Thermo Project: Assessment of high temperature thermal conductivity reference materials, inter-comparison protocols and methodology / early indication of results

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12 May 2016

EMRP Thermo 2nd Stakeholder Meeting at NPL

Outline

- Objectives
- Results of surveys
- Provisional assessments
- Selection of candidate reference specimens
- Round-Robin Comparison
- Star-Shape Comparison
- Early Indication of Results (PTB)

Objectives

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- One of the aims of this project is to develop reference materials with thermal conductivity in the range 0.02 $Wm^{-1}K^{-1}$ to 1 $Wm^{-1}K^{-1}$ and with a target maximum temperature of 800°C (revised to 650°C).
- The first stage is the provisional assessment of candidate reference materials. The aim is to select one or two best candidate reference materials for further detailed assessment within the next stage.
- The second stage is the detailed assessment of candidate reference material(s) – selection of candidate reference specimens.
- The third stage is the inter-laboratory comparisons.

Results of Surveys

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- Results of surveys at the beginning of the Thermo project

  Sixteen companies and organizations have contributed to the survey that leads to a shortlist of potential candidate materials:

- Low density calcium silicate: ‘LDCaSi’
- High density calcium silicate: ‘HDCaSi-N’
- Amorphous silica: ‘AmSi’
- Exfoliated vermiculite: ‘EV’

Provisional Assessments

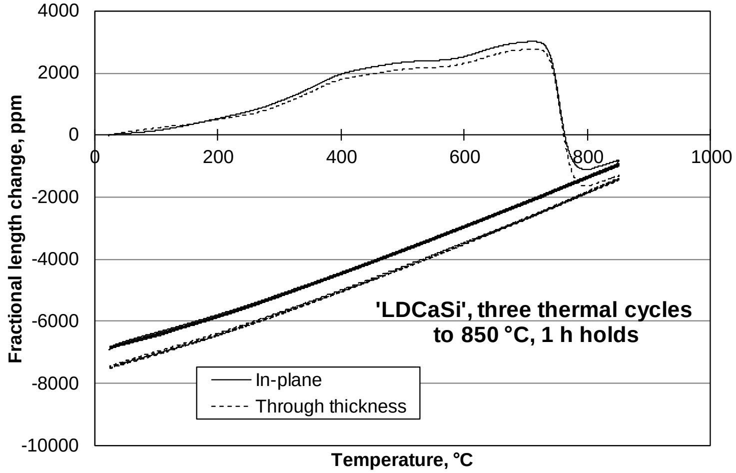
- Test Matrix

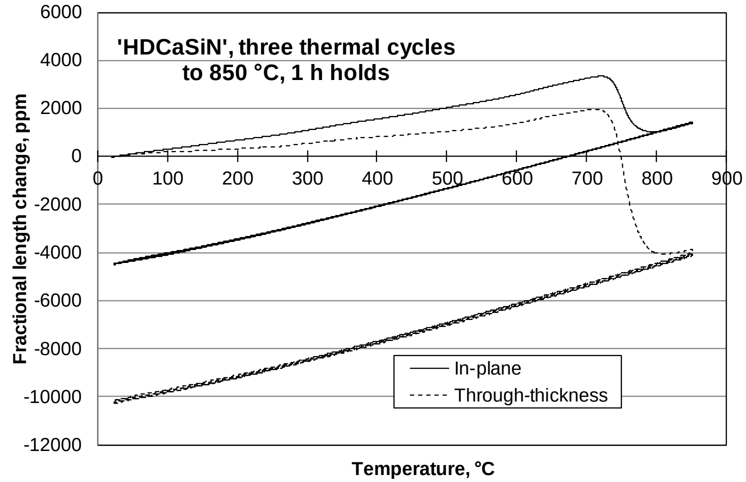
No.	Item	Owner
1	XRF for element composition of the material	NPL
2	XRD for material component	NPL
3	SEM for microstructure and bases for long term stability	NPL
4	Mechanical stability and isotropy aspect of the material via Thermal expansion	NPL
5	Chemical stability via TGA	NPL
6	Chemical stability via DSC	NPL
7	Uniformity of fresh material via density distribution	NPL

