Thermal Interface Materials:

I. Categorization, Developments, and Selection
II. Testing Methodologies and Test Systems

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Agenda:

I. Categorization, Selection, and New Developments for TIMs
    A. Categorization methodology for thermal interface materials (TIMs)
    B. Identification of TIM designations
    C. Recent developments of new materials in new TIM categories

II. TIM Industry Testing Methodologies and Test Systems

Addendum

Note: This presentation is focused primarily for materials useful for components and mil/aerospace systems. In addition, many TIM types described may be selected to meet other applications.

A. Categorization, Selection, and Developments for Thermal Interface Materials

Importance of Thermal Resistance: Power Semiconductors

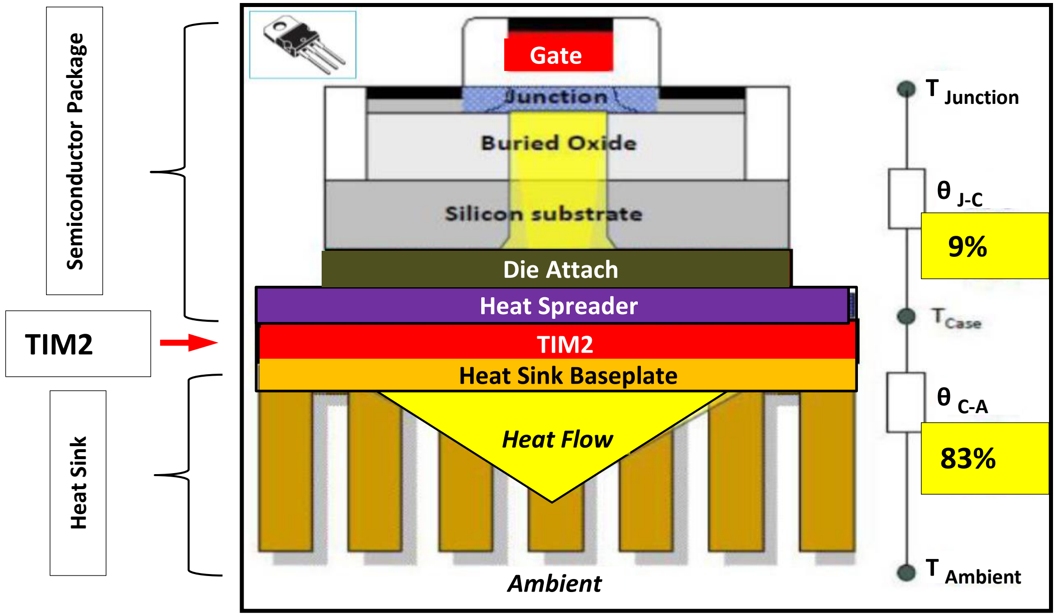


Figure 2 – TO-220 Thermal Resistance Stack

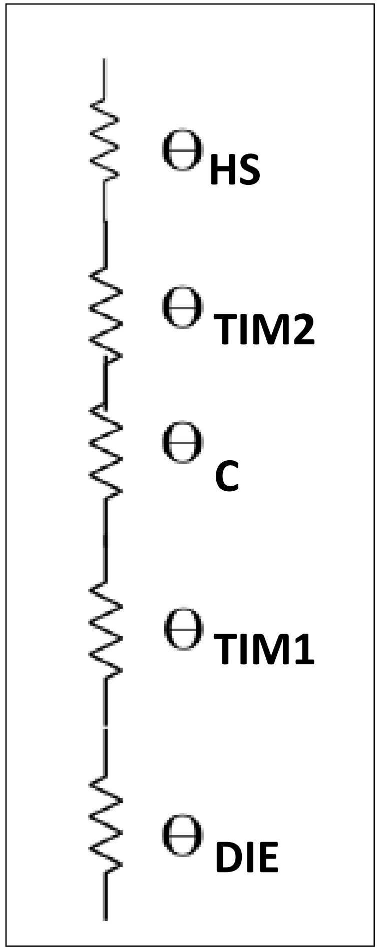
Empirical Analysis, TO-220 Package Materials Thermal Resistance Contribution

Material Layer	BLT (µm)	λ (W/mK)	Percent of Total (%)
Die (Si)	100	150	6
Die Attach (Solder)	20	50	3
Substrate (Cu)	200	380	2
TIM2 (Thermal Grease)	100	5	71
Heat Sink (Al)	2000	180	10
Other	-	-	8
Total	-	-	100

Data Source: Berliner Nanotest und Design GmbH (Germany). Used with permission.

Overview: Thermal Interface Material Function

System thermal management is the sum of a series of thermal resistances:



Heat sink: Inherent bulk material property – typically aluminum or copper (heat sink, liquid cold plate, vapor chamber)

TIM2: *External* to semiconductor package; $\theta_{\mathbf{T2}}$ is determined by material resistance, which consists of bulk value plus (2) contact resistances (case surface, heat sink)

Case (or lid): Inherent bulk material property – typically nickel-plated copper*

TIM1: *Internal* to semiconductor package; $\theta_{\mathbf{T1\text{-}C}}$ is determined by the material resistance which consists of bulk value plus (2)contact resistances (die surface, lid interior surface);

or, **TIM0**: Semiconductor package without lid (“bare die” package)

Die: Inherent bulk material property (Si, SiC, GaN, GaAs, other)

Note: Aluminum for low-power IC packages; aluminum or nickel-plated copper for “paddle” for TO-style power semiconductors and “top-side cooled” power semiconductor modules.

Overview: Thermal Interface Material Function

The primary performance value for a thermal interface material is thermal resistance per unit area (previously referred to as thermal impedance):

$^{\circ}C\text{-}in^2/W$ or $^{\circ}C\text{-}cm^2/W$ (also commonly used, $^{\circ}C\text{-}mm^2/W$)

- *Vendor data sheet performance values* are typically expressed as thermal resistance versus clamping force applied (range, typically 0-100PSI).
- In a performance graph, thermal resistance appears on the Y-axis versus clamping force on the X-axis.
- Increased clamping force has a large impact on thinning a TIM and improving (*lowering*) the thermal resistance value.
  - Values are typically asymptotic.
  - General function of a TIM is heat transfer across an interface.

Overview: TIMs for Specific Semiconductor Segments

Many different semiconductor package and module types are utilized across industry, with widely-varying TIM application requirements:

- Semiconductor segment, type, and package or module type will impact specific performance requirements, applicable TIM types, performance;
- Individual market segments have widely varying:
  - Package construction, type of heat source, relative heat flux
  - Current major market needs: (A) Very high performance *thin* TIMs
    (B) Thick high conductivity *gap-filling* TIMs

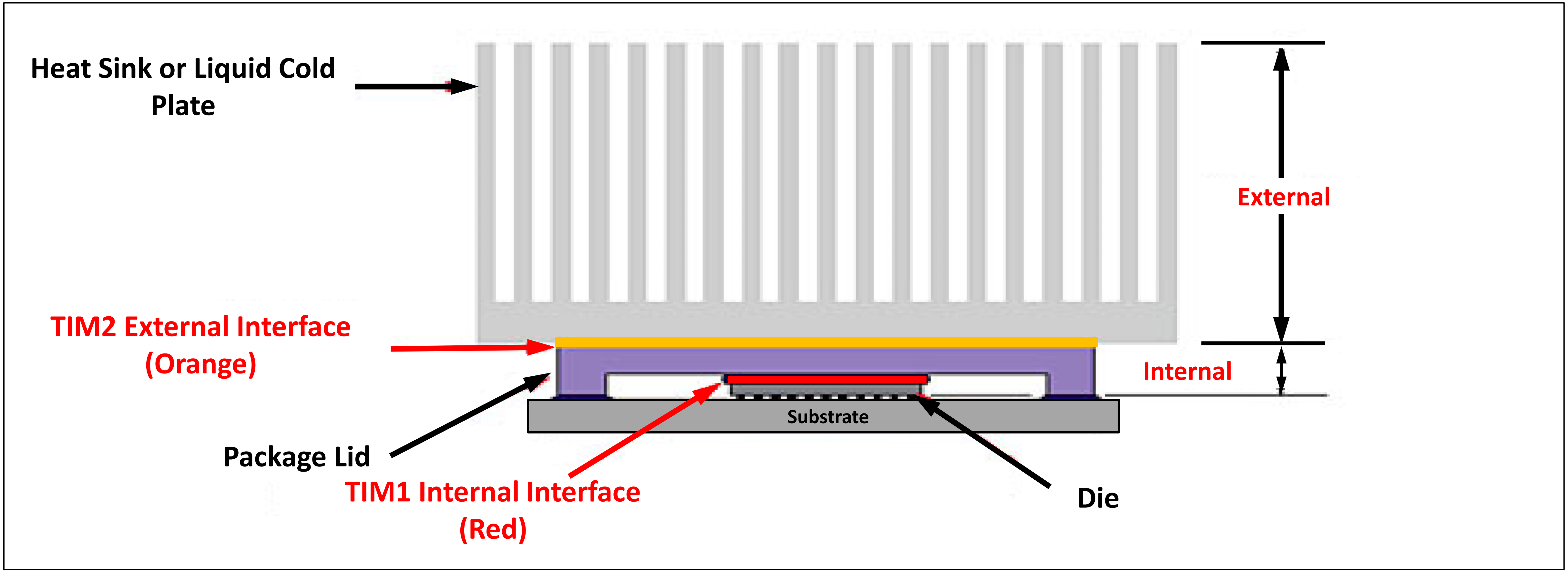
Example: Military X-band radar modules -- extremely high cost and test is critical at different stages of module assembly -- with reworkability a critical requirement, which drives TIM selection criteria.

- Major components requiring TIMs: diode lasers, power, RF, ICs, EV batteries, photonics

TIM Terminology

Proper use of terminology is important:

- All applications *external to the semiconductor package* are TIM2:



Note: The earlier term adopted as "TIM 1.5" was replaced with "TIM 0" twenty years ago. The three standard terms are TIM0, TIM1, TIM2.

Source: Boteler, L., "Thermal Design of Power Electronics," US Army RDECOM Army Research Laboratory, APEC 2019 Conference, Tutorial (March 2019).

TIM Terminology

Thermal Interface Material Application Terminology		
Package Level	**Generally Accepted Definition**	**TIM Terminology**
1	Semiconductor die to heat sink (external, bare die package)	TIM0
1	Semiconductor die to package lid (internal, lidded package)	TIM1
2	Semiconductor lid, case, or baseplate: external to the package, conducting heat to a heat sink, liquid cold plate, or metal component	TIM2
3	Conducting heat from one or more components across a relatively large gap to a metal component. Material thickness $\geq$ 250µm (0.10" (typ.)	Gap-filler
4	Platform or subassembly level, conducting heat from the case of a power supply or other large module, large heat sink, metal component	Gap-filler

TIM Terminology

Proper use of terminology is important:

- Intel/AMD use of *reflowed indium solders* for TIM1 (within the semiconductor package) is a separate category of TIM application.
  - The term “Solder TIM (STIM)” is appropriate only to these packages.
  - The term “STIM” is *not appropriate* for use in other TIM applications and only creates confusion.
  - A *Solder TIM* has different formulation and requirements than a *liquid metal TIM*.
- Die attach materials are not categorized as TIMs.
- Solders are otherwise not categorized as TIMs.

Note: All solders and die attach materials generally have thermal characteristics and provide a heat flow path -- but are not considered in the general terminology usage as thermal interface materials (TIMs). Solders and die attach materials are selected by different criteria as the primary function is electrical interconnect. In very limited cases, a die attach gel has been used as a TIM1.

TIM and Thermal Performance Terminology

Thermal Interface Material Performance Terminology		
Term	**Generally Accepted Definition**	**Value (Typ.)**
Thermal Resistance (Bulk)	Barrier to flow of heat from heat source through a material or component	°C/W
Thermal Resistance (Interfacial)	Barrier to the flow of heat at the surface of a component	°C/W
Thermal Resistance (Contact)	Alternative term for interfacial thermal resistance (per above)	°C/W
Thermal Resistance (per unit area)*	Barrier to the flow of heat through a material, per unit area (most useful value for selecting a TIM)	°C-in^2/W (or) °C-cm^2/W
Thermal Impedance	Alternative term for thermal resistance per unit area	°C-in^2/W (or) °C-cm^2/W
Heat Flux (Heat Density)*	Amount of power dissipated per unit area (e.g., from a point on the surface of a processor die or across the baseplate of a GaN RF device)	W/in^2 (or) W/cm^2

Note: The above terminology may be used casually and identifying the most useful term is important for selecting a TIM to propose for a given application. The most important term for determining performance of a TIM is thermal resistance per unit area, marked above with an asterisk (*).

TIM Categorization System

General Functions and Categories of Thermal Interface Materials		
Primary Function	**Material Category**	**Sub-Categories**
Large (≥250µm) air gaps	**Gap-fillers**[1]	▪ Die-cut sheet or preform ▪ Dispensable liquid
Electrically non-conductive	**Dielectric**[1]	
Large area: X-Y-direction heat flow	**Graphite Sheets**[2]	▪ Many different, generally poor Z
Large area: Z-direction heat flow	**Elastomeric Sheets**[1]	▪ Many different, generally poor conductivity
Adhesive TIM attachment	**Thermally-conductive adhesives***[1]	▪ Pressure-sensitive adhesives (PSAs) ▪ Curable or two-part dispensed
Die-to-lid (internal to package)	**TIM1**[#1, 3]	▪ Die-attach (DA) Adhesives (used as TIM1) ▪ Gels ▪ Phase-change, Thermal greases ▪ Reflowed soft solders (indium), liquid metals (gallium) ▪ VA-CNT (carbon nanotubes), VA-CNF carbon (fibers)
Thin multipurpose (TIM0/1#/2)	**Thermal greases (compounds)**[1]	▪ Many thermal filler percentages (user beware)
	Phase-change compounds[1]	▪ Many different PC temperatures, formulations

*Notes: * Pressure-sensitive adhesives (PSAs) preforms may be manufactured with a carrier, with adhesive coating on two surfaces. # TIM1 selection is determined by the semiconductor device manufacturer.*

Key: 1. Polymeric. 2. Graphitic. 3. Metallic.

TIM Categorization System

General Functions and Categories of Thermal Interface Materials		
Primary Function	**Material Category**	**Sub-Categories**
Very high performance	**Graphene-enhanced graphite[2]**	▪ Graphene-enhanced graphitic preforms
	Carbon-Based Arrays[2]	▪ VA-CNF: Vertically-aligned carbon fiber arrays ▪ VA-CNT: Vertically-aligned carbon nanotube arrays
	Metallic[3]	▪ TIM0/1[#] Liquid metal (LM) ▪ TIM0/1[#] Liquid metal/solid hybrids (LMH) ▪ TIM1[#] Reflowed soft solders (STIM, indium) ▪ TIM0/1[#]/2 Phase-change metal alloys (PCMA) ▪ TIM2 Metallic preforms (flat foils, patterned foils)

*Notes: * Pressure-sensitive adhesives (PSAs) preforms may be manufactured with a carrier, with adhesive coating on two surfaces. # TIM1 selection is determined by the semiconductor device manufacturer.*

Key: 1. Polymeric. 2. Graphitic. 3. Metallic.

TIM Categorization System

Thermal Interface Material Categorization and Evaluation

Prioritized TIM Requirements	Property	Typical Value		Alternative/Opposing Value	
1. Electrical	Dielectric Properties	Electrically Non-Conductive		--	
2. Mechanical	Fastening	Mechanical Fasteners Required		Adhesive	
	Thickness	Minimum: Highest Performance		Maximum (≥ 250µm): Gap-filling	
	Surface Roughness, Flatness, Warpage	Minimum Specification		Maximum Specification	
3. Application Process	Dispensing/Placement	Automated, Semi-auto		Manual	
4. Thermal	Thermal Resistance	Minimum	Maximum	Minimum	Maximum
	Operating Temperature Range	Minimum	Maximum	Minimum	Maximum
	UL Flammability Rating	UL V-0		(Not specified)	
5. Cost	Material only/application process/total cost	Material (only)		Application process cost, shipping requirement cost	
6. Health and Safety, Climate and Environmental	Constituent analysis: silicones, toxicity, environmental, H&HS	Government, industry, company regulations			

TIM Performance

Determining performance for selecting an appropriate TIM:

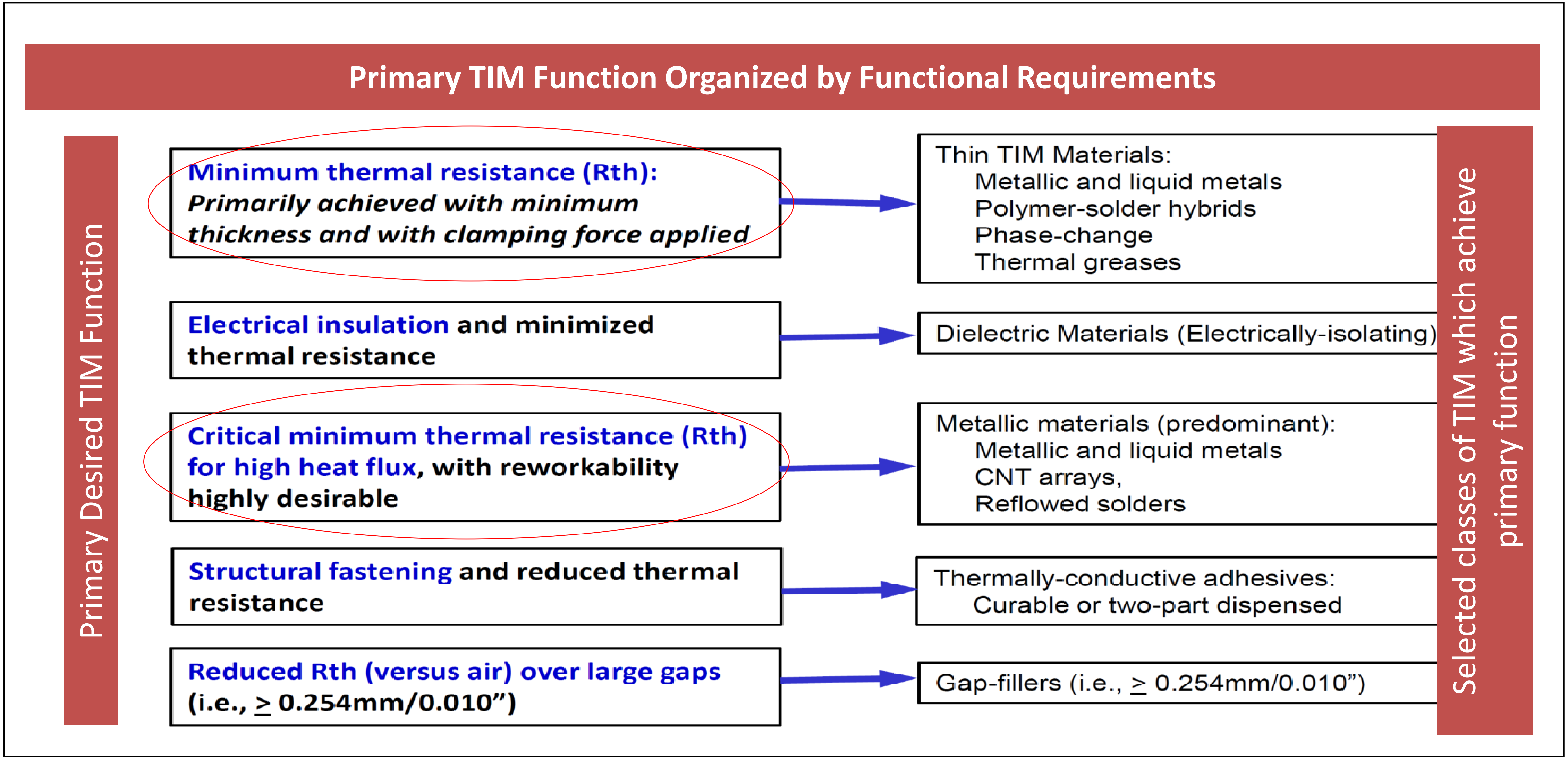
- Clamping force uniformly applied is intended to achieve:
  - Maximized surface wetting;
  - Thinnest possible TIM thickness (to minimize bulk thermal conductivity);
- Degree of surface wetting achieved is critical to overall performance, to minimize contact thermal resistance at each of two contact surfaces.
  - *Contact resistance dominates TIM bulk resistance for most materials.*
  - *Achieving the thinnest possible thickness with highest clamping pressure is critical to achieving minimum thermal resistance.*
- Relatively good bulk thermal conductivity is needed when only limited clamping force and sufficient surface wettig are available.

