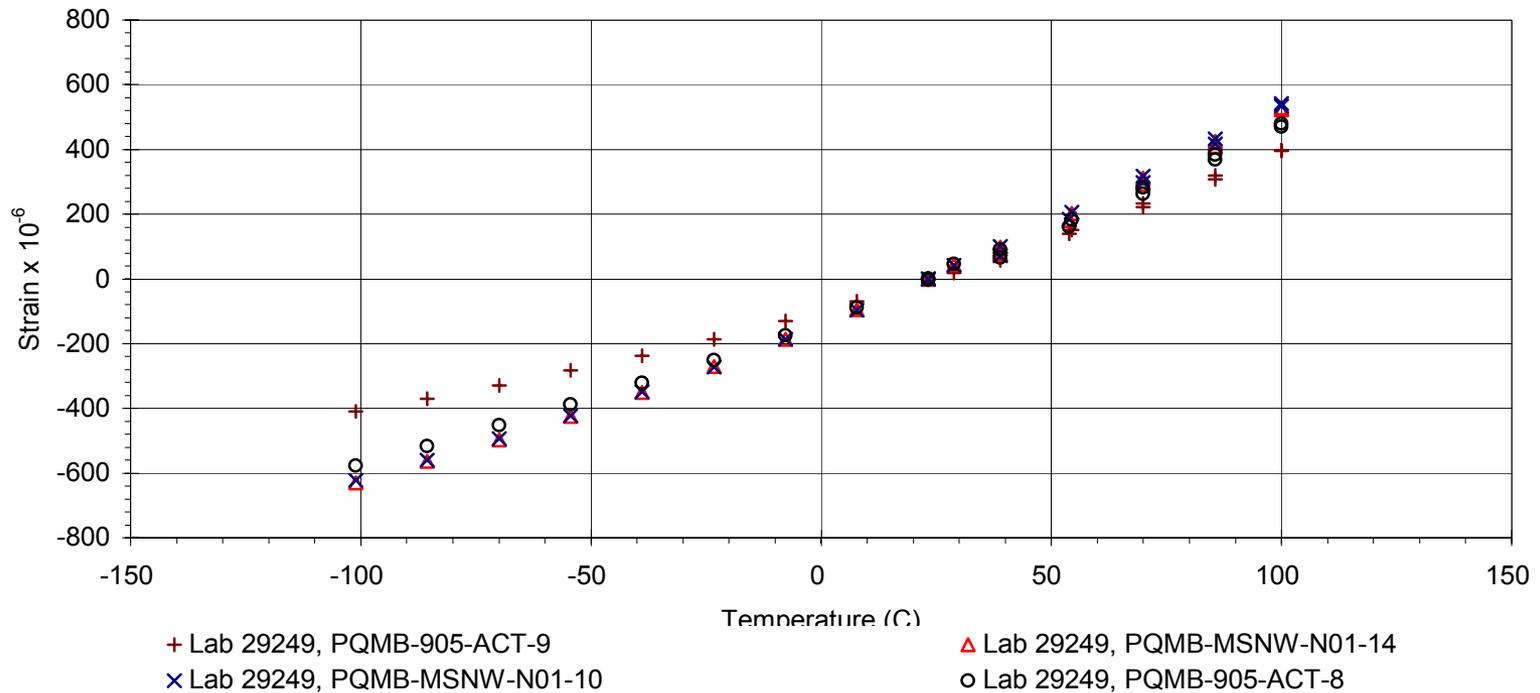
Sample Designation	Heat Treatment Temp (°C)	Time (Hr)	Atm (Air)	Density (g/cc)	RT to 100°C MSNW Screening CTE (in/in/°C)	RT to 100°C COI CTE (in/in/°C)	-100°C to RT COI CTE (in/in/°C)
PQMB-905 activator-1	1100°C	10	Air	0.457	$3.61 \times 10^{-6}$	-	-
	1250°C	4	Air	0.688	$6.13 \times 10^{-7}$	-	-
	1350°C	4	Air	0.701	$3.08 \times 10^{-7}$	-	-
PQMB-905 activator-2A	1100°C	10	Air	0.512	$7.45 \times 10^{-7}$	-	-
PQMB-905 activator-2B	1100°C	10	Air	0.513	$5.18 \times 10^{-7}$	-	-
PQMB-905 activator-7	1100°C	10	Air	0.518	$6.01 \times 10^{-7}$	-	-
PQMB-905 activator-8	1100°C	10	Air	0.401	-	$6.21 \times 10^{-6}$	$4.58 \times 10^{-6}$
PQMB-905 activator-9	1100°C	10	Air	0.502	-	$5.19 \times 10^{-6}$	$3.26 \times 10^{-6}$

## Task 3 Progress – CTE Data for Aluminosilicate Microballoons Bonded With MSNW No. 1 Binder

Sample Designation	Heat Treatment Temp (°C)	Time (Hr)	Atm (Air)	Density (g/cc)	RT to 100°C MSNW Screening CTE (in/in/°C)	RT to 100°C COI CTE (in/in/°C)	-100°C to RT COI CTE (in/in/°C)
PQMB-MSNW No. 1-1	900	10	Air	0.559	$1.46 \times 10^{-6}$	-	-
PQMB-MSNW No. 1-2	900	10	Air	0.556	$2.09 \times 10^{-6}$	-	-
PQMB-MSNW No. 1-3	900	10	Air	0.535	$6.14 \times 10^{-7}$		
PQMB-MSNW No. 1-4	900	10	Air	0.561	$1.74 \times 10^{-6}$	-	-
PQMB-MSNW No. 1-11	900	10	Air	0.540	$1.96 \times 10^{-6}$	-	-
PQMB-MSNW No. 1-10	900	10	Air	0.515	-	$7.01 \times 10^{-6}$	$4.97 \times 10^{-6}$
PQMB-MSNW No. 1-14	900	10	Air	0.519	-	$6.86 \times 10^{-6}$	$5.04 \times 10^{-6}$

## Thermal Expansion of Bonded Aluminosilicate Microballoon Substrates

### Task 3 Progress – ATK/COI CTE DATA



## **Summary**

- **MSNW, Inc. Has Successfully Fabricated**
  - **Low CTE TiO<sub>2</sub> – SiO<sub>2</sub> microspheres and a corresponding frit**
  - **Two bonded mirror substrate compositions demonstrating**
    - **Low CTE (10<sup>-6</sup> in/in/°C or lower)**
    - **Low density (0.5 g/cc or lower)**
    - **Low processing temperature (1100°C max)**
- **Task 3 Testing In Progress**

## **Future Work**

- **Immediate plans include**
  - **Continued use of zirconium tungstate additions**
  - **Continued CTE testing**
  - **Initial flexure and compression testing**
    - **Samples are currently being manufactured for testing at ATK/COI**
- **MSNW, Inc. is interested in teaming with a mirror component producer and continuing into Phase II**