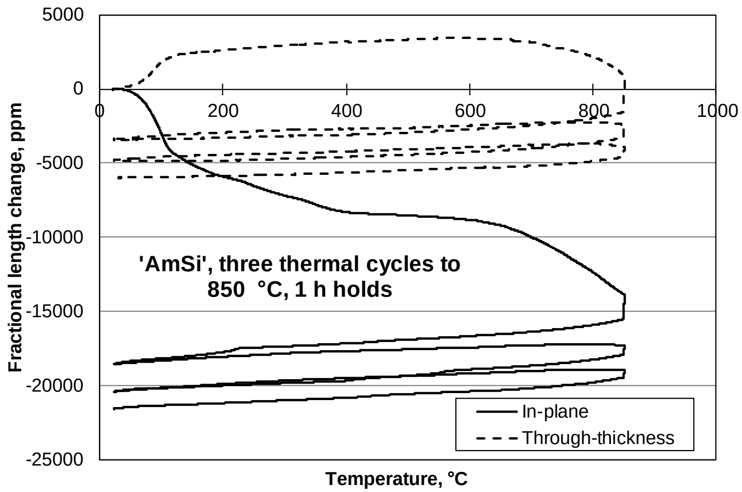
No.	Item	Owner
8	Heat treatment curve	NPL
9	Shrinkage/warp	NPL
10	Thermal cycling and compressive load tests at high temperatures	CMI
11	Heat treatment and machining trial tests	MKEH
12	Transparency	LNE
13	Material handleability	LNE
14*	Density distribution within heat treated specimens	PTB

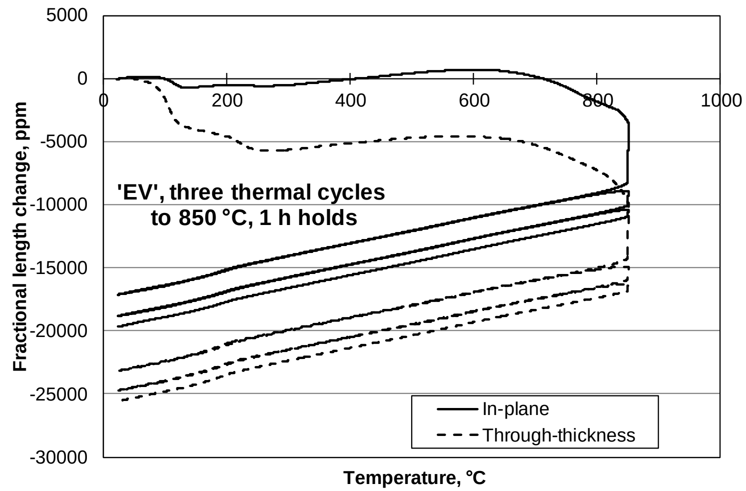
Provisional Assessments

- Mechanical and Dimensional Stability under Thermal Cycling and Isotropy Aspects – Thermal Expansion