TIM Selection Methodology

Thermal performance is not the only criteria for selection of a TIM.

- A *holistic application view* may require a specific temperature range, application process, or reliability requirement that determines material selection, over thermal performance;
- As a result, the single *lowest thermal resistance* material or the material having the *highest bulk thermal conductivity* may not be selected.
- A material must meet thermal resistance *and* all other requirements: material compatibility, wetting, compression, rheology, assembly procedures, reliability, product life.

TIM Selection Methodology



Primary TIM Function Organized by Functional Requirements

Primary Desired TIM Function	Selected classes of TIM which achieve primary function
Minimum thermal resistance (Rth): ***Primarily achieved with minimum thickness and with clamping force applied***	Thin TIM Materials: Metallic and liquid metals Polymer-solder hybrids Phase-change Thermal greases
Electrical insulation and minimized thermal resistance	Dielectric Materials (Electrically-isolating)
Critical minimum thermal resistance (Rth) for high heat flux, with reworkability highly desirable	Metallic materials (predominant): Metallic and liquid metals CNT arrays, Reflowed solders
Structural fastening and reduced thermal resistance	Thermally-conductive adhesives: Curable or two-part dispensed
Reduced Rth (versus air) over large gaps (i.e., ≥ 0.254mm/0.010”)	Gap-fillers (i.e., ≥ 0.254mm/0.010”)

TIM Selection Methodology

Selecting an appropriate thermal interface material:

- *Degree of surface wetting* achieved is critical to overall performance, to minimize contact thermal resistance at each of two contact surfaces.
  - Contact thermal resistances dominate TIM total thermal resistance value for most materials that are thin by design;
    - Exception: Gap-filler bulk thermal resistance will dominate over contact thermal resistance, due to intentional extreme thickness.
    - Certain of these factors (such as filler percentage by volume) can impact the ability of a TIM to move and not suffer “run-out” (highly thixotropic), affecting relative *thermal performance* and *reliability over time*.
  - Driving to *highest wetting* and *thinnest clamped* thickness is *critical to successful TIM selection* in traditional TIM applications.

Major TIM Categories

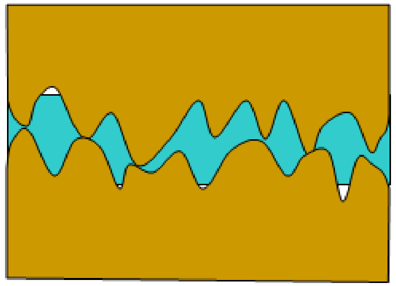
Presentation Note: Slides in this section are included for future review by those interested, but the majority will not be covered in today's oral presentation.

Gap-Fillers: Where Are These Needed?

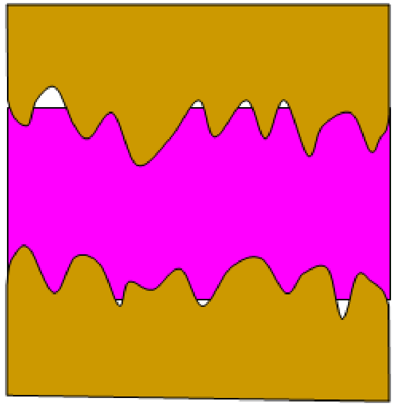
One general definition for TIMs versus gap-fillers:

- Thermal Interface Materials: Replace air in an interface that would otherwise present due to <u>surface roughness;</u>
  - <u>Thinnest TIM is typically best performer</u>
- Thermal Gap-fillers: Replace air in an interface that would otherwise be present due to <u>tolerance stack-up.</u>
  - Typically defined as <u>>250µm thickness</u>

Note that these are very generalized definitions.



Thermal Interface Material
(TIM)



Thermal Gap Filler
(TGF)

Source: Ross Wilcoxon PhD, Engineering Fellow, Advanced Technology Center, Collins Aerospace Inc., Cedar Rapids IA USA. Unpublished; Used with permission.

TIMs for High Temperature Applications

Higher operating temperature can severely restrict available thermal solutions:

- May lead to increased focus on TIM *performance,* if the available thermal operating range is reduced;
- Requires selection of high-temperature-capable TIMs matched to specific expected application operating temperatures;
- Higher ambient temperature → higher device junction temperature;
- Ambient, junction, and processing temperature ranges will dictate parameters for suitable TIM selection.

Graphite Sheet Heat Spreaders

Graphite Sheet Heat Spreader Materials				
Vendor	**Product Designation**	**Thickness (µm)**	**Bulk Thermal Conductivity**	
			X-Y axis W/mK	**Z-axis W/mK**
DSN (China)	DSN5017	17	1600-1900	15-20
TTCL (China)	TGS-17	17	1700	15
Panasonic (Japan)	PGS EYG-S-25	25	1600	N/A
NeoGraf (US)	eGraf® SpreaderShield Flexible Graphite SS1500	17	1500	3.4
Panasonic (Japan)	PGS EYG-S-100	100	700	N/A
NeoGraf (US)	eGraf® SpreaderShield Flexible Graphite SS600	127	600	3.5
NeoGraf (US)	eGraf® HiTHERM™ 700	127	240	6

Data Source: Vendor presentations and technical data sheets, DS&A LLC.

Carbon Nanotube Materials

Developments with vertically-aligned carbon nanotube TIMs (VA-CNTs):

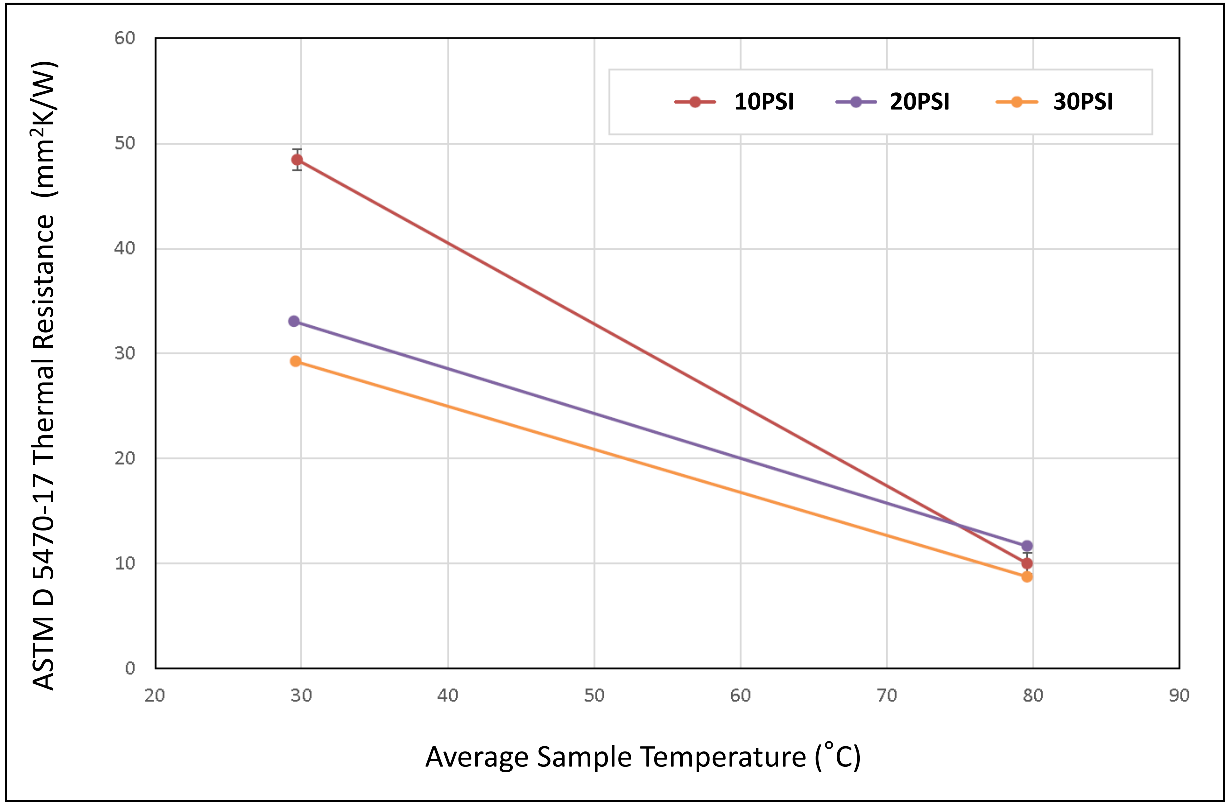
- Advantages:
  - Perceived high bulk thermal conductivity of CNTs
- Disadvantages:
  - Significant difficulties in developing a manufacturable TIM product
  - Development materials in some cases have very low bulk thermal conductivity
  - High perceived manufacturing cost
- Fujitsu:
  - Tested values to date for bulk thermal conductivity (Z): 10-20W/mK

VA-CNT and VA-CNF TIMs are generally high temperature-capable (depending on carrier utilized).

Phase-Change Materials

Phase-change compounds have been manufactured for use as TIMs for more than thirty years and are well-known for TIM2 applications:

- Compounds and pre-forms are available with phase-change temperatures from 45°C to 60°C:
  - Purpose of the phase-change temperature is to achieve thickness change to minimize resistance, at a given pressure (graph)
- Bulk thermal conductivity values are available in a wide range (depending on formulation): 0.6W/mK – 8W/mK
- Certain new materials are available that utilize single-side and double-sided compound coatings on carriers:
  - Aluminum (dead soft)
  - Durable graphite films (certain manufacturers)
  - Dielectric films (i.e., DuPont™ Kapton® MT, MT+)
- Application of a TIM pre-form with single-side coating in a test system to face the test head prevents marking or detritus on the DUT.
- Phase-change TIMs are high temperature limited (specifically depending on phase-change designed temperature and formulation characteristics).



Source, Graph: Berliner Nanotest und Design GmbH (Berlin, Germany). Well-known phase-change preform TIM (8µm initial thickness, prior to application of pressure).

High Performance Metallic TIMs: Indium Foils

Indium flat foils used as TIM2 for decades, for very specific applications:

- Flange-mount RF discrete power amplifiers and modules (telcom, radar, radio communications, satellite communications);
- Standard TIM2 for diode laser arrays[1];
- Reflowed indium solder as a high-volume TIM1 for server processors;
- TIMs and indium foils as seals for cryogenic systems;
- Semiconductor test and burn-in;
- Current research for high-volume BGA processor packages[2].

Notes: 1. *Koechner, W.,* Solid-State Laser Engineering, *6th Edition (Springer Verlag, 2006).*

2. *Koh, Y. J.; Kim, S.H.; Sohn, E.S.; Khim, J.Y.; Amkor Technology Korea Inc., "Thermal Performance of Advanced TIMs for High-Power FCLBGAs," IEEE ECTC Conference 2022, San Diego CA USA, May 31, 2022.*

High Performance Metallic TIMs: Patterned Metal Alloy Foils

Developments by Indium Corporation for indium and metal alloy foils as TIMs include:

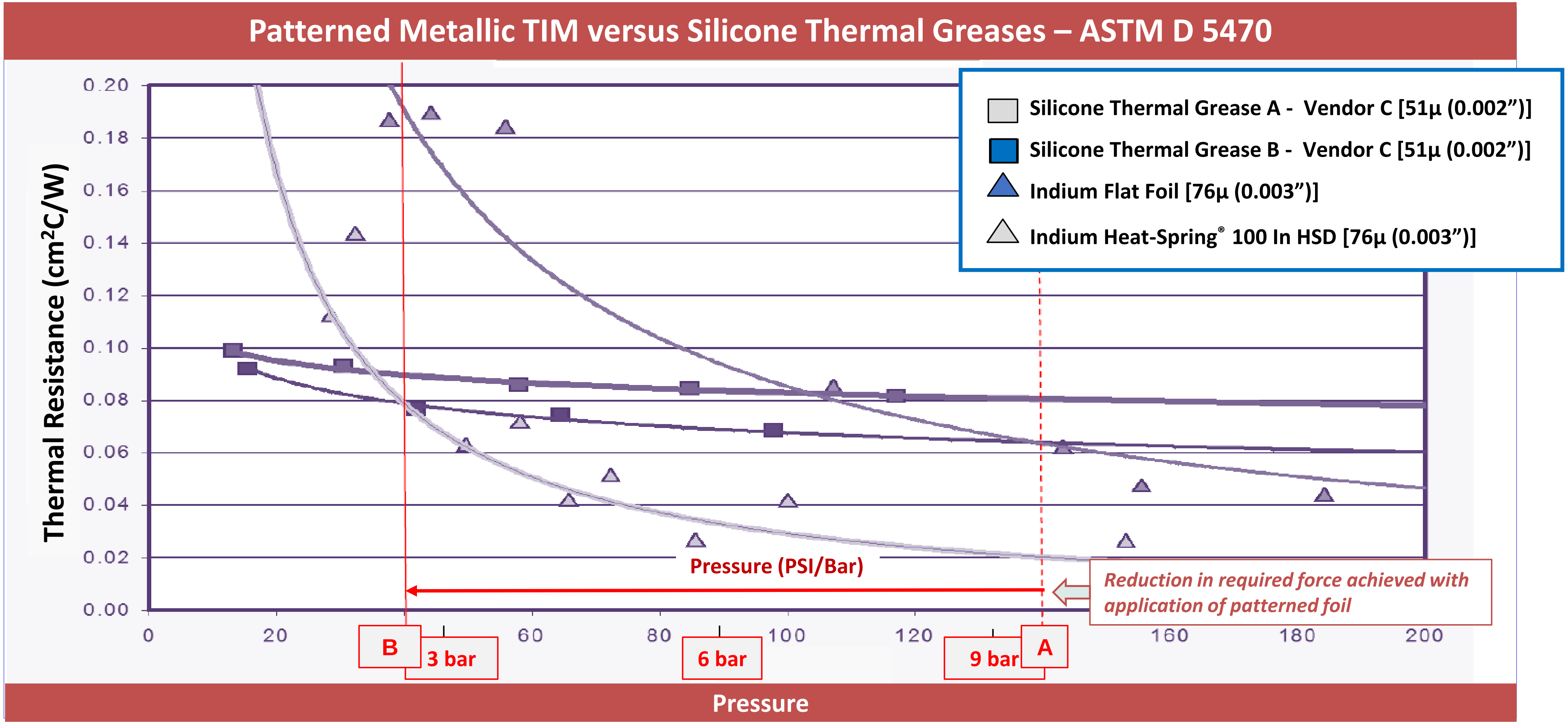
- "Heat-Spring®": Application of patterning to improve gap-filling and improve thermal performance for uneven surfaces;
- Laminated aluminum foil (5µ thickness, typ., one surface) available for:
  - Eliminating "tackiness" of indium metal on contact
  - Eliminate potential for residue in contact cycling for semiconductor test;
- Expansion of range of metal alloys available with above enhancements;
- Applications for gimbaled test heads and higher temperature testing.
- Taller patterns to handle greater die warpage and worse non-flat surfaces.
- Metallic TIMs may be temperature limited depending on alloy selected.

Note: "Heat-Spring" is a Registered Mark of Indium Corporation. US Patent 7,593,228-B2.

High Performance Metallic TIMs: Patterned Metal Alloy Foils

Comparative test data: indium flat foils vs. Indium "Heat-Spring" patterned In100 foil and thermal greases:

- Improvement: Patterning vs. flat indium foils, greases at > 40PSI (Note force reduction from points A to B)



Data Source: Indium Corporation. DS&A LLC Model 101 ASTM D5470-12 Test Stand. "Heat-Spring" is a registered mark of Indium Corporation.

High Performance Metallic TIMs: Patterned Metal Alloy Foils

Bulk thermal conductivity and suggested maximum operating temperatures for metallic TIMs:

Maximum Bulk Thermal Conductivity and Suggested Operating Temperature for Metallic TIMs		
Metallic TIM Composition	Bulk Thermal Conductivity (W/mK)	**Suggested Maximum Operating Temperature (°C)**
52In/48Sn Indalloy 1E	34	100
80 In/20 Sn	53	110
100 In	86	125
In/Al Clad	-	125
Sn, “Sn+”	73	200
100 Pb	35	250
100 Cu	395	750

Table shows suggested values for selected metals and alloys; other alloys are possible.

- Characteristics of interface surfaces may affect maximum temperature.

*Notes: * “Indalloy”, “Sn+” are Indium Corporation products. Data Source: R. Jarrett, Indium Corporation , Utica NY USA; Bulk conductivity values, G. Wilson, Indium Corporation, Milton Keynes UK.*

High Performance Metallic TIMs: New Developments

Examples: Relative bulk thermal conductivity values, development metallic TIMs of different types:

Bulk Thermal Conductivity Values – Metallic TIMs

Basis[1]	Category	Type (Typical Intended Usage)	Value (W/mK, Typ.)
Indium Based	Solid	Solder TIM (TIM1)	70-86
		Compressible TIMs (Patterned, TIM2)	86
	Phase-Change	Phase-change metal alloy TIMs (TIM2)	40-50
Indium/Gallium[2]	Hybrid Liquid Metal	Indium® m2TIM™ (TIM1)	40-50
		Liquid metal pastes (TIM0, TIM1, TIM)	15-25
Gallium Based[2]	Liquid	Liquid metal TIMs (TIM0, TIM1)	20-45

Notes: 1. Primary metal by percentage.   2. Generalized statements regarding intended usages shown in parentheses. Multiple materials available from suppliers.

Source: Adapted from: Miloš Lazić, Indium Corporation, "Advanced Gallium-Based Thermal Interface Materials," IMAPS New England Symposium 49, Boxborough MA USA, May 2, 2023.

High Performance Metallic TIMs: Liquid Metal Developments

Characteristics of Liquid Metal TIM Alloys

Composition	Melting Point (°C)	Density (g/cm³)	Specific Gravity	Thermal Conductivity (W/mK)
61.0Ga/25.0In/13.0Sn/1.0Zn	7.6	6.50	6.50	15*
66.5Ga/20.5In/13.0Sn**	10.7	6.32	6.50	16.5
62.5Ga/21.5In/16.0Sn	16.3	6.50	6.50	16.5
75.5Ga/24.5In	15.7	6.35	6.35	26*
95Ga/5In	25.0	6.15	6.15	25*
Base Elemental Properties				
100Ga	29.78	5.90	5.904	31
100Sn	235	7.28	7.28	73
100In	157	7.31	7.31	87

*Notes: * Estimated value. ** Tradename "Gallinstan", Geratherm Medical AG. Sources: (1) Geratherm Medical AG, Material Safety Data Sheet, 93/112/EC, 2004. (2) Michael D. Dickey, et al., "Eutectic Gallium-Indium (EGaIn): A Liquid Metal Alloy for the Formation of Stable Structures in Microchannels at Room Temperature, "* Advanced Functional Materials, *2008, 18, 1097-1104. (3) C.Y.Ho, et al., "Thermal Conductivity of the Elements,"* Journal of Physical Chemical Reference Data, *Vol. 1. No. 2, 1972. (4) Charles Kittle,* Introduction to Solid State Physics, *7th Ed., Wiley and Sons, 1996.*
Source: Adapted from: Jensen, T., Indium Corporation, "Innovative Metal TIM Technology for High Performance Computing." Semi-Therm 39 Symposium, San Jose CA USA, March 2023.