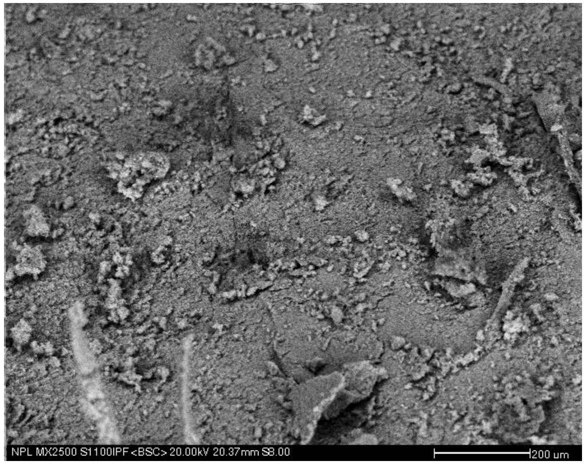




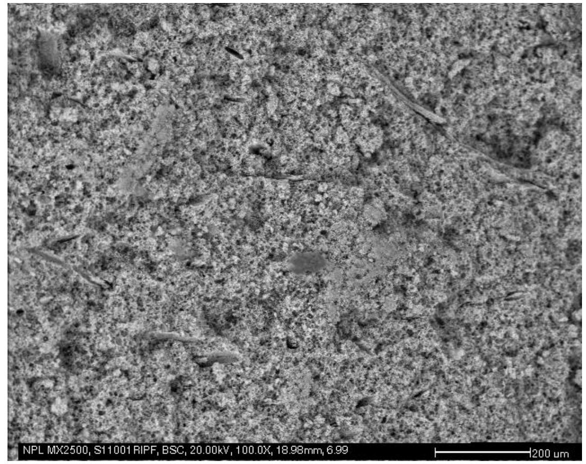


Provisional Assessments

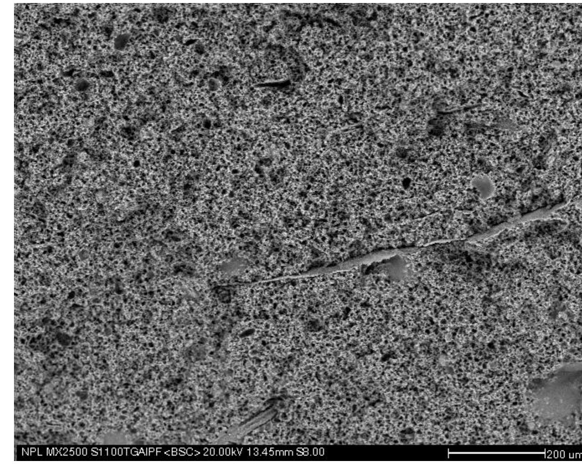
- Microstructure – SEM Micrographs



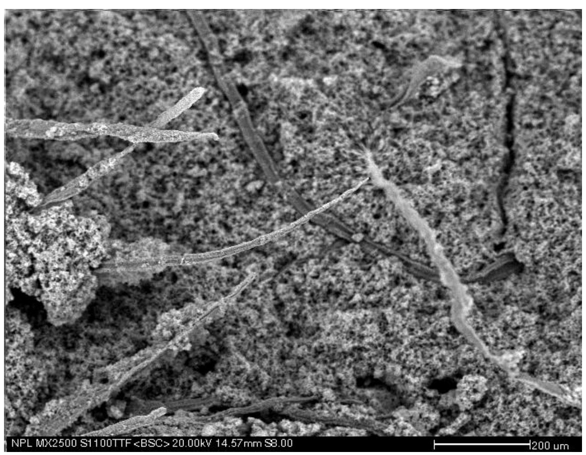
(a) as received, in-plane



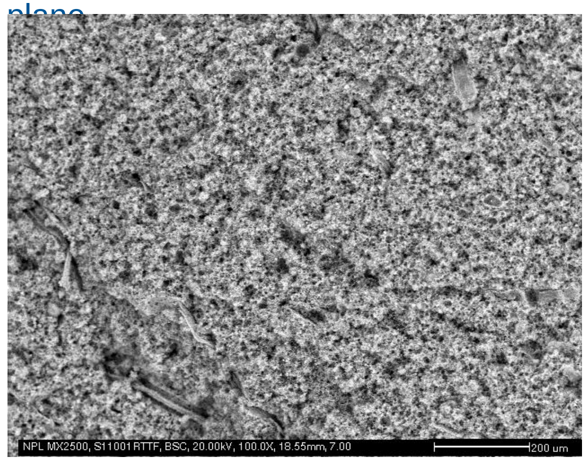
(c) after 1st HT at 850 ° C for 24 hrs, in-plane