High Performance Metallic TIMs: Liquid Metal Developments

Applicability of liquid metal TIMs for semiconductor test:

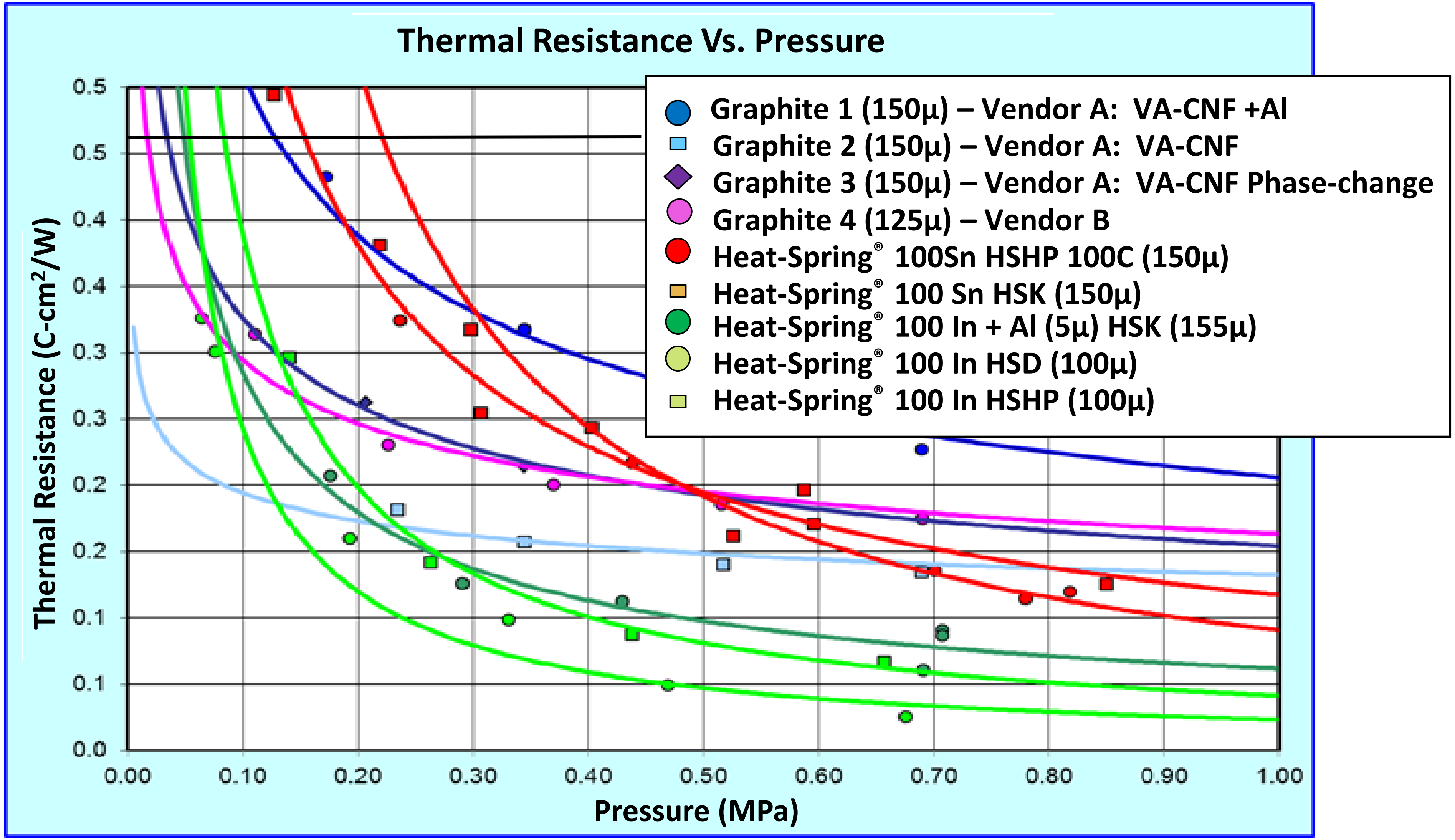
- High relative thermal conductivity and excellent wetting characteristics;
- A very thin bond line can be achieved without reflow;
- Liquid metals must be contained, to prevent spread during application;
- Once applied, surface tension will typically hold material as desired;
- Varying metal constituencies will allow tailored melting points as required;
- Metals listed are not known to be toxic but care in handling is required;
- Liquid metals can be jetted to form dots on the intended surface;
- Significant global development interest for TIM0/TIM1 applications;
- Suitability for traditional semiconductor test applications has not been demonstrated, given contact/release and no-marking requirements.

Vertically-Aligned Carbon Fiber (VA-CNF) TIMs

VA-CNF				
Vendor	**Product Designation**	**Thickness (µm)**	**Bulk Thermal Conductivity**	
			X-Y axis W/mK	**Z-axis W/mK**
Dexerials (Japan)	EX20200XX Gap-filler	100-200	N/A	15-20
NeoGraf (US)	Grafoil® GTA-005, GTA-030	130-760	140	5.5-7.0
Hitachi (Japan)	TC-001	150-500	N/A	40-90

Data Source: Vendor presentations and technical data sheets, DS&A LLC.

Metal Alloy TIMs vs. VA-CNF and Graphite Films

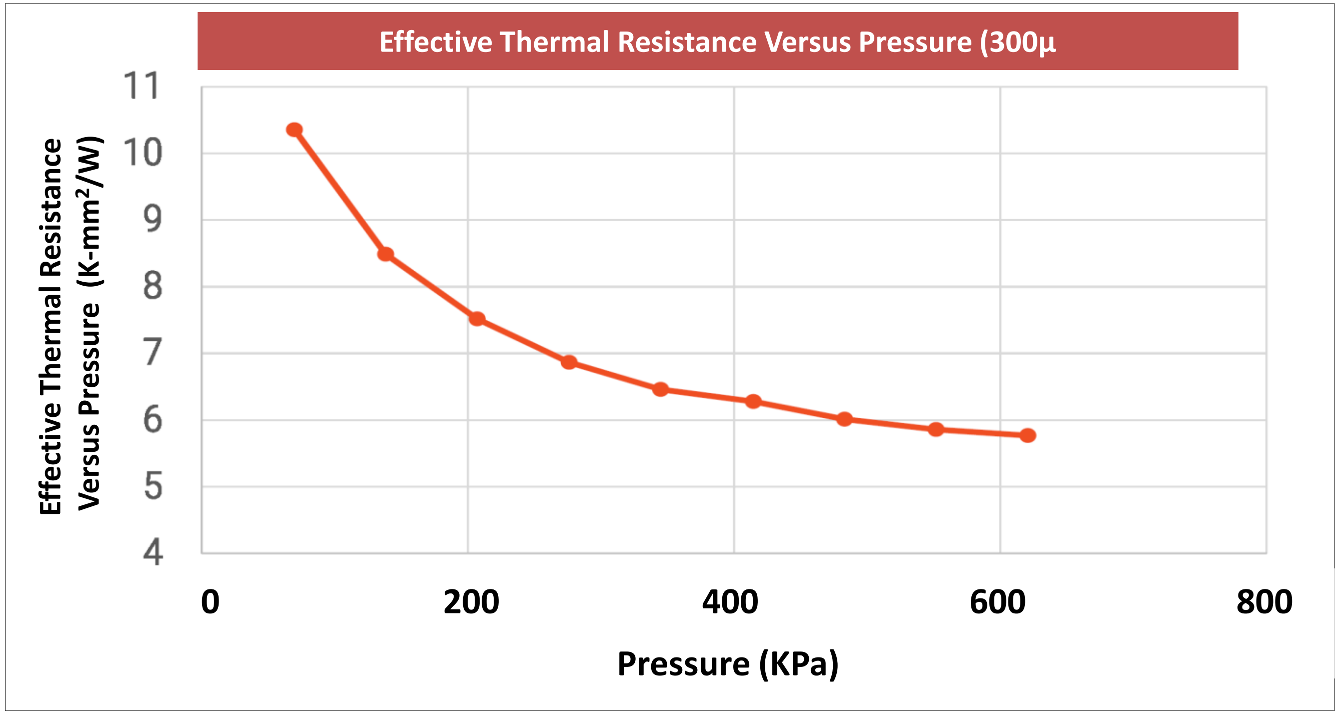


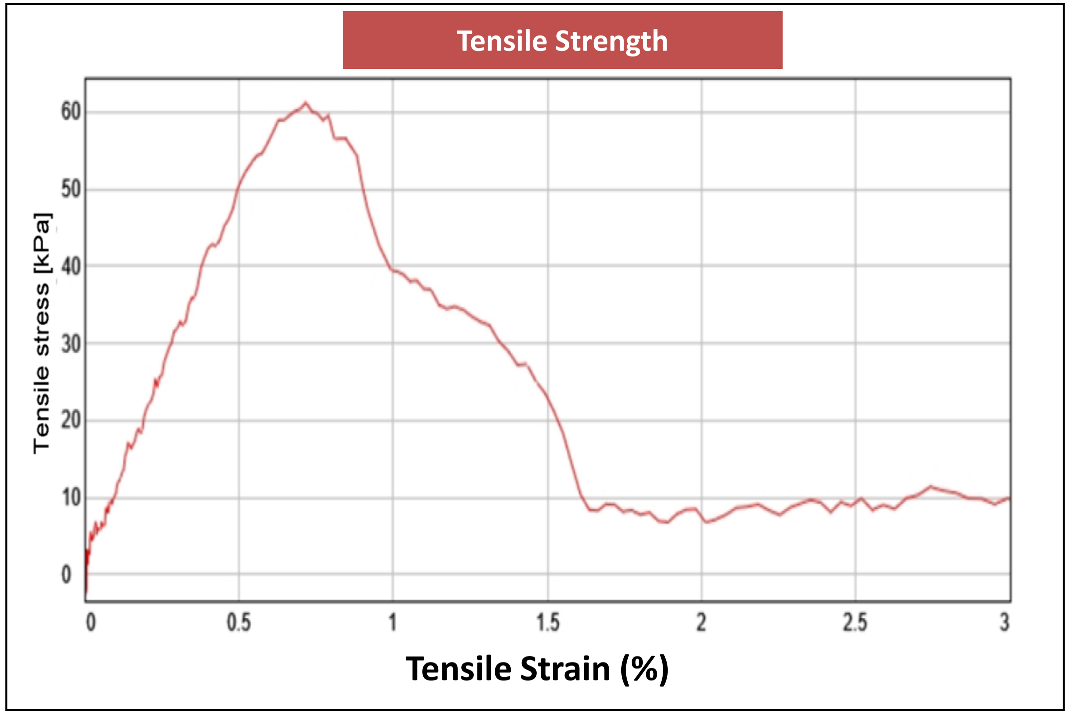
Data Source: Indium Corporation. DS&A LLC Model 101 ASTM D5470-12 test stand.

Graphene-enhanced Graphitic Materials

New TIM developments include graphene-enhanced performance of graphite films:

- SHT "FrostSheet" is an example of such a newly-developed thermal material



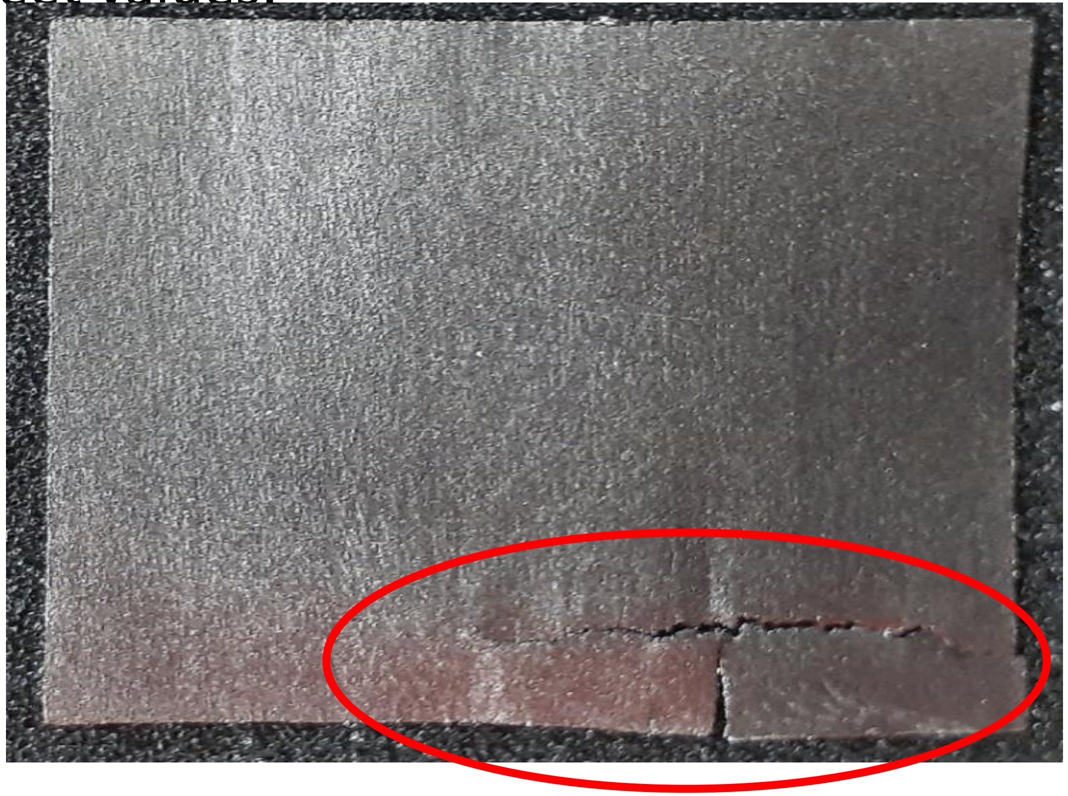


Source: Murugesan, M.; Martinson, K.; Enmark, M.; Zhang, H.; Liu, J.; Almhem, L., "Applications of High Thermal Conductivity Graphene Enhanced Thermal Interface Materials," Smart High Tech AB, IMAPS France Thermal and Micropackaging Workshop 2023, Poitiers, France, March 8-9, 2023.

Graphene-enhanced Graphitic Materials

"Graphene-enhanced" performance of materials such as graphite films:

- SHT FrostSheet is an example of such a newly-developed thermal material;
- Such materials include developments for both TIMs and heat spreaders;
- SHT FrostSheet and GT-TIM GT-90SPRO data sheet values:
  - Bulk through-plane thermal conductivity: 90+/- 10 W/mK
  - Thickness: 300µm (0.012")
  - Fragile, relatively thin graphite materials
  - Subject to easy handling damage
  - No testing to date by DS&A to confirm test values.



Source: Murugesan, M.; Martinson, K.; Enmark, M.; Zhang, H.; Liu, J.; Almhem, L.; Smart High Tech AB, "Applications of High Thermal Conductivity Graphene Enhanced Thermal Interface Materials," IMAPS France Thermal and Micropackaging Workshop 2023, Poitiers, France, March 8-9, 2023. Photograph: Smart High Tech AB (Göteborg, Sweden) "FrostSheet" enhanced graphite film: DS&A LLC (January 16, 2024).

Summary of New Material Developments

High Performance Commercial TIM Materials - Examples			
TIM Classification	**Vendor**	**Product**	**Status**
Thermal Grease	Shin-Etsu/Japan	Gallium silicone grease	Commercial product
	Sumitomo/Japan	Nanoparticle Ni/Fe grease	(Indeterminate)
Metallic TIM	Enerdyne Corporation/USA	Liquid indium alloy on carrier	(Indeterminate)
	Indium Corporation/USA	Heat-Spring® patterned TIM	Commercial product
	Indium Corporation/USA	Indium alloy foil	Commercial product
	AIM/Canada	Indium alloy foil	Commercial product
	Kester/USA	Indium alloy foil	Commercial product

Summary of New Material Developments

High Performance Commercial TIM Materials - Examples			
TIM Classification	**Vendor**	**Product**	**Status**
Aligned Carbon Fiber/Polymeric Matrix Carrier Preform	Btech Corporation/USA	Graphite fiber/polymeric carrier preform	Commercial product (Current status N/A)
	DuPont E&C/USA	Carbon fiber vertical array/polymeric carrier preform	Development
	Honeywell Electronic Materials/USA	Graphite fiber/polymeric carrier preform	Commercial product (withdrawn)
	Hitachi/USA	Carbon fiber vertical array/polymeric carrier preform	Commercial product
CNT-Based	SHT AB/Sweden	Vertically-aligned CNT array in polymeric carrier	Development
	Carbice/Georgia Tech/USA	Infinity™ Vertically-aligned CNT-array in polymeric carrier	Commercial product

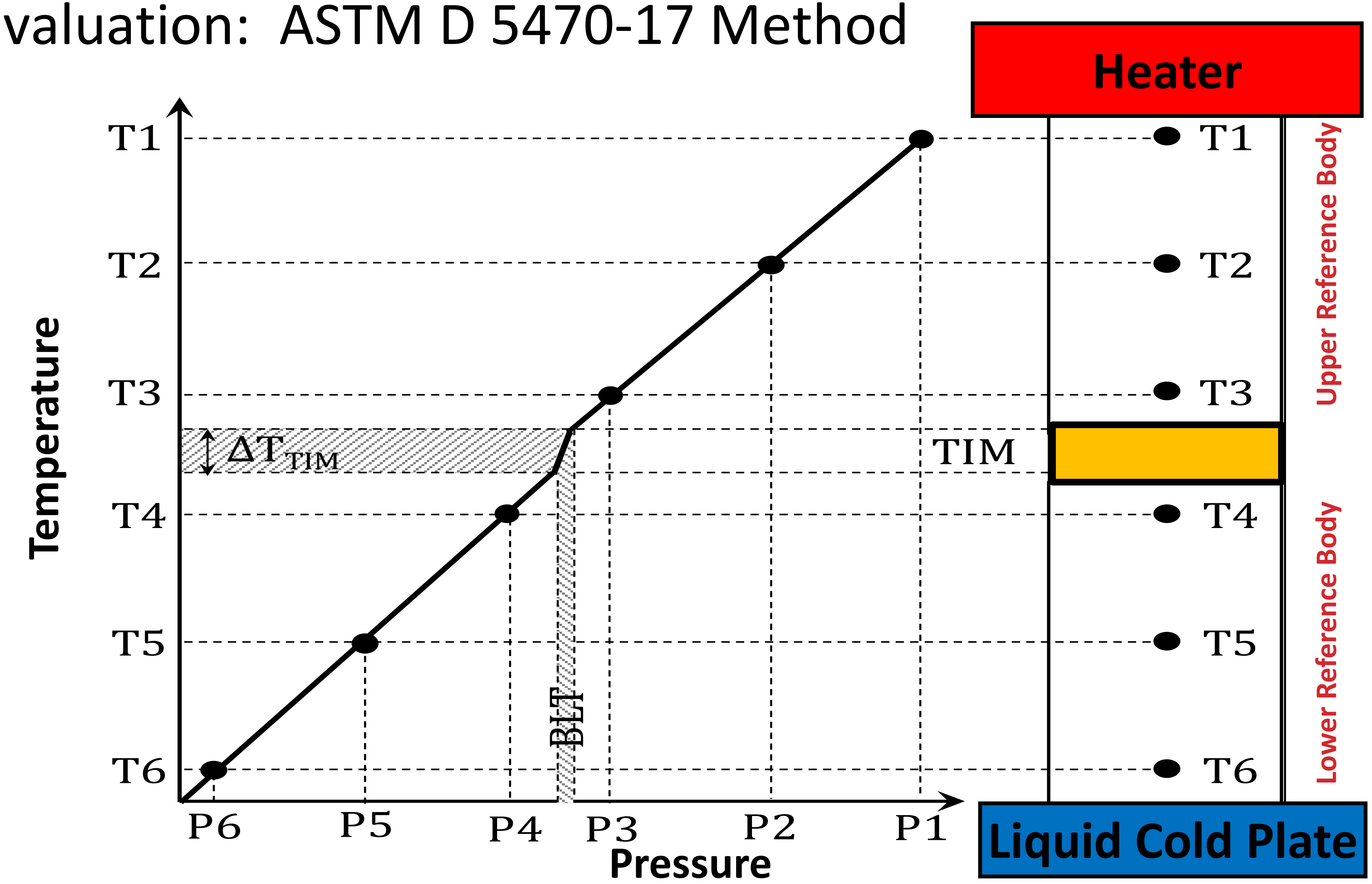
B. Test Methodologies and Test Systems for Thermal Materials

TIM Test Methodologies: Overview

Performance Property	Property Parameter	Method/Value
Thermal Resistance	Through-plane (primary) bulk + contact = total thermal resistance	ASTM D 5470-17 (Steady-state, unidirectional controlled heat flow) JEDEC JESD 51-14 (In-situ, Transient with structure function calculations from electrical resistances) Thermal Test Vehicle (TTV, in-situ)
Thermal Conductivity	Homogeneous, bulk (isotropic)	ASTM D5470-17 (Steady-state) JEDEC JESD 51-14 (Transient) Laser flash (Homogeneous materials) 3Ω Characterization
	Non-homogeneous, bulk (through-plane)	ASTM D5470-17 (Steady-state, unidirectional flow) JEDEC JESD 51-14 (Transient) 3Ω Characterization
	Non-homogeneous, bulk (in-plane)	Nanotest LaTIMA (Steady-state, in-plane flow) Scanning pulsed laser

Note: Not all test methods are suitable for testing certain categories of TIMs such as anisotropic and/or non-homogeneous structures (examples are compounds coated on a dielectric carrier or multilayer TIMs.)

TIM Evaluation: ASTM D 5470-17 Method



Note: ASTM D 5470-17 methodology may be purchased for a nominal fee from ASTM International (www.astm.org).

TIM Evaluation: ASTM D 5470-17 Method

Standard TIM test stand: Berliner Nanotest TIMA5:

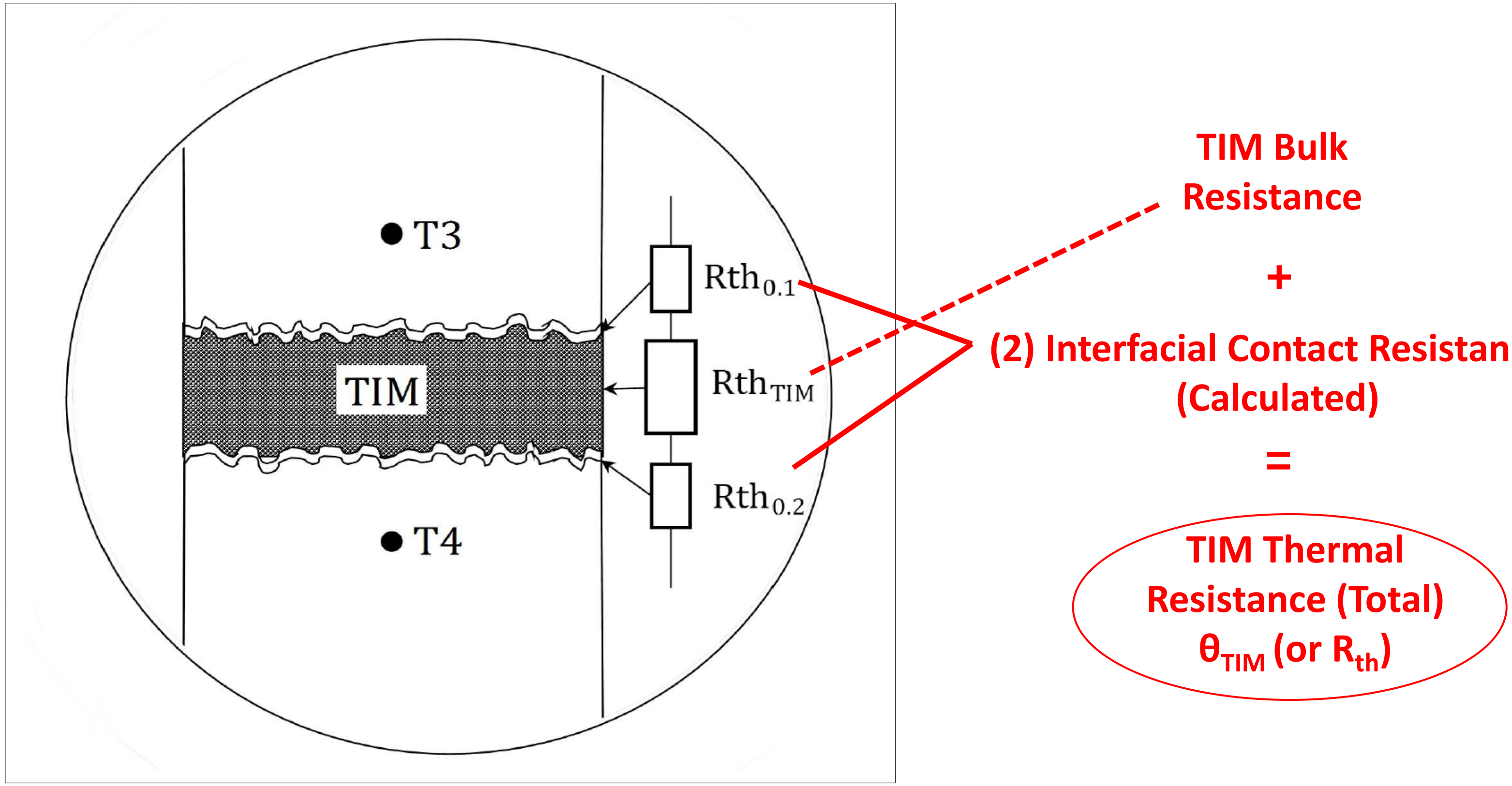
- Designed per ASTM D 5470-17, the industry-standard TIM test methodology for comparative material testing;
- System measures force applied, power (heat), thickness, thickness – with uniform heat flow.
- Measures thermal conductivity, calculated thermal resistance values w/error bars.
- Servo motor controls allow:
  - Extreme precision in measuring placement and thickness
  - Automation of functions (i.e., repeated contact/release cycle testing, to test for TIM durability for semi test.

Photograph: Berliner Nanotest und Design GmbH, Berlin, Germany. Nanotest TIMA5 ASTM D 5470-17 test stand.

TIM Evaluation: ASTM D 5470-17 Method

ASTM D 5470-17 describes a methodology to provide measurement of a thermal resistance value that is the sum of three constituent values:

- TIM thermal resistance *total* (θ_{TIM} or R_{th}) is the important value, in practice.



TIM Bulk Resistance

+

(2) Interfacial Contact Resistances (Calculated)

=

TIM Thermal Resistance (Total) θ_{TIM} (or R_{th})

TIM Test Methodologies: ASTM D 5470-17 and Transient

ASTM D 5470-17 and transient methods are the primary test methods for determining bulk thermal conductivity and thermal resistance values.

- *TIM vendor data sheet values should be developed utilizing ASTM D 5470-17* for comparative values generated under:
  - *Controlled surface conditions*
  - *Unidirectional heat flow conditions*
  - *Parallel contact surfaces*
  - *Precisely known clamping forces*

ASTM D 5470 Purpose: Develop comparative test data under identical conditions with all extraneous factors (such as die warpage or non-co-planar contacting surfaces) removed.

- Use of JESD 51-14 transient methodology *follows after* ASTM D 5470 testing. Goal is to develop *in-situ* performance test values with a specific package surface, clamping mechanism, other variables.
- Transient methods use electrical characteristics of a DUT, such as a power semiconductor, in-situ.
- These two methods are complementary: *One does not replace the other*.

TIM Test Methodologies: ASTM D 5470-17 and Transient

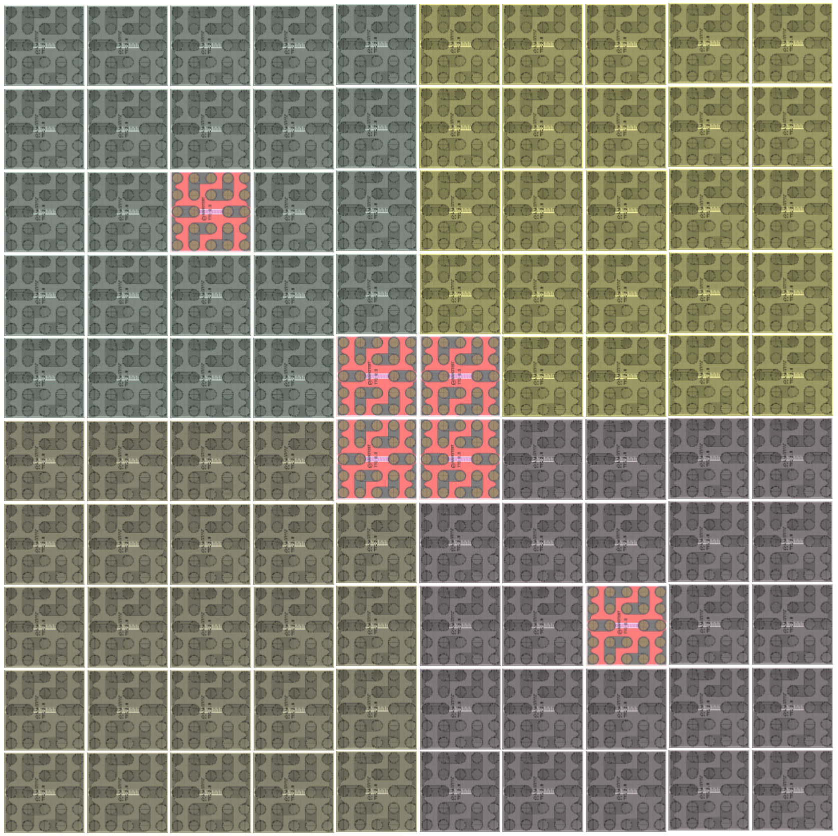
Method	Steady-State	Transient/Structure Function © 2022 - 2024 DS&A LLC
Designation	**ASTM D 5470-17 industry-wide standard**	**JEDEC JESD 51-14 semiconductor industry**
Purpose	Highly-defined test data generated under specific controlled, known unidirectional flow conditions • Primary industry standard for comparative performance	Data is generated under application-specific ("in-situ") conditions • Structure function knowledge required
Benefit	• Enables independent test lab data comparisons across multiple labs, test stands • Multiple types of tests, including mechanical reliability • All types of TIM and thermal materials, including adhesives • Multiple types of data generated: • Thermal conductivity • Thermal resistance vs. pressure range (0-140 PSI, typical)	• Requires specific package type and die, to generate useful in-situ test data • Characterizes internal package electrical and thermal performance • Results can be imported to CFD models for that one specific package and one set of conditions.
Data Output Types	• Known: Power, pressure, surfaces, heat flow • Application-dependent variables removed • Generate data sheet values under known conditions – *vital for TIM manufacturers and for material-to-material performance comparisons* • Data is used for *down selection* to determine materials to test and evaluate.	• Results will *not* necessarily correspond to ASTM D 5470-17 data – *by definition* • Results will be tightly aligned to a specific package type, only
Ideal User	• TIM and thermal material developers and manufacturers • Industry OEM mechanical engineers • Selecting TIMs for system design	Universities, semiconductor manufacturers

TIM Evaluation: Thermal Test Vehicle (TTV)

Construction of a TTV:

- Standard 200mm wafer design with 4000 cells
- Diced to custom requirement

- 2.4 × 2.4 mm² unit cells
- 8” wafer / > 4000 cells
- 500 µm undoped silicon
- Flip-chip assembly
- 50 × 50 mm² maximum die size
- Backside metallization:
  - Option 1: NiV 300 nm | Pt 100 nm | Au 200 nm
  - Option 2: Ti 100 nm | NiV 300 nm | Au 200 nm
  - Option 3: Pure silicon
- Power density: Up to 10 W/mm²
- RTD Sensitivity 10 Ω/K

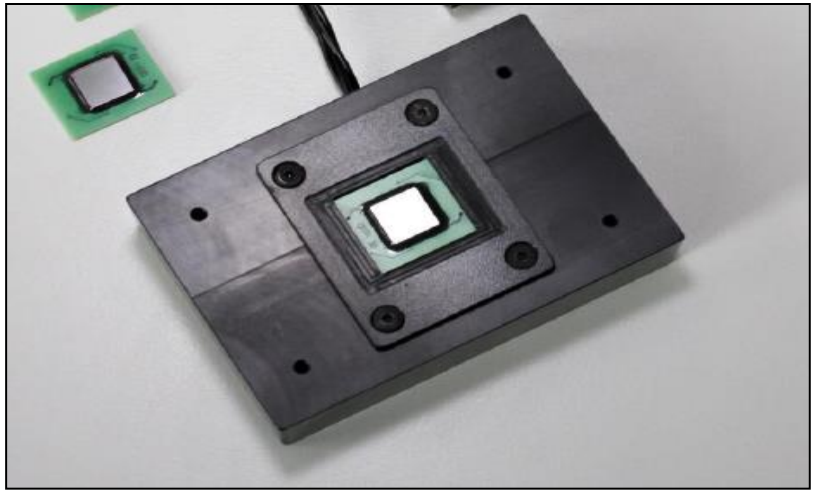


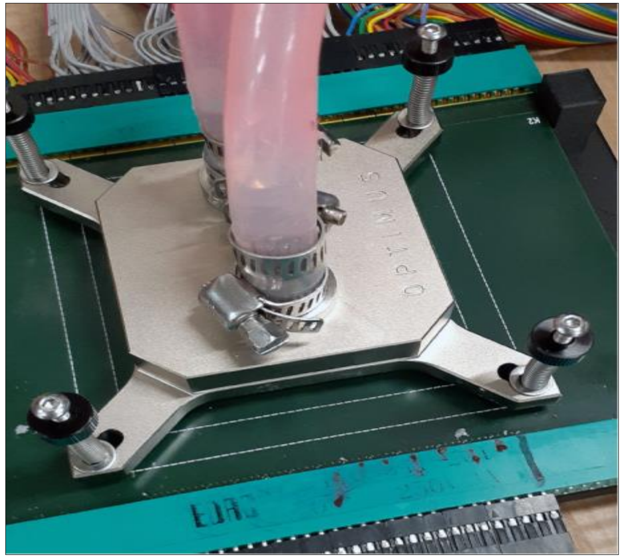
Illustrations: Abo Ras, M., Berliner Nanotest und Design GmbH, Berlin, Germany. March 26, 2024.

TIM Test Methods: Thermal Test Vehicles (TTV)

Thermal test vehicles are used for examining TIM performance in in-situ applications to measure:

- Performance of a TIM2 with a production semiconductor package;
- Performance of a TIM0 or TIM1 in contact with a die, to evaluate performance:
  - Given specific die warpage
  - With contact to lid (TIM1) or liquid cold plate/heat sink assembly (TIM0)
  - When well-designed, a tool that can provide very useful and detailed analytical capabilities for *in-situ* measurement for applications with a specified package type.





Sources: (Left) Berliner Nanotest und Design GmbH; (Right) Indium Corporation (with liquid cold plate applied to bare die TTV on engineering test board (ETB). (Photograph, DS&A LLC, January 16, 2024.)

Comparative Thermal Resistances: Reliability Testing

Impact of power cycling and bake testing on TIM types (following slides):

- *Demonstrating the importance of comparative thermal resistance testing beyond time zero*, for material evaluation

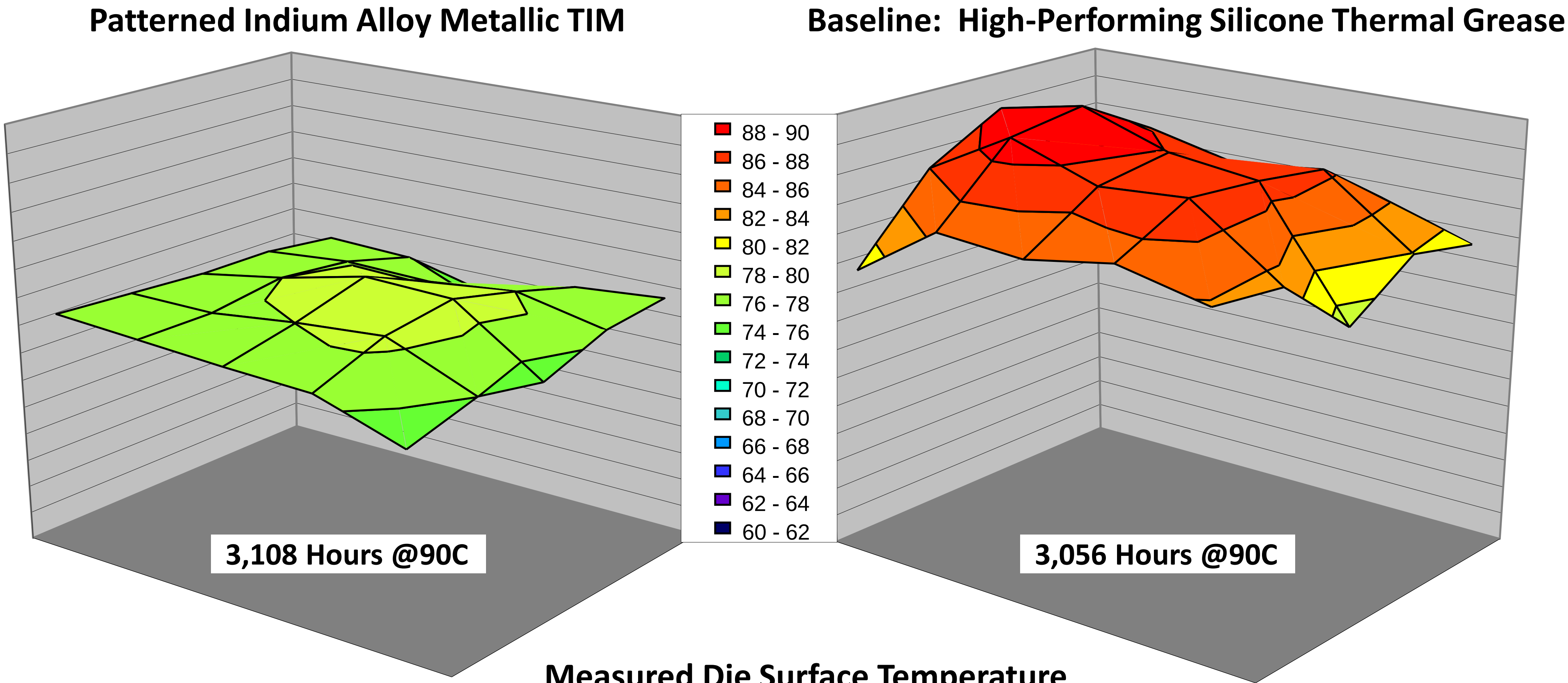
✓ Power cycling

  - Increasing thermal resistance values indicates decay in performance over time.
  - Declining thermal resistance values indicate TIM performance is *improving* over time.

✓ Bake cycling (90C)

  - Declining thermal resistance indicates bake-out of silicone oil carrier from thermal grease.

Patterned Metallic TIMs: Reliability



Measured Die Surface Temperature

Thermal Test Vehicle Comparative 3,000-Hour Reliability Bake Test

Note: Measured die surface temperature at time zero was shown to be approximately equivalent. Above test data taken after 3,000-hour bake test. Increased die surface temperature for Figure 4B reflects increased thermal resistance due to dry-out of silicone oil in the tested premium silicone-based thermal grease.

Data source: R. Jarrett, Indium Corporation, Utica NY USA.   Die thermal test vehicles: Provided by Intel Corporation.

TIM Testing – Process Steps for Test Performance Methods

Example, aerospace systems manufacturer procedural sequence for TIM test methods:

Phase 1	Phase 2	Phase 3
1 **ASTM D-5470-17 Conduction Test Stand** • Baseline commercially available materials • Preliminary characterization of Phase 1 “new technology investigation” (NTI) material	• Characterize Phase 2 NTI material	• Characterize Phase 3 NTI material
2 **Thermal Test Vehicle** • Design and validation of modular flip-chip TTV • Preliminary testing with baseline (currently qualified for production) materials	• Preliminary reliability testing (cycling, rework) w/baseline materials • Preliminary comparative NTI material testing vs. baseline materials	• Extended reliability testing (cycling, rework) w/ NTI materials
3 **In-Situ Testing** • Requirements definition for in-situ test device • Preliminary device identification, calibration	• Design and validation of in-situ test device (RF PA) • Preliminary testing with baseline materials	• Design and validation of in-situ test device (digital component) • Testing of NTI materials

Summary

TIMs are critical to efficient heat transfer from a semiconductor source.

- Understanding TIM types, testing methods is critical to proper evaluation.
- Specialized TIM types with very different requirements are required for challenging, highly-specialized applications in very different markets.
- Selection of a TIM is not based *only* on maximum bulk thermal conductivity.
- New development materials such as graphene-enhanced graphite, carbon fiber preforms, and certain new forms of liquid and hybrid liquid metallic TIMs are in development.
- Significantly higher temperatures, higher heat fluxes, greater die warpage, and cryogenic temperatures are challenging new requirements for TIMs.

Addendum

Additional information on related topics:

Comparative thermal resistance – Dry junction

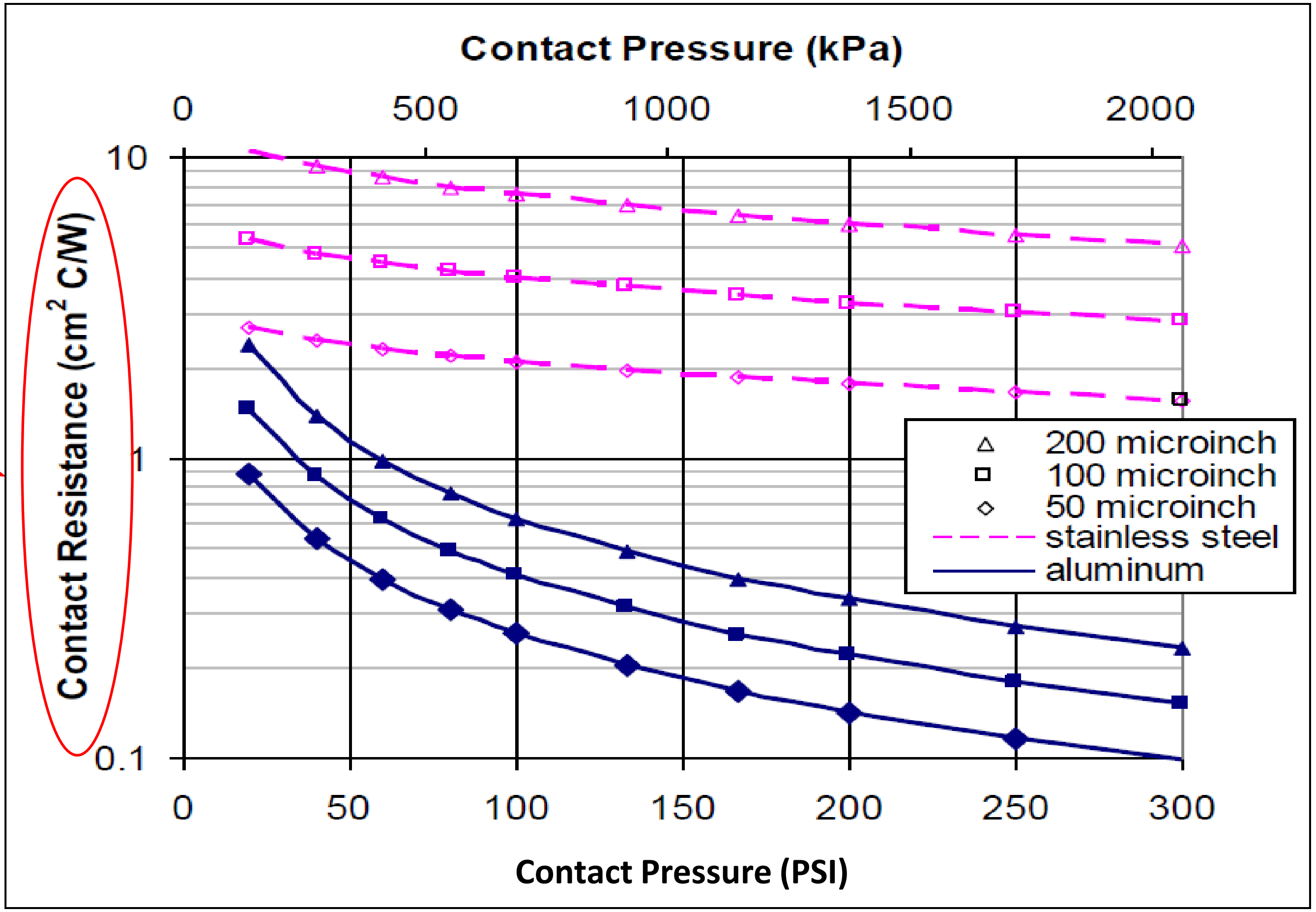
TIM Vendor Development Requirements
TIM Development specifications for semiconductor test
Phase Change-Coated Graphite Films and Reliability Testing
Liquid immersion systems – Contamination sources
Liquid immersion systems – Contamination and mitigation
Liquid immersion systems – Challenges (Single- and two-phase)
Quantum computing systems – TIMs suitable for cryogenic temperatures
Cabot aerogel particles – Energy storage material developments

References

Comparative Thermal Resistances

Metal-to-metal surface contact resistance – comparison of two metals, three surface finishes (roughness), under load:

The primary function of a TIM is to minimize thermal contact resistance between two mating surfaces

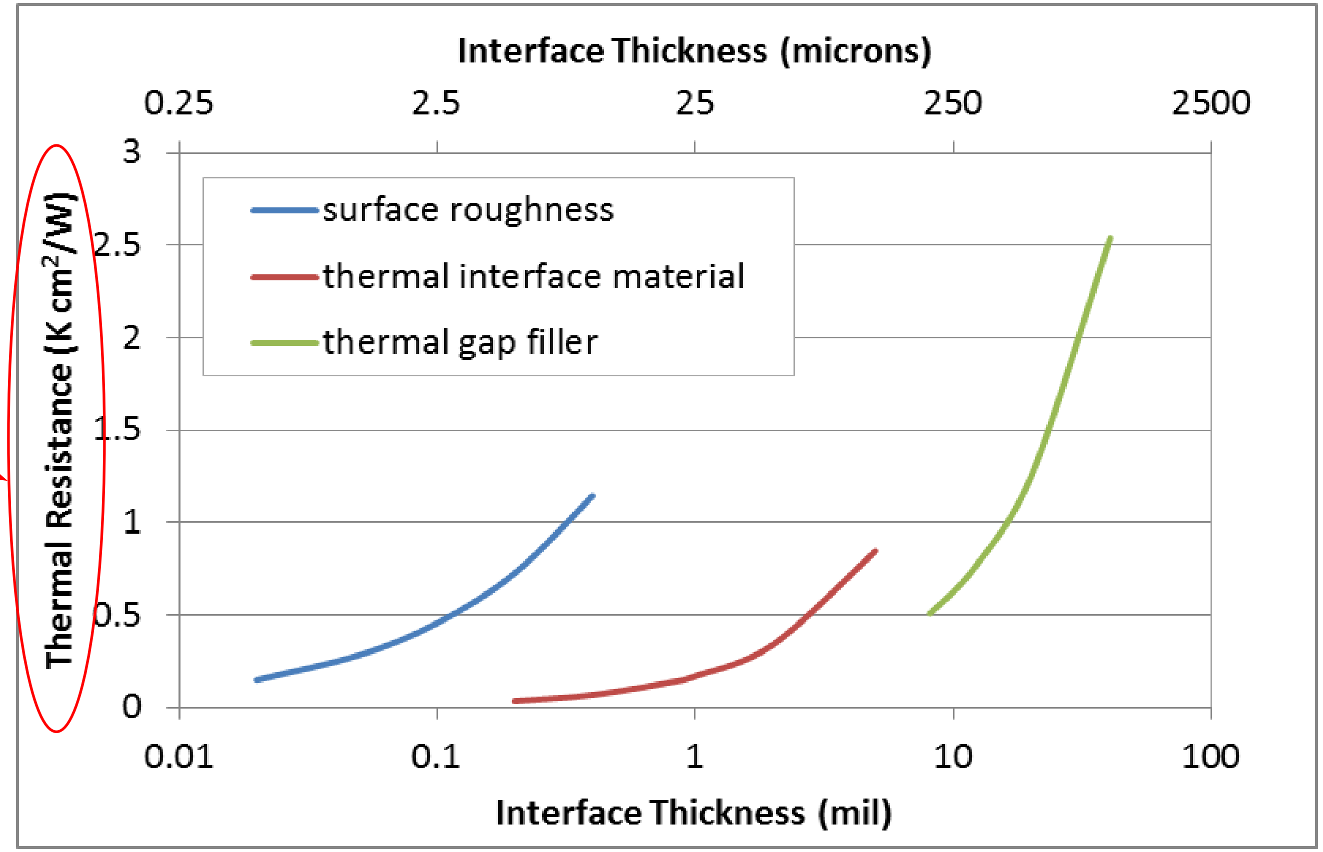


Source: M. Yovanovich, et al., "Calculating Interface Resistance", Electronics Cooling Magazine*, May 1997, Vol. 3, No. 2. Note: Values are RMS values. 100 microinch = 2.54 microns.*

Comparative Thermal Resistances

Comparison of surface roughness and relative interface thicknesses, to define a *general* application range for TIMs versus gap-fillers, under load:

The primary function of a TIM is to minimize the thermal contact resistance between two mating surfaces.



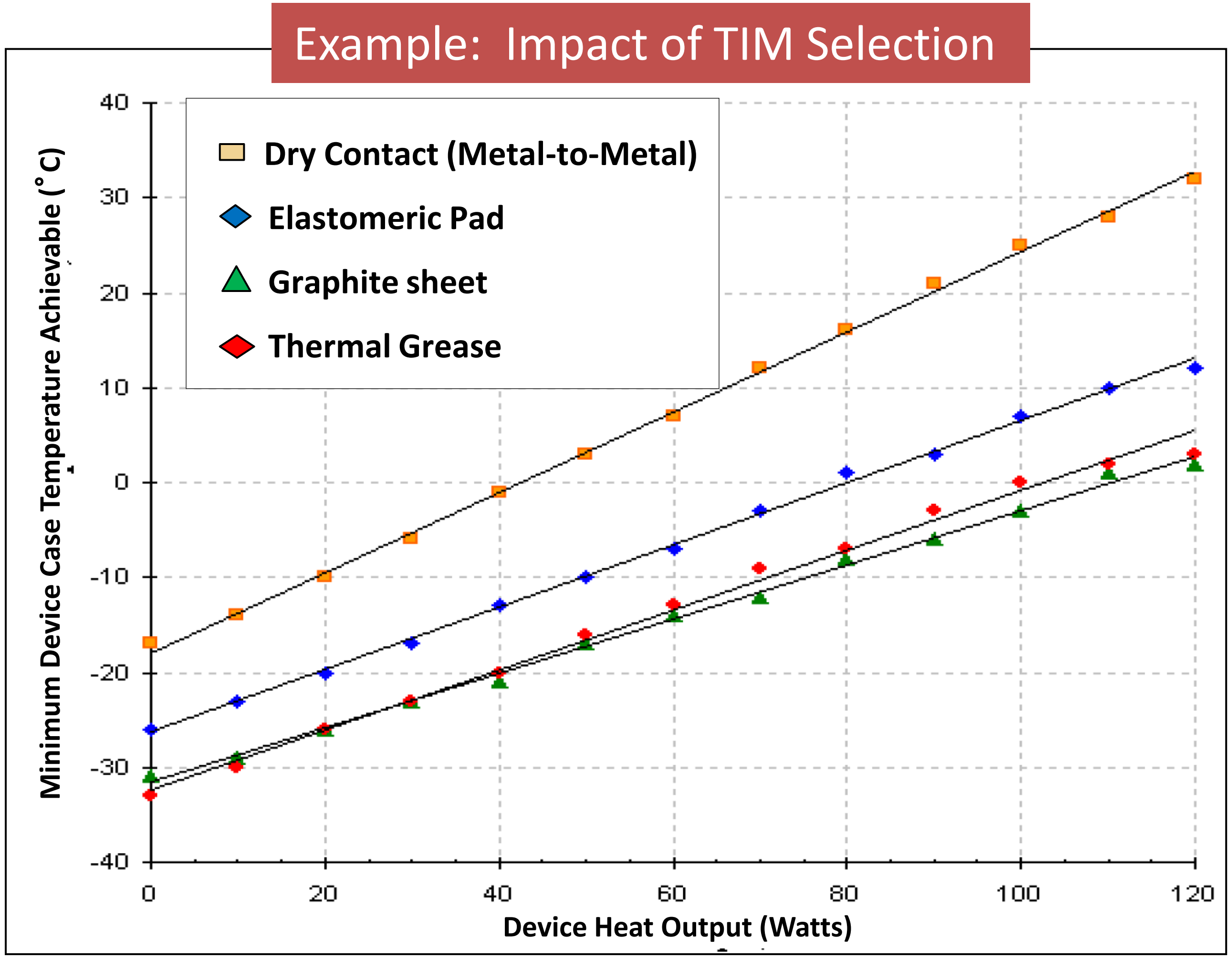
These are rough estimates and values will vary with material properties and contact pressure (clamping force) applied.

Source: Ross Wilcoxon PhD, Senior Engineering Fellow, Advanced Technology Center, Collins Aerospace Inc., Cedar Rapids IA USA. Unpublished; Used with permission. Note: 100 microinches = 2.54 microns. Values are RMS values.

Comparative Thermal Resistances

Impact of a TIM versus dry contact on device operating temperature:

- Three different TIM types tested versus dry contact

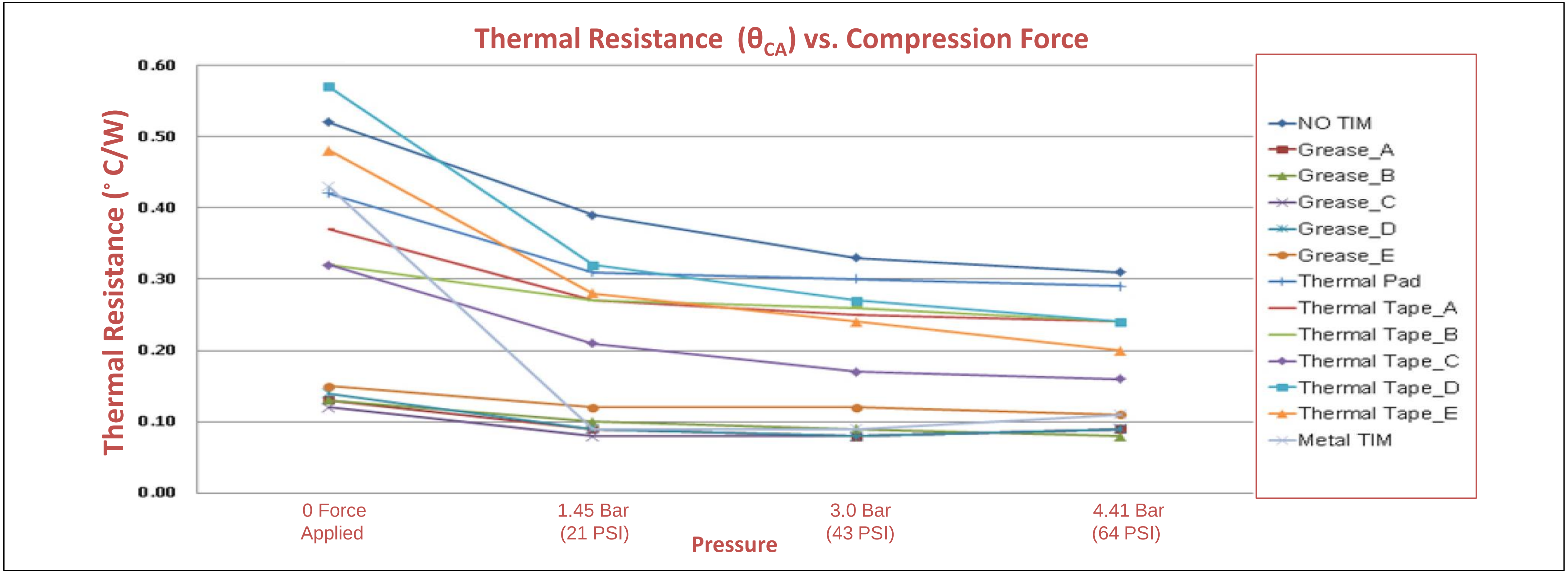

Example: Impact of TIM Selection

Source: K. Moody, R&D Manager, Kulicke & Soffa (USA). Note: All other conditions held equal.

TIM Test Methods: ASTM D 5470-17

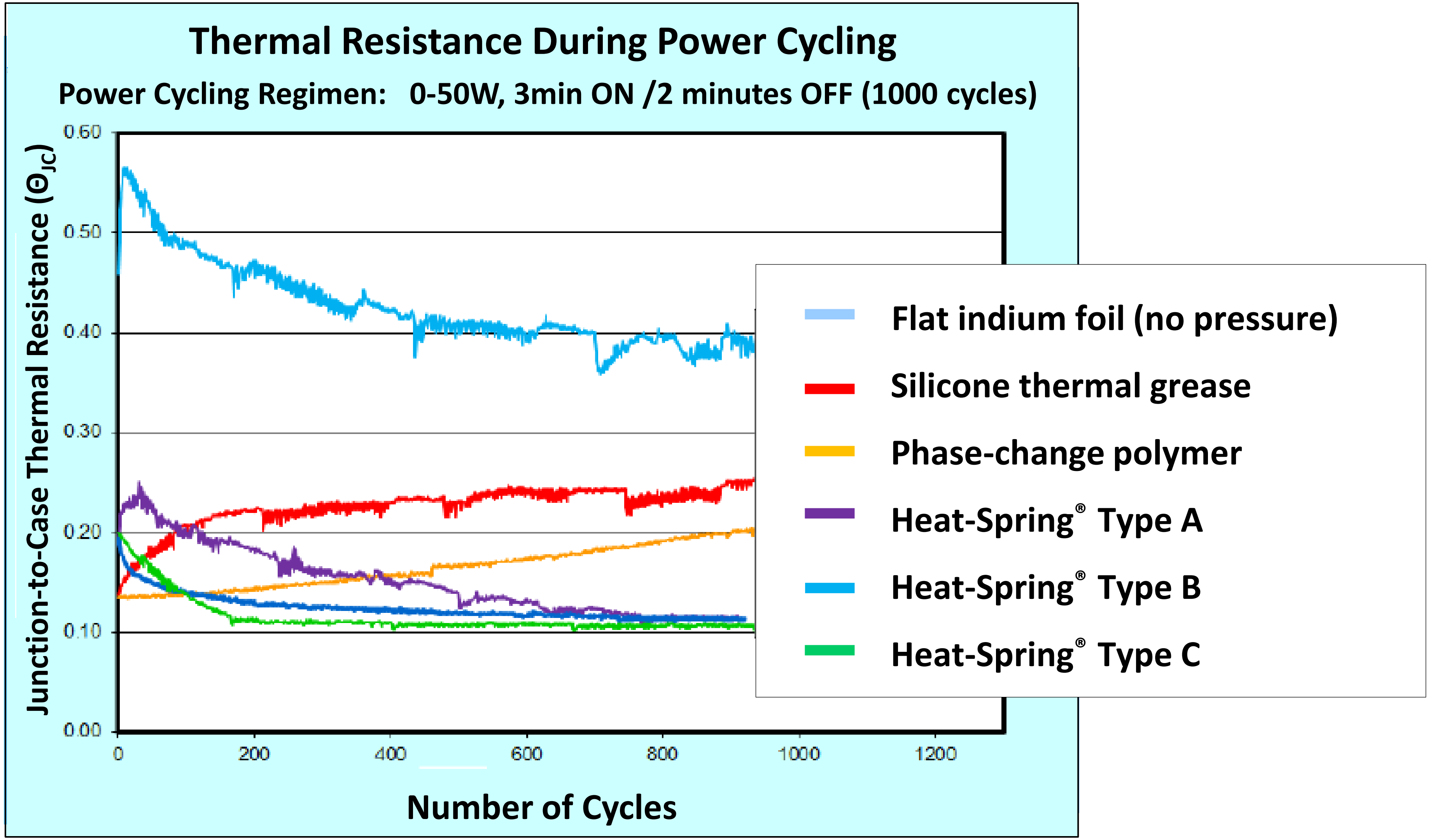
ASTM D 5470-17 test methodology -- Example of comparative test data generated:

- Application of specified pressures significantly improves thermal resistance of many TIM types;
- Properly-designed test stand provides apples-to-apples comparative data, all factors equal.



Note: Specific TIM materials are not identified by vendor and vendor product identification. "Metal TIM" is indium metal flat foil.
Source: Ng Hooi Hooi, Thermal Test Solutions, Inc.; "Introduction to Thermal Interface Materials," BiTS Test Workshop, Mesa AZ USA, March 5-8. 2017.

Comparative Thermal Resistances: Reliability Testing



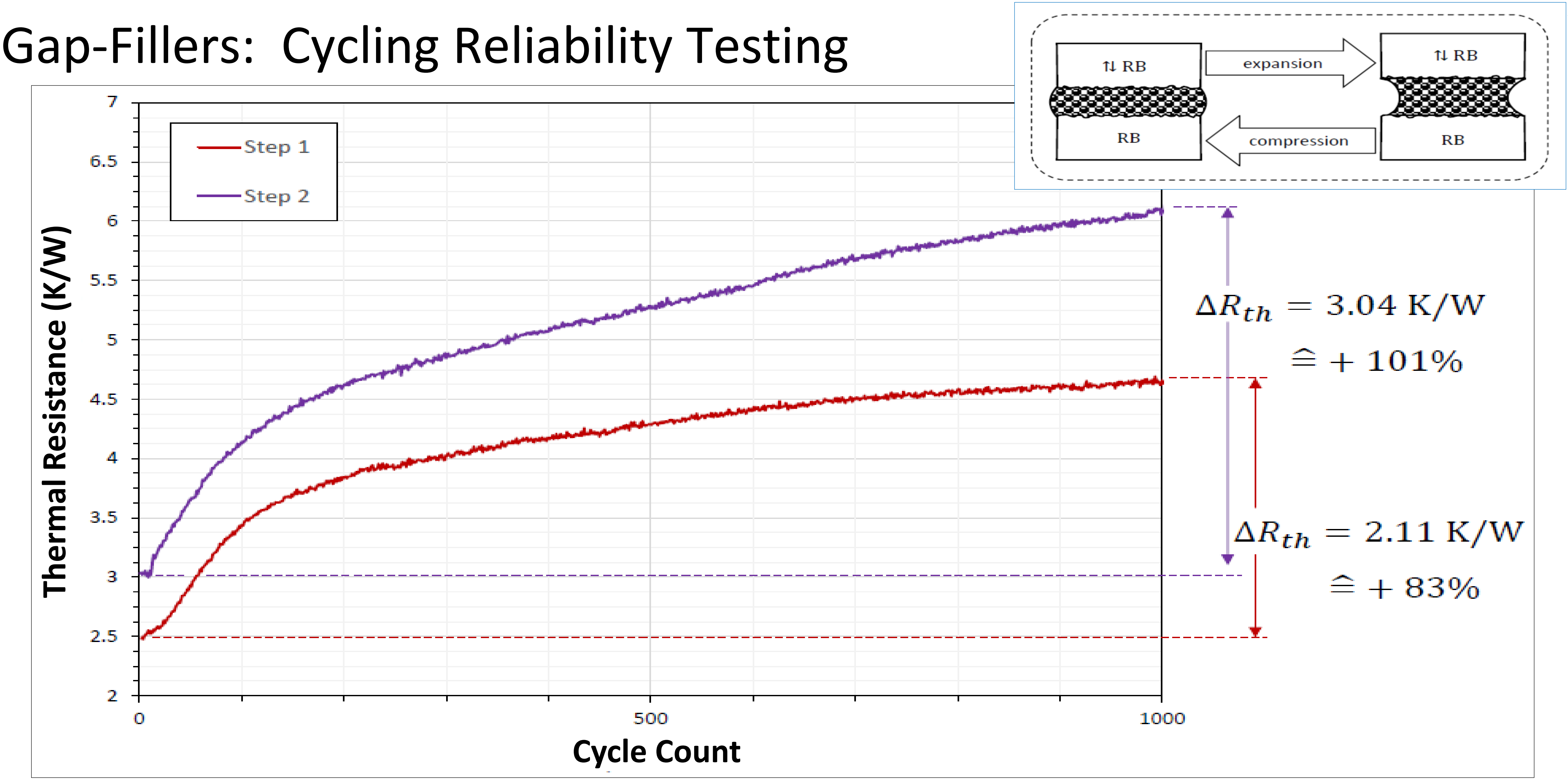
Source: Indium Corporation

Gap-Fillers: Cycling Reliability Testing

Mechanical cycling and other types of reliability testing over time can demonstrate useful results for evaluating TIMs.

- Previous reliability testing has been undertaken by Berliner Nanotest of “gap-filler” TIMs, examining cyclic compression and relaxation
- Gap-fillers may also be useful for test/burn-in – with different reliability testing requirements.
  - An example is the use of so-called “gap-filler” TIMs for testing with PCBs and other substrates.
  - The same TIM test equipment described has also been used for reliability testing of metallic TIMs with contact/dwell/release cycling, to mimic semiconductor test requirements.

Gap-Fillers: Cycling Reliability Testing



Source: Berliner Nanotest und Design GmbH. February 16, 2018.

TIM Testing/Evaluation Methodologies

TIM Testing – Environmental Conditions		
General Property Type	**Failure Mode or Property**	**Methodology**
Environmental	Outgassing, Weight Loss	Thermogravimetric analysis (TGA); ASTM E595
	Humidity, Moisture	HAST 85/85
Electrical	Voltage Breakdown	ASTM D149
	Flame Rating	UL-94
Thermal	Thermal Resistance	ASTM D 5470-17
	Thermal Conductivity	ASTM D 5470-17
	Thermal Resistance (Alternative)	Transient Testing
	Thermal Resistance (Alternative)	Thermal Test Vehicle or TTC

TIM Development – Test Equipment Manufacturers

Selected TIM Test Equipment Manufacturers		
Company	**Test Stand General Type**	**Status**
Berliner Nanotest und Design GmbH **Berlin, Germany**	TIMA® ASTM D 5470-17 (Modified)	Production
	LaTIMA® In-Plane Bulk Thermal (X-Y) Conductivity Test Stand	Production
	Thermal Test Die, Thermal Test Wafers, Thermal Test Vehicles (TTVs)	Production
	Three-Omega Method Liquid/Gel Thermal Conductivity Test Stand	Production
Siemens (Mentor Graphic Mechanical Analysis Division)	"T3Ster" Structure Function Transient Test Stand; DynTIM™ Test Head	Custom
Zentrum für Wärmemanagement **Stuttgart, Germany**	Multiparameter Long-term Cyclic Load Test Stand Tensile and Compressive Load Test Stand	Custom

Source: DS&A LLC . Selected vendors shown.

TIM Vendor Development Requirements

TIM Vendor Typical Development Requirements	
Thermal Impedance	Dielectric Strength**
Bond Line Thickness Post-Assembly	Cut-Through Resistance**
Thermal Conductivity	Thermal Cycling
Clamping Force Applied	Power Cycling
Wettability	Humidity and Bake
Thixotropicity	HAST
Dispensing/Placement Process Automation	Shock and Vibration
Cure Schedule*	Flammability
Adhesion, Peel Test*	Working Life
Contaminants	Storage/Transit Temperature Range (As Supplied)
Modulus of Elasticity	Shipment/Storage Temp Range (Complete Assembly)
Material Stability	Removability and Rework Process
Silicone Stability	Environmental and Recycling Process
Flammability	Cost

*Notes: *Applies only to adhesive TIMs. **Applies only to dielectric TIMs.*

TIM Development Specifications for Semiconductor Test

High Performance TIM Material Target Specifications *for Test*	
Material Attribute	**Goal[1]**
Material Stability	No constituent run-out, no mechanical pump-out. Dimensionally stable; no moisture sensitivity during processing or normal operation in specified ambient environmental conditions. No fretting.
Silicone Stability	No silicone content; no dry-out, no silicone oil separation; zero measurable separation by weight (TGA).
Surface Wetting	TIM provides sufficient surface contact to approach 100% surface wetting in clamped condition, including expected warpage and specified surface conditions.

Notes: 1. Generalized statements, applicable to all levels of TIM (TIM0, TIM1, TIM2).

TIM Development Specifications for Semiconductor Test

High Performance TIM Material Target Specifications for Test	
Material Attribute	**Goal[1]**
Thermal Performance	Target and stretch goals for thermal resistance to meet system maximum heat load and heat flux.
Outgassing	No permissible outgassing per NASA, aerospace applications requirements; no outgassing for medical, optical, optoelectronic applications and systems
Environment	Suitable for shipment, storage, processing, operational temperatures (ambient, junction/module)
Cost	Budget goals met with volume manufactured TIM.

Notes: 1. Generalized statements, applicable to all levels of TIM (TIM0, TIM1, TIM2).

TIM Development Specifications for Semiconductor Test

High Performance TIM Material Target Specifications for Test	
Material Attribute	**Goal[1]**
Conformability	Same TIM conforms to different die sizes, lid sizes without damage or change in performance.[2] TIM conforms to 90 bending and wrapping around test head/socket lid configuration. [2]
Particulates	No permissible loss of particulates, fibers.[2] No residue visible, remaining on DUT after contact; no detritus.[2]
Durability	Tested cycling survival through X number of repeated contact-and-release cycles. [2]

Notes: 1. Previous statements are applicable to all levels of TIM (TIM0, TIM1, TIM2).
2. Statements specific to semiconductor test and liquid immersion systems.

TIM Development Specifications for Semiconductor Test

High Performance TIM Material Target Specification for Test	
Product Attribute	**Goal***
Thermal Resistance	Target: < 0.35°C-cm^2/W @ Minimum clamping force applied Stretch: < 0.15°C-cm^2/W @ 60PSI clamping force applied
Contact, Non-Coplanar Surfaces	Target: 1,000 – 5,000 Cycles Stretch: 5,000 – 15,000
Thermal Conductivity	30W/m-K ■ (Minimum) >100W/m-K ▲ (Ideal)
Operating Temperature	-15°C to 120°C ■ (Minimum) -40°C to 200°C ▲ (Ideal)

*Key to symbols: ▲ Market leading product. ■ Market improvement w/equivalent or better pricing. * Generalized statements. Source: DS&A LLC.*

TIM Development Specifications for Semiconductor Test

Examples of TIM2 Developments					
Thermal Material General Type	**Thermal Resistance**	**Temperature Range Capability**	**Suppliers**	**Cost**	**Development Status**
VA-CNT*	Very Low	Wide	Limited	Very High	Development, Early Prototyping
VA-CNF**	Very Low	Wide	Limited	Moderate	Development, Early Prototyping
Graphite Heat Spreaders	High	Very Wide+	Many	Moderate	Production
Al Foils+Compound (Non-Silicone)	Low	Wide	Limited	Low	Production
Patterned Metallic Foils	Very Low	Wide	Limited	Moderate	Production

Notes: VA-CNT: Vertically-aligned carbon nanotube array in carrier. VA-CNF: Vertically-aligned carbon fiber or graphite particulates in carrier.
+ Graphite heat spreaders are highly anisotropic and are not TIMs; temperature tolerance to 400+ ℃.
Source: DS&A LLC .

Phase Change-Coated Graphite Films and Reliability Testing

TIM commercial products developed for semiconductor test requirements, included in Phase I test program:

Thermal Interface Materials Tested	
Graph Key	**Description**
CLAD	Indium (99.99%) flat foil, one side only 5µ aluminum cladding
CLAD HSK	Indium (99.99% foil, one side only 5µ aluminum cladding, HSK pattern applied*
Al Foil, One-side coated	Aluminum foil 50µ thickness, coated one side with dry thermal compound**

*Note: * Indium Corporation Heat-Spring® HSK. ** Development material only.*

Phase Change-Coated Graphite Films and Reliability Testing

Current development of phase-change coated graphite film carriers by Streuter Technologies:

- Data sheet test values per ASTM D 5470.
- Decades of proven compound coating experience processes for TIM materials, to date;

Streuter Technologies is now offering different versions of this product type:

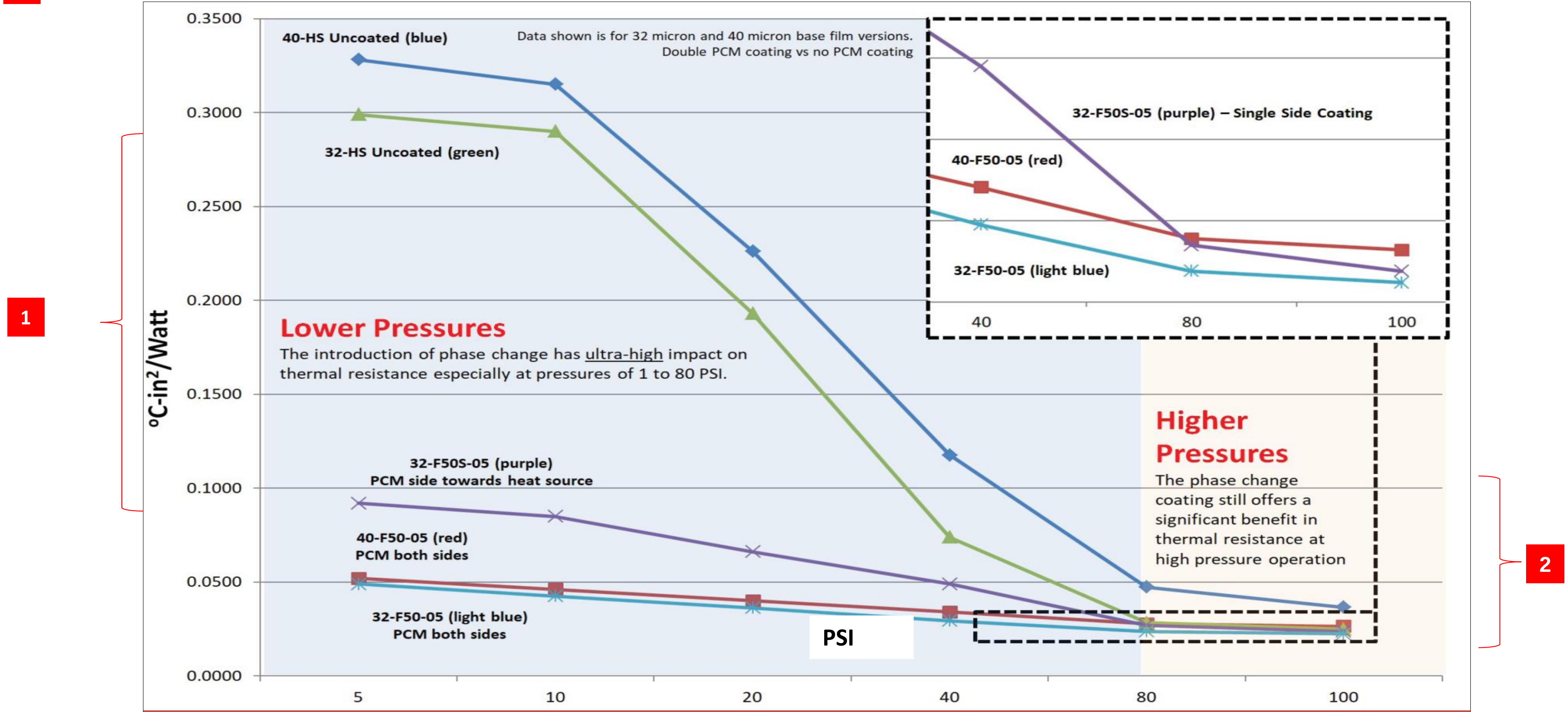
- Single-side phase-change coating on film graphite film for semiconductor test applications;
- Single- and double-sided coatings in different thicknesses, as required;
- Multiple combinations of options in performance and durability testing:
    - Carrier (20-, 32-, 40-, 70-micron thicknesses)
    - Coating thickness and custom footprint/offset compound coatings
    - Phase-change temperature (52°C, 60°C)
- High degree of surface wetting achieved – addressing surface warpage and roughness.
- Certain graphite films are highly durable and have passed significant 90-degree bend testing over tens of thousands of bend cycles – appropriate for semiconductor test.

Source: Streuter Technologies, Inc. (www.stretech.com)

Phase Change-Coated Graphite Films and Reliability Testing

1 32micron graphite film: 71% Improvement at low pressure (5 PSI), single-side coating vs. uncoated

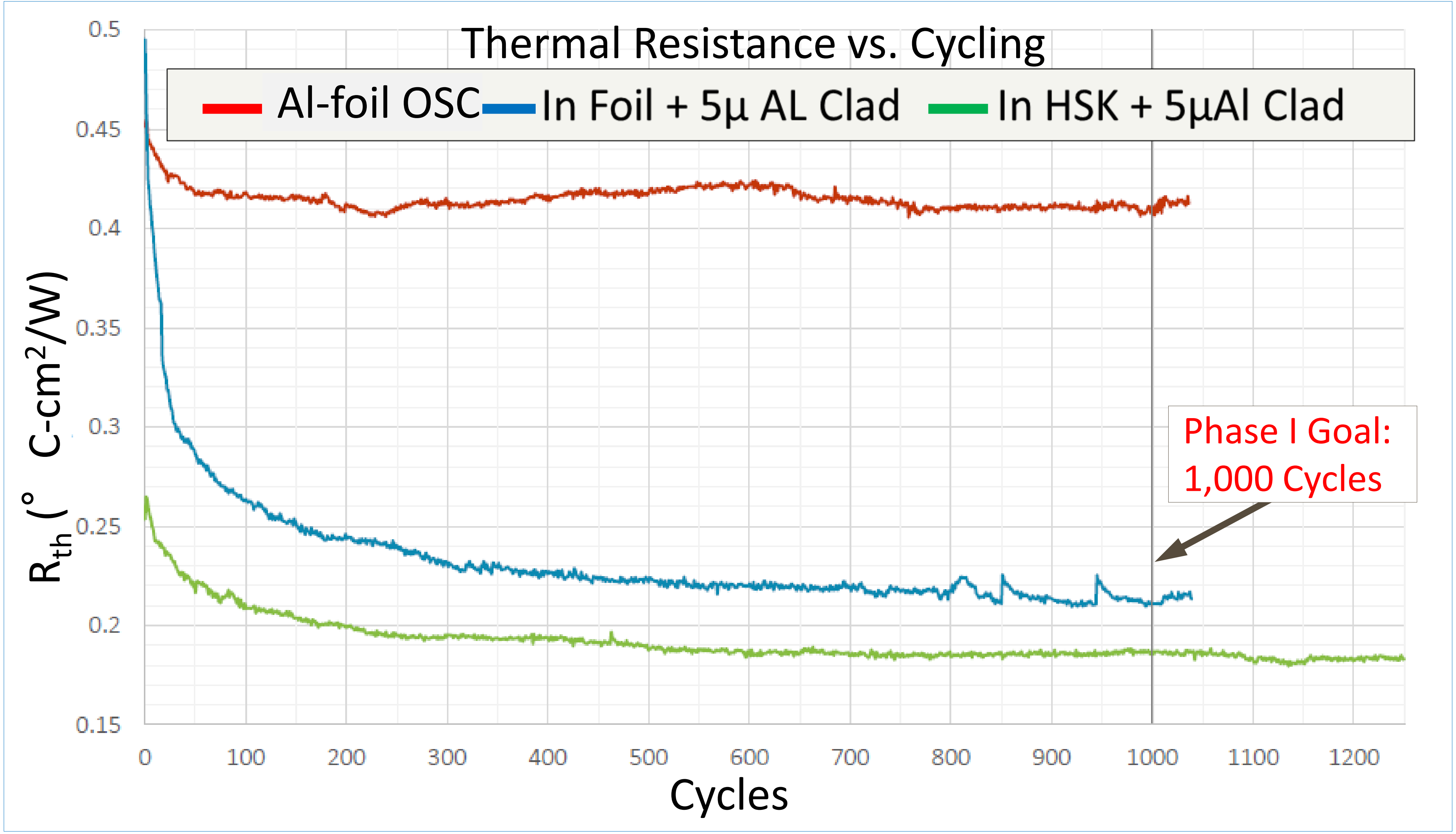
2 40micron graphite film: 70% Improvement at medium (40 PSI) pressure, double-side coating vs. uncoated



Thermal Resistance, PCM-Coated Graphite Films vs. Uncoated Graphite Films

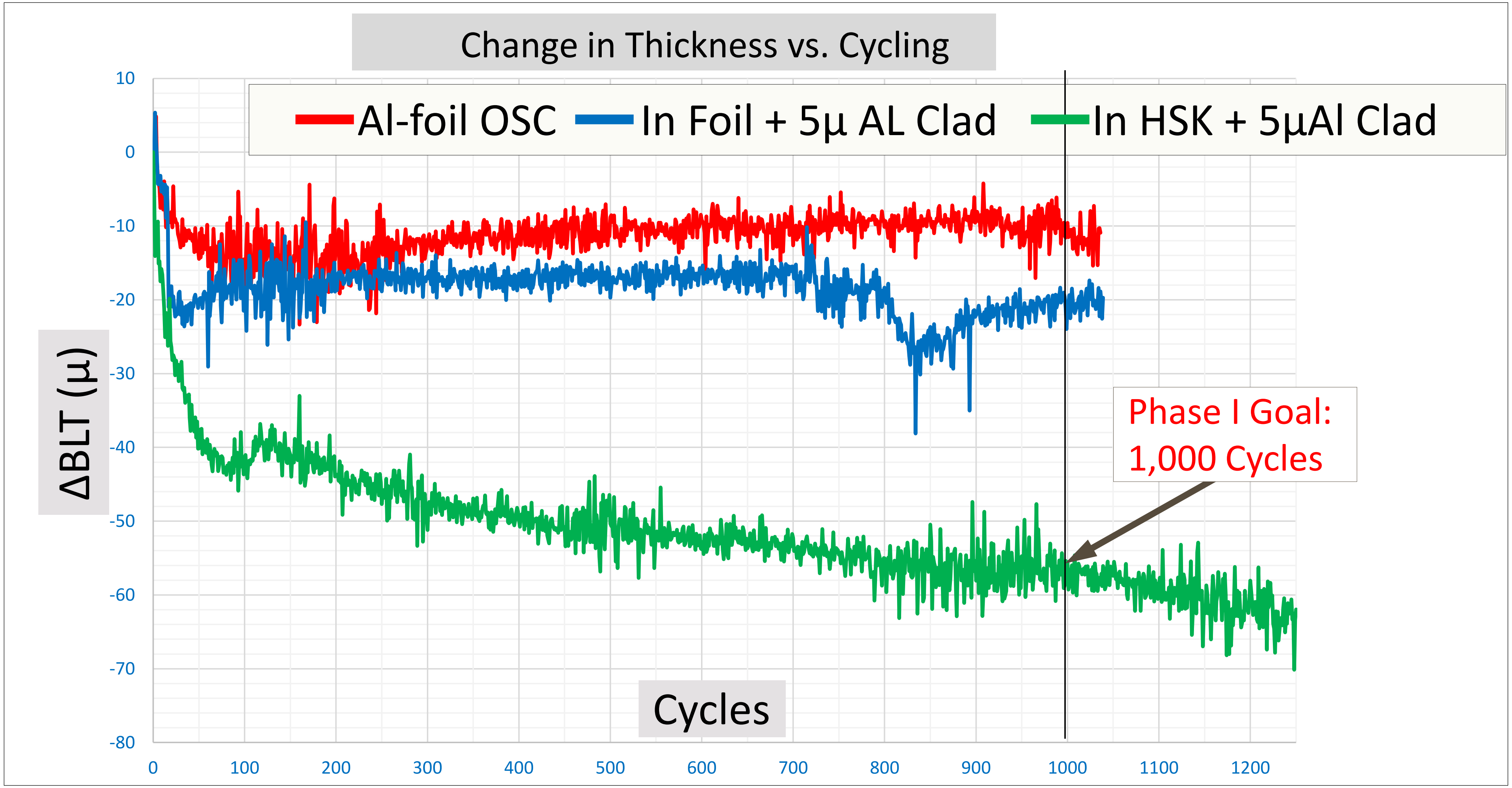
Source: Streuter Technologies, Inc. (www.stretech.com)

Phase Change-Coated Graphite Films and Reliability Testing



Source: Berliner Nanotest und Design GmbH.

Phase Change-Coated Graphite Films and Reliability Testing



Source: Berliner Nanotest und Design GmbH. Observed variation (>800 cycles) for indium flat foil with al-cladding caused by an error with cold plate water supply. Testing concluded at 1,000 cycles (with exception of patterned In HSK +5µ Al cladding).

Challenges for Immersion Liquid Cooling Systems

Liquid Immersion Systems: Contamination Sources

Immersion Liquid Cooling Systems – Potential Contamination Sources						
Material Source of Contaminant	**Rectifier**	**Server**	**Cabling**	**Tank**	**Example of Component Containing Contaminant**	**Mitigation/Comment**
PVC insulation	•	•	•		Wiring: Communication and power cables, etc.	Use cleaner alternative
Silicone RTV	•				Transistor potting, vibration dampening capacitors	
Elastomers				•	Tank o-rings, seals, etc.	Minimize; use cleanest option
Hot-melt adhesives	•				Wire retention	
Electrical Isolation Pads	•				Power supply transistors and diodes	*
Heat-Shrink Tubing	•				Wiring, cable assemblies	Minimize use
TIM - Thermal grease	•	•			Transistor and diode heat sinks	*
TIM - Thermal grease		•			Heat sink attach to processors	OK to use but alternatives exist
TIM – Gap-fillers	•	•			Attach heat sinks for voltage regulators, diodes, etc.	*

*Note: * Eliminated in two-phase operation.   Source: Panel Discussion, "One Year of Two Phase (2P) Immersion Cooling in the Cloud: Lessons Learned", Raniwala, A. (moderator); Alissa, H., Manousakis, I., Shaw, M., Microsoft Corporation; Tuma, P., 3M Company; Chen, S., Wiwynn. OCP Global Summit, November 9-10, 2021.*

Liquid Immersion Systems: Contamination Sources

Immersion Liquid Cooling Systems – Potential Contamination Sources

IC or Power Component Type	Processor	AI/GPU	ASIC	RDIMM	VRM	Power Device	Material	Challenge
TIM1	•	•	•				Polymeric TIM, gel, thermal grease	Potential for cavitation, extraction, or solvating of hydrocarbon-based substances (such as silicone oil carrier)
TIM1	•	•	•				Hybrid/liquid metallic TIM	
TIM0	•	•	•	•			Polymeric TIM, thermal grease	
TIM2	•	•	•	•	•		Polymeric TIM, gel, thermal grease	
Gap-fillers				•	•	•	Silicone/siloxanes and polymeric	
Lid seal adhesives	•	•	•				Epoxy, one-part silicones, other	

Liquid immersion (1P/2P) requires evaluation of many materials within a server:

- Potential for extraction of constituents from each material type by the fluid;
- *Potential for fouling of the immersion fluid* by extracted hydrocarbons, constituents, other particulates;
- *Potential for fouling of filters* and other system components with extracted and redeposited constituents.

*Note: * Eliminated in two-phase operation.   Source: Panel Discussion, "One Year of Two Phase (2P) Immersion Cooling in the Cloud: Lessons Learned", Raniwala, A. (moderator); Alissa, H., Manousakis, I., Shaw, M., Microsoft Corporation; Tuma, P., 3M Company; Chen, S., Wiwynn. OCP Global Summit, November 9-10, 2021.*

Liquid Immersion Systems: Contamination Sources

Example -- Simple immersion fluid contamination test with four different types of TIM2:

Fluid: PAO6 with 30-second agitation after insertion of TIM:

- Polymeric phase-change compound
- Silicone oil carrier thermal grease
- Indium metal foil

Source: Test and photograph - A. Mackie, Indium Corporation, February 2023. Used with permission. Similar testing undertaken at 3M Company, Specialty Liquids.

Liquid Immersion Systems: Contamination, Mitigation

TIMs for Liquid Immersion		
TIM Type	**Potential for Contamination**	**Mitigation/Comment**
Silicone Thermal Grease (<4% silicone)	Minimal	Currently in use
Silicone Thermal Grease (4-16% silicone)	Significant	Select better formulation w/reduced silicone oil content
Gap-fillers	Significant	Concern for silicone content. Eliminated in 2/P operation.
Graphite film/sheet	None	Concern for potential fretting, electrically conductive (not yet tested) Evaluation needed: Potential for air entrapment in interface[2] (Heat spreaders w/poor through-plane effective conductivity)
Indium foil **Patterned indium foils[2]**	None	Currently in use
Hybrid/liquid metals; solid/liquid hybrids	Unknown	Evaluation needed
Soldered joint	None	Residual flux will redeposit on contacts for power components, causing shorting (Two-phase systems)
Sintered foil/sintered joint	None	No compatibility testing identified to date*

▨ *Joining materials, not TIMs. Included for completeness.*

Notes: 1. Indium Corporation Heat-Spring® and similar.

2. See investigation: Coles, H., Herrlin, M., "Immersion Cooling of Electronics in DoD Installations," DOD ESTCP Project EW-201347, Lawrence Berkeley National Laboratory Report (May 2016).

** Suggested for academic investigation.*

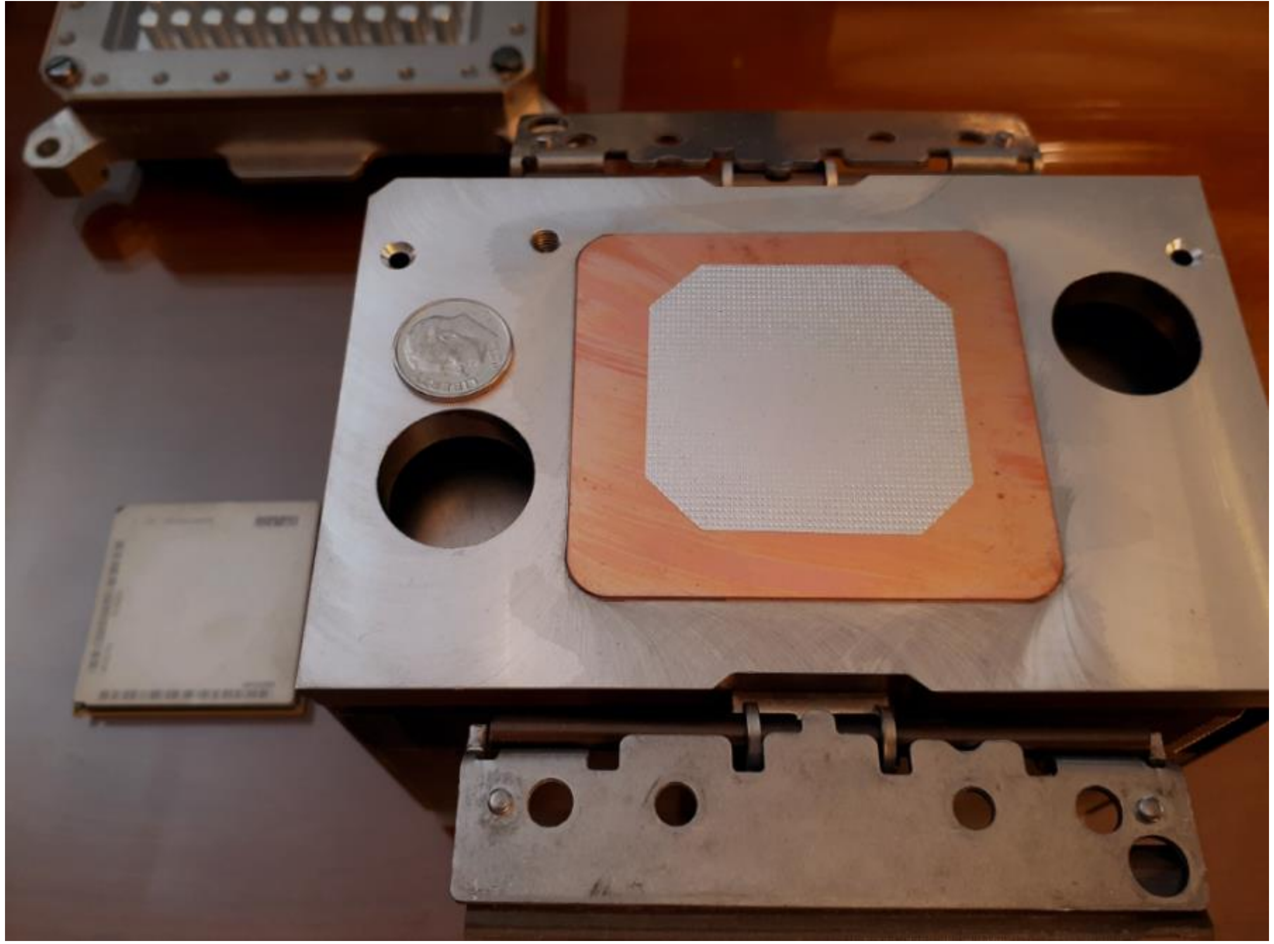
Liquid Immersion: TIM Challenges (Single-, Two-Phase)

Pre-attachment of TIM2 to heat sink or boiling enhancement plate:

- Important for both single- and two-phase expanded heat transfer components
- Pre-attachment process methods for indium foil and patterned indium foils:

  A: Pressure attachment, Indium Heat-Spring® patterned foils (see illustration of TIM2 applied without adhesive);

  B: Indium Corporation NC-702 tacking agent (pending liquid immersion fluid testing for potential for fluid contamination).



Source: Source: T. Jensen, Indium Corporation, USA.   Photograph: DS&A LLC. IBM Power 9 Monza heat pipe/heat sink assembly with Indium Heat-Spring® pre-attached TIM2 by heat sink vendor.

Quantum Computing Systems: Suitable TIMs for Cryogenics

Cryogenic cooling systems for quantum computing: *Other metals and materials as potential TIMs*

Reference (Bradley, Radebaugh) details the following properties for a number of materials operating at cryogenic temperatures (generally, 4K to 300K), as calculated values based on the original works:

- Thermal conductivity
- Specific heat
- Linear thermal expansion
- CTE
- Young's modulus

This materials data is consolidated from a large testing database created by the (US) National Institute of Standards and Technology (NIST) in the 1970 time period and published in multiple out-of-print agency reports. The purpose of the reference document was to consolidate and make available all of this data in a single on-line reference source.

- Materials included are commonly used in cryogenic system assembly.
- A very limited number of TIMs are suitable for use at cryogenic temperatures: See table following.

Source: Bradley, P., Radebaugh, R., "Properties of selected Materials at cryogenic temperatures," CRC Press, Boca Raton FL, 2013. Web: https://tsapps.nist.gov/publication/get_pdf.cfm?pub_id=913059

Quantum Computing Systems: Suitable TIMs for Cryogenics

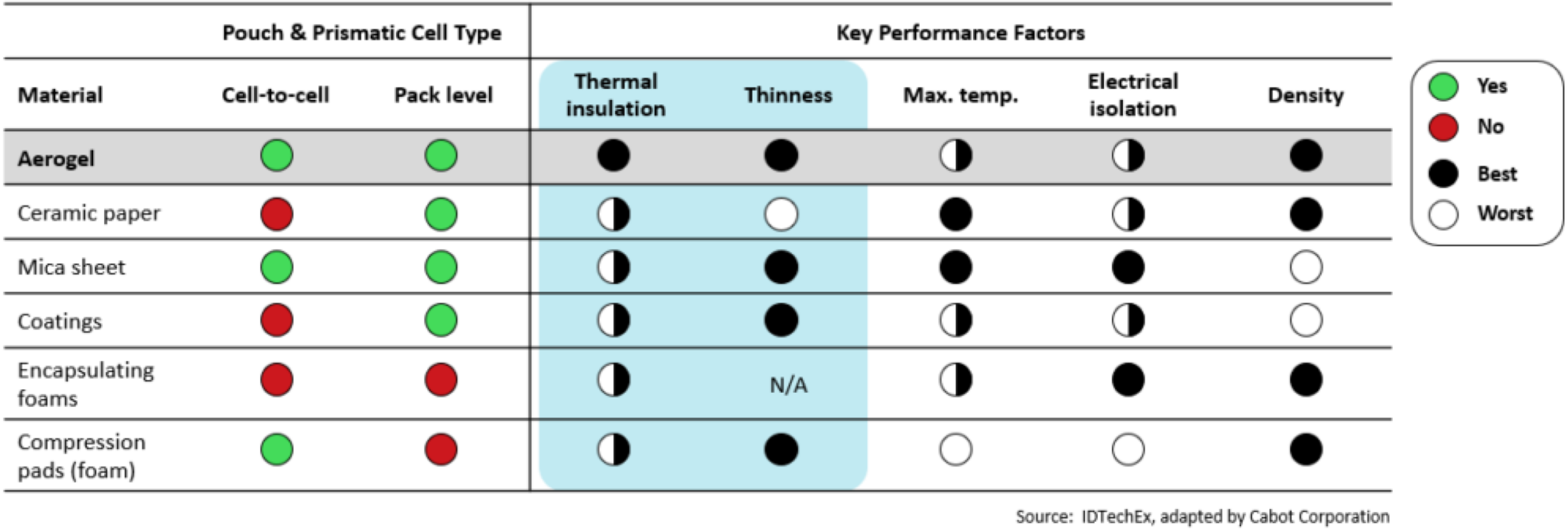
Thermal Interface Materials for Cryogenic Temperatures			
Material	**Vendor(s)**	**General Type**	**Format**
Apiezon®-N	M&I Materials Ltd. (UK)	Thermal Grease	Dispensed compound
Indium foil (In 99.99%)	Many	Metal foil	Pre-form, die-cut
Indium wire (In 99.99%)	Many	Metal wire	Drawn wire
Heat-Spring® (In 99.99%)	Indium Corporation (US)	Metal foil, patterned	Pre-form, die-cut
Tin	Many	Metal foil	Pre-form, die-cut
Indium-copper	Many	Metal foil laminate or clad	Pre-form, die-cut

Source: Dillon, A.; McCusker, K.; Van Dyke, J.; Isler, B.; Christiansen, M.; "Thermal interface material characterization for cryogenic electronic packaging solutions," IOP Conference Series: Materials Science and Engineering 278 (2017) 012054. doi: 10.1088/1757-899X/278/1/012054.

Notes: Apiezon® is a registered trademark of M & I Materials Ltd., UK. Heat-Spring® is a registered mark of Indium Corporation (US).

Aerogel Particles – Energy Storage Materials Development

Cabot aerogel particles for *thermal barrier* developments for energy storage:



	Pouch & Prismatic Cell Type		Key Performance Factors				
Material	**Cell-to-cell**	**Pack level**	**Thermal insulation**	**Thinness**	**Max. temp.**	**Electrical isolation**	**Density**
Aerogel	🟢	🟢	●	●	◑	◑	●
Ceramic paper	🔴	🟢	◑	○	●	◑	●
Mica sheet	🟢	🟢	◑	●	●	●	○
Coatings	🔴	🟢	◑	●	◑	◑	○
Encapsulating foams	🔴	🔴	◑	N/A	◑	●	●
Compression pads (foam)	🟢	🔴	◑	●	○	○	●

🟢	Yes
🔴	No
●	Best
○	Worst

Source: IDTechEx, adapted by Cabot Corporation

Source: Cabot Entera™ Aerogel: Mitigating Thermal Runaway Risk in Battery Electric Vehicles (webinar). https://www.cabotcorp.com/solutions/products-plus/aerogel/particles-for-ev-thermal-barriers

Aerogel Particles – Energy Storage Materials Development

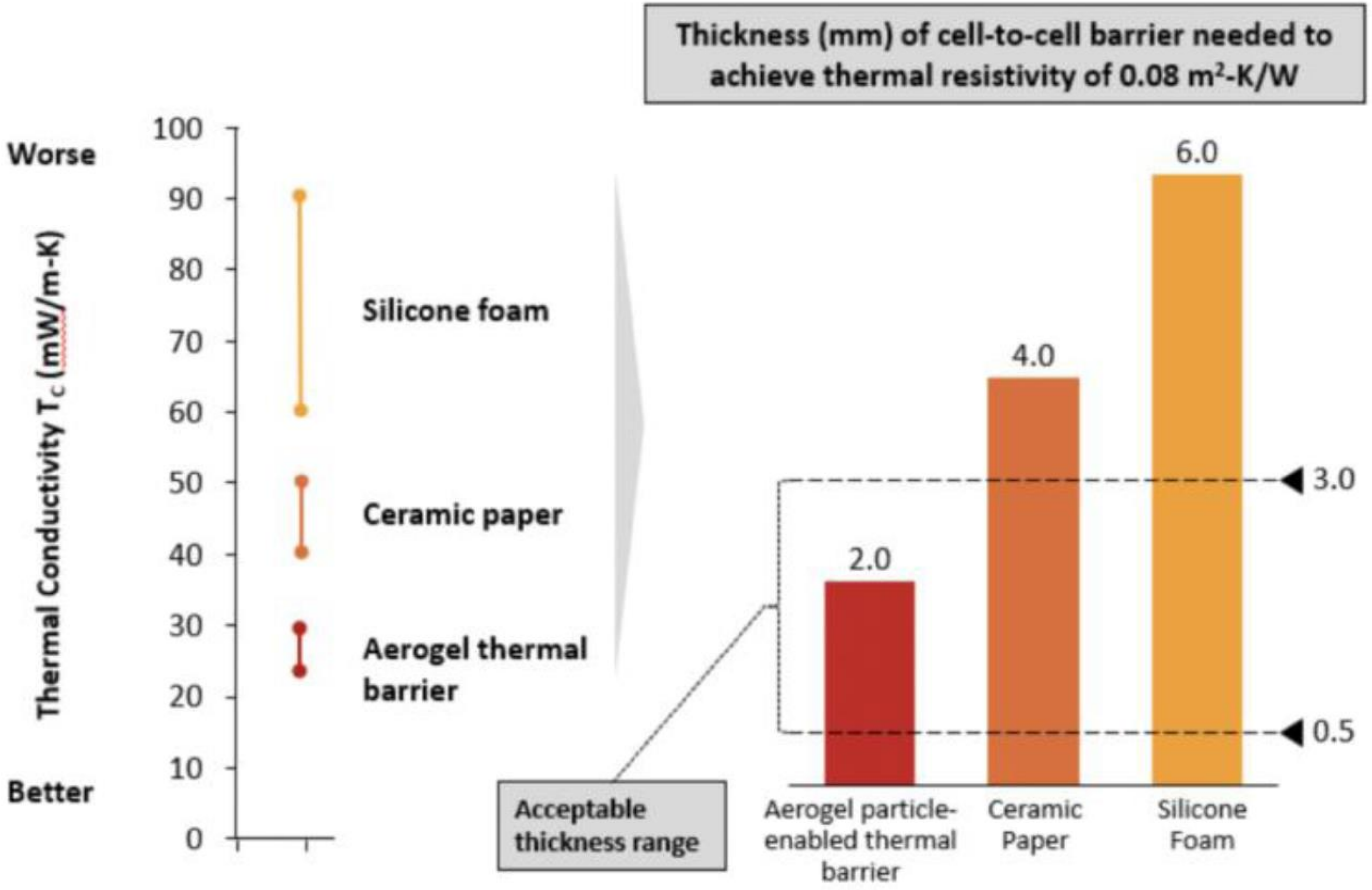
Cabot aerogel particles for *thermal barrier* developments for energy storage:

PRODUCT FEATURES		
PROPERTY	ENTERA EV5200	ENTERA EV5400
Particle size range	0.1-1.2 mm	0.1-0.5 mm
Pore diameter	~20 nm	~20 nm
Bulk density	75-95 kg/m^3	75-100 kg/m^3
Particle thermal conductivity	12 mW/m-K	12 mW/m-K
CAS RN	102262-30-6	102262-30-6
Surface chemistry	Hydrophobic	Hydrophobic

Source: Cabot Entera™ EV5400 aerogel data sheet. https://www.cabotcorp.com/solutions/products-plus/aerogel/particles-for-ev-thermal-barriers

Aerogel Particles – Energy Storage Materials Development

Cabot aerogel particles for *thermal barrier* developments for energy storage:



Note: Thermal resistivity is the thickness needed to achieve a certain level of insulation (measured by dividing thickness by thermal conductivity).

Source: Cabot Entera™ Aerogel: Mitigating Thermal Runaway Risk in Battery Electric Vehicles (webinar). https://www.cabotcorp.com/solutions/products-plus/aerogel/particles-for-ev-thermal-barriers

References – TIMs and TIM Testing, General

1. Gustavsson, M.; Gustafsson, S.E.; "On the Use of Transient Plane Source Sensors for Studying Materials with Direction Dependent Properties", Chalmers University of Technology, IEEE ITCC 26, 2001.
2. Lasance, C.; Murray, C.T.; Saums, D.; Renz, M.; Challenges in Thermal Interface Material Testing, *Proceedings*, Semitherm Symposium 22, Dallas TX USA, March 13-15, 2006.
3. Jarrett, R.N.; Ross, J.; Saums, D.; Comparison of Test Methods for High Performance Thermal Interface Materials, *Proceeding*s, Semitherm Symposium 23, San Jose CA USA, March 2007.
4. Martin, Y.; van Kassel, T.; "High Performance Liquid Metal Thermal Interface for Large Volume Production", IMAPS Symposium 2007, San Jose CA USA, November 12-15, 2007.
5. Khuu, V. "Evaluation of Thermal Interface Mateirals and the Laser Flash Method", Thesis, Doctor of Philosophy, University of Maryland, USA. 2009. Online: https://drum.lib.umd.edu/handle/1903/9873
6. Barnes, C. M., Tuma, P.E., "Practical Considerations Relating to Immersion Cooling of Power Electronics in Traction Systems, IEEE Vehicle Power and Propulsion Conference 2009, Dearborn MI USA.
7. Khuu, V.; Osterman, M.; Bar-Cohen, A.; Pecht, M.; "Effects of Temperature Cycling and Elevated Temperature/Humidity on the Thermal Performance of Thermal Interface Materials," *IEEE Trans. Device Materi. Reliability,* vol. 9, no. 3, pp. 379-391, Sep. 2009; DOI: 10.1109/TDMR.2009.2025367.
8. Liang, Q.Z., et al.; A Three-Dimensional Vertically Aligned Functionalized Multilayer Graphene Architecture: An Approach for Graphene-Based Thermal Interface Materials," *ACS Nano*, Vol. 5, pp. 2392-2401, March 2011.

References – TIMs and TIM Testing, General

9. Shahil, K. M. F., and Balandin, A. A., “Graphene-multilayer Graphene Nanocomposites as Highly Efficient Thermal Interface Materials,” *Nano Letters,* 12, 861-867. 2012.
10. Nelson, C.; Galloway, J.; Fosnot, P.; “Extracting TIM Properties with Localized Transient Pulses,” *Proceedings,* IEEE SEMI-THERM 30, San Jose CA USA, March 2014.
11. Dincer, I. Hamut, H.; Javani, N.; *Thermal Management of Electric Vehicle Battery Systems*, John Wiley and Sons Ltd., 2017; Chapter 3, Phase Change Materials for Passive TMSs.
12. Streb, F., et al.; “Evaluation of Characterization Methods for Solid Thermal Interface Materials,” Mentor Graphics, Mechanical Analysis Division, white paper, Dec. 2017; www.mentor/mechanical.
13. Feng, J.; Mei, Y.; Li, X.; Lu, G.; “Characterizations of a Proposed 3300V Press-pack IGBT Module Using Nanosilver Paste for High-Voltage Applications”, IEEE *Journal of Emerging Selected Topics on Power Electronics*, Vol. 6, No.4, pp. 2245-2253, December 2018.
14. Chen, S.W.; Lin, J.M.; Yang, T.C.; Du, Y.H. “Interfacial Reactions in the Cu/Ga/Co and Cu/Ga/Ni Samples”. *J. Electronic Materials.* **2019**, *48*, 3643, 3654.
15. Fosnot, P.; Galloway, J.; “Localized TIM Characterization Using Deconstructive Analysis,” *Proceedings,* IEEE SEMI-THERM 31, San Jose CA USA, March 2015. Chen, S.W.; Lin, J.M.; Yang, T.C.; Du, Y.H. “Interfacial Reactions in the Cu/Ga/Co and Cu/Ga/Ni Samples”. *J. Electronic Materials.* **2019**, *48*, 3643, 3654.

References – TIMs and TIM Testing, General

16. Fu, Y.; Hansson, J.; Liu, Y.; Chen, S.; Zehri, A.; Samani, M.K.; Wang, N.; Ni, Y.; Zhang, Y.; Zhang, Z.-B.; Wang, Q.; Li, M.; Lu, H.; Sledzinska, M.; Torres, C.M.S.; Volz, S.; Balandin, A.A.; Xu, X.; Liu, J.; "Graphene Related Materials for Thermal Management", 2D Materials J. 7 (2020) 012001. DOI 10.1088/2053-1583/ab48d9. October 22, 2019.
17. Saums, D., "Developments in Thermal Materials for Power Semiconductors", IMAPS France 15th European Advanced Technology Workshop on Micropackaging and Thermal Management, La Rochelle, France, 5-6 February, 2020.
18. Wang, J.; Liu, H.; Zin, J. "Interfacial Reaction Between Sn-Bi Alloy and Ni Substrate". *J. Electronic Materials.* DOI 10.1007/S11664-006-0166-1.
19. Naghibi, S. et al., "Noncuring Graphene Thermal Interface Materials for Advanced Electronics," *Adv. Electron. Mater.*, 6, 1901303. 2020.
20. Wargulski, D., "Failure Analysis of Silver-sinter Layers, Molding Compounds, and Other Composites in Electronics by Pulsed Infrared Thermography", IMAPS France 15th European Advanced Technology Workshop on Micropackaging and Thermal Management, La Rochelle, France, 5-6 February, 2020.
21. Lin, Y.; Genzer, J.; Dickey, M.D.; "Attributes, Fabrication, and Applications of Gallium-based Liquid Metal Particles". *Advanced Science*, 2020 Jun; 7(12): 2000192. DOI: 10.1002/advs.202000192.
22. Mackie, A., Jensen, T., Saums, D.; "Thermal Interface Materials in the HPC Era". IMAPS Device Packaging Conference 2021 (Virtual), April 12-15, 2021.
23. Lee, D.; Kim, C.-L.; Sohn, Y. "Formation and Growth of Intermetallic Compounds during Reactions Between Liquid Gallium and Solid Nickel," *Materials* **2021**, *14,* 5694. https://doi.org/10.3390/ma14195694.

References – TIMs and TIM Testing, General

17. Lazić, Miloš, “Solid Liquid Hybrid TIMs”, IPC APEX 2022, January 22-27, 2022.
18. Wang, J.; Liu, H.; Zin, J. “Interfacial Reaction Between Sn-Bi Alloy and Ni Substrate”. *J. Electronic Materials.* DOI 10.1007/S11664-006-0166-1.
19. Wang, X.; Li, H.; Yao, R.; Lai, W. “Thermal Contact Resistance Optimization of Press-Pack IGBT Device Based on Liquid Metal Thermal Interface Material”, IEEE *Transactions on Power Electronics*, Vol. 37, No. 5, May 2022.
20. Wang, X.; Li, H.; Yao, R.; Lai, W. “Thermal Contact Resistance Optimization of Press-Pack IGBT Device Based on Liquid Metal Thermal Interface Material”, IEEE *Transactions on Power Electronics*, Vol. 37, No. 5, May 2022.
21. Enmark, M.; Murugesan, M.; Nkansah, A.; Fu, Y.; Nilsson, T.M.J.; Liu, J.; “Reliability Characterization of Graphene Enhanced Thermal Interface Material for Electronics Cooling Applications”, IMAPS Nordic 2022 Proceedings, Gothenburg, Sweden, July 12-14, 2022.
22. Jensen, T., “Metal TIMs for High Performance BGA Packages,” Semi-Therm 40 Symposium, San Jose CA USA, March 23-28, 2024.
23. ASTM D 5470-12, issued by ASTM International, is available for purchase and download at www.astm.org
24. JESD51, JEDEC “Standard Transient Dual Interface Test Method for the Measurement of the Thermal Resistance Junction-to-Case of Semiconductor Devices with Heat Flow Through a Single Path,” Online, www.jedec.org.
25. Heat-Spring® is a Registered Mark of Indium Corporation.
26. Kapton® and Kapton® MT are Registered Marks of DuPont de Nemours, Wilmington DE USA.
27. Other trademarks and Registered Marks are the property of their respective owners.

References – Thermal Materials, Capacitors

1. Parler, S.G., “Thermal Modeling of Aluminum Electrolytic Capacitors,” IEEE Industry Applications Society Conference, Phoenix AZ USA, October 3-7, 1999.
2. TDK Electronics, “Thermal Design of Capacitors for Power Electronics,” Web: https://www.tdk-electronics.tdk.com/pdf-thermaldesign.pdf
3. Smith, Ben, “Energy and Power Handling Capabilities of Thin Film and Ceramic Capacitors,” AVX Corporation (2002).
4. Callegaro, A.D., et al., “Bus Bar Design for High-Power Inverters,” IEEE Transactions on Power Electronics,, 2017. Web: https://doi 10.1109/TPEL.2017.2691668.
5. Zhang, Y.X., et al., “Theoretical Connection from the Dielectric Constant of Films to the Capacitance of Capacitors Under High Temperature. *High Voltage*, 8(4), 707–716 (2023). https://doi.org/10.1049/hve2.12308
6. Demcko, R., “Capacitor Reliability Seminar,” AVX Corporation (2022).
7. Sun, X.-W., et al., “Effect of Thermal Stress on the Life of DC Link Capacitors for the Smart Grid,” Research Square (unpublished, open for peer review). Web: https://doi.org/10.21203/rs.3.rs-3847779/v1

References – TIMs for Liquid Immersion

1. ASTM D 5470-12, issued by ASTM International, is available for purchase and download at www.astm.org.
2. P.E. Tuma, “The merits of open bath immersion coolnig of datacom equipment,” IEEE 26th Annual Semiconductor Thermal Measurement and Management Symposium (SEMI-THERM), San Jose CA USA, March 2010.
3. C.M. Barnes, P.E. Tuma, “Practical Considerations Relating to Immersion Cooling of Power Electronics in Traction Systems,” *IEEE Transactions on Power Electronics*, 25(9), pp. 2478-2485, September 2010.
4. M.S. El-Genk, A.F. Ali, “Enhanced nucleate boiling on copper micro-porous surfaces,” *International J. Multiphysics Flow*, vol. 36, no. 10, pp. 780-792, October 2010.
5. L.A. Campbell, R.C. Chu, M.P. David, M.J. Ellsworth Jr., M.K. Iyengar, and R.E. Simons, “Multi-fluid, two-phase immersion cooling of electronic component(s),” US20130105120A1, 02 May 2013.
6. Polyalphaolefins, general: Fink, Johannes K., Science Direct, 2013. See https://www.sciencedirect.com/topics/engineering/poly-alpha-olefins/pdf
7. R. Brink, “Asperitas Immersed Cooling,” white paper. See: http://www.asperitas.com
8. P.E. Tuma, “Emerging Applications for Boiling Enhancement Coatings in Immersion Cooling,” 3M Company, April 2016.
9. K.C. Leong, J.Y. Ho, K.K. Wong, “A critical review of pool and flow boiling heat transfer of dielectric fluids on enhanced surfaces,” *Applied Thermal Engineering,* vol. 112, pp. 99-1019, February 2017.

References – TIMs for Liquid Immersion

10. Source: Sarangi, S.; McAfee, E.; Damm, D.; Gullbrand, J.; Intel Corporation, “Single-Phase Immersion Cooling Performance in Intel Servers with Immersion Influenced Heat Sink Design,” SEMI-THERM 38 Symposium, San Jose CA USA (Virtual, March 2022).
11. Example, fluid formulation: http://multimedia.3m.com/mws/media/1998180/3mtm-novectm-7100-engineered-fluid
12. Fluorinert™ and Novec™ are trademarks of 3M Company. Unilever®, Vaseline® are Registered Marks of Unilever plc.

References – TIMs for Cryogenic Temperatures

1. Sharma, R.; Singh, M.; Sonara, D., “Design development and testing of vacuum compatible seals at cryogenic temperatures.” Institute for Plasma Research, Gujarat, India. Web: http://www.ipr.res.in/ivsns05/manuscript/PVS6.pdf
2. Sebastiano, F., et. Al, (Invited) “Cryo-CMOS Electronic Control for Scalable Quantum Computing,” *DAC 17 Proceedings*, ACM, ISBN 978-1-4503-4927-7/17/06.
3. Bradley, P. and Radebaugh, R. (2013), “Properties of selected Materials at cryogenic temperatures,” CRC Press, Boca Raton, FL. Web: https://tsapps.nist.gov/publication/get_pdf.cfm?pub_id=913059
4. Graphite Metallizing Corporation, “Graphalloy®: Maintaining lubricity at cryogenic temperatures,” Turbomachinery International, May/June 2014.
5. Keltner, N.J.; Mulcahey, T.I.; Ghiaasiaan, S.M., “Graphite foam as a cryogenic heat exchanger,” *Cryocoolers 18 Proceedings*, International Cryocooler Conference, Boulder CO USA, 2014.
6. Graphite Metallizing Corporation, “Graphalloy®: Maintaining Lubricity at Cryogenic Temperatures,” *Turbomachinery International*, May/June 2014.
7. Custom Thermoelectric Inc., Specification Sheet TF-IF5050, Pure indium foil thermal interface material, 04-16-2020. www.customthermoelectric.com
8. Park, J-S, et. Al, “A Fully Integrated Cryo-CMOS SoC for Qubit Control in Quantum Computers Capable of State Manipulation, Readout and High-Speed Gate Pulsing of Spin Qubits in Intel 22nm FFL FinFET Technology,” *Proceedings*, IEEE International Solid-State Circuits Conference 2021.
9. Nataj, Z., et al., “Cryogenic Characteristics of Graphene Composites – Evolution from Thermal Conductors to Thermal Insulators,” *Nature* Communications, 2023, 14:3190. Web: https://doi.org/10.1038/s41467-023-38508-3

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