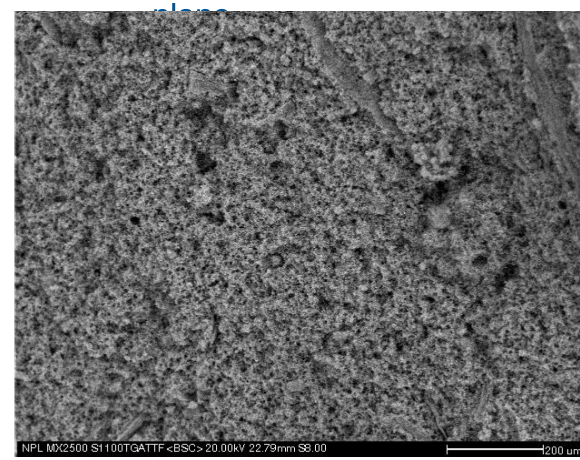
(e) post TGA, in-plane



(b) as received, through-thickness



(d) after 1st HT at 850 ° C for 24 hrs, through-thickness

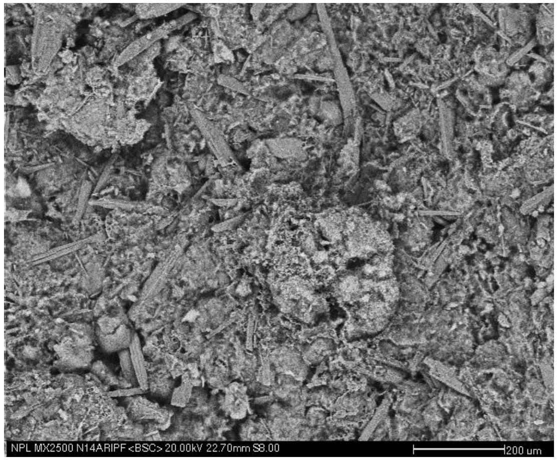


(f) post TGA, through-thickness

Backscattered electron images of fractured surfaces of LDCaSi

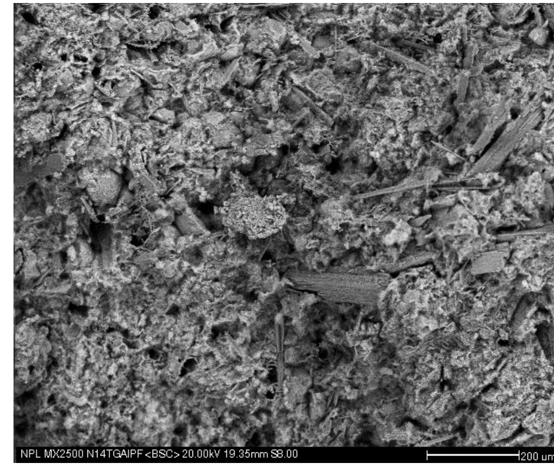
Provisional Assessments

National Physical Laboratory



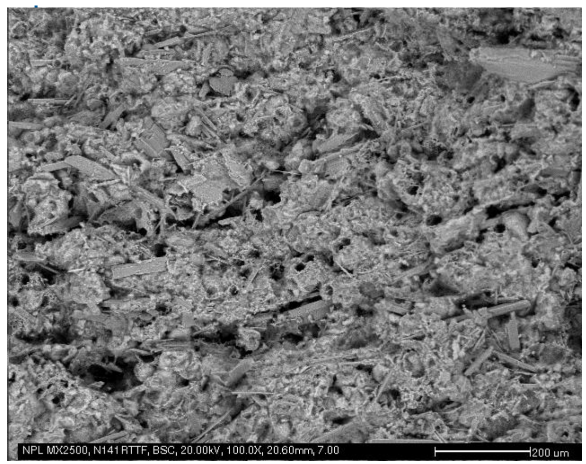
(a) as received, in-plane

(c) after 1st HT at 850 ° C for 24 hrs, in-

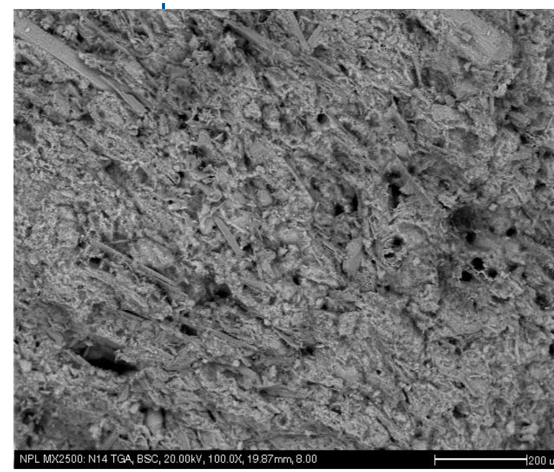


(e) post TGA, in-

(b) as received, through-thickness



(d) after 1st HT at 850 ° C for 24 hrs, through-thickness



(f) post TGA, through-thickness

Backscattered electron images of fractured surfaces of HDCaSi-N

Conclusions of Provisional Assessments

- The two most promising candidate materials, LDCaSi and HDCaSi-N will be further developed into high temperature thermal conductivity reference materials in the next stage.
- Although permanent changes were observed during the first heat treatment, these two materials are dimensionally, mechanically and chemically stable after the first heat treatment.
- The microstructures of these two materials remain stable, and they are more robust and easier to handle.
- The dimensions and mass of the two candidate materials remain stable after thermal cycling and compressive load testing at temperatures up 850°C and pressure up to 5050Pa.
- The heat treatment curves have been adjusted after the trial tests at large scale.
- Not expecting any transparency issues for 50mm thick specimens. The normal hemispherical spectral transmittance of a 3 mm thick HDCaSi-N is less than 0.01, and that of a 3 mm thick LDCaSi is less than 0.025.
- **However, it is important that the specimens of reference materials meet the requirement for material uniformity, i.e. density varies within 2 %, hence, there need to be a stringent selection process to ensure the material uniformity.**

Selection of Candidate Reference Specimens

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(1) Select the specimens that meet the criteria of bulk density variation within +/-1 % and the density band has the maximum number of specimens based on the density, thickness and flatness measurements at MKEH.

↓

(2) Select the specimens based on the confirmation tests at NPL on those within the 800 $kg{\cdot}m^3$ to 816 $kg{\cdot}m^3$ density band. The tests include bulk density, thickness and flatness measurements.

↓

(3) Acoustic non-destructive density mapping of each specimen at NPL on the specimens that passed stage 2. Select the ones with density mapping showing variation:

For specimens with 500mm by 500mm lateral dimensions – the density vary within +/-1% for all 25 points measured at the centre of each grid (100mm by 100mm).

For specimens with 500mm by 500mm lateral dimensions – the density vary within +/-1% for the centre 300mm by 300mm area and the average density in the surrounding area agree with the average density in the centre area within +/-1%.

For specimens with 320mm by 320mm lateral dimensions – the density vary within +/-1% for all 9 points measured at the centre of each grid (100mm by 100mm).

↓

(4) Thermal conductivity measurements at NPL on specimens that passed all the three stage selection. Select those with thermal conductivity varying within +/- 1 % at 20°C.

Selected Candidate Reference Specimens – 15-off HDCaSi-N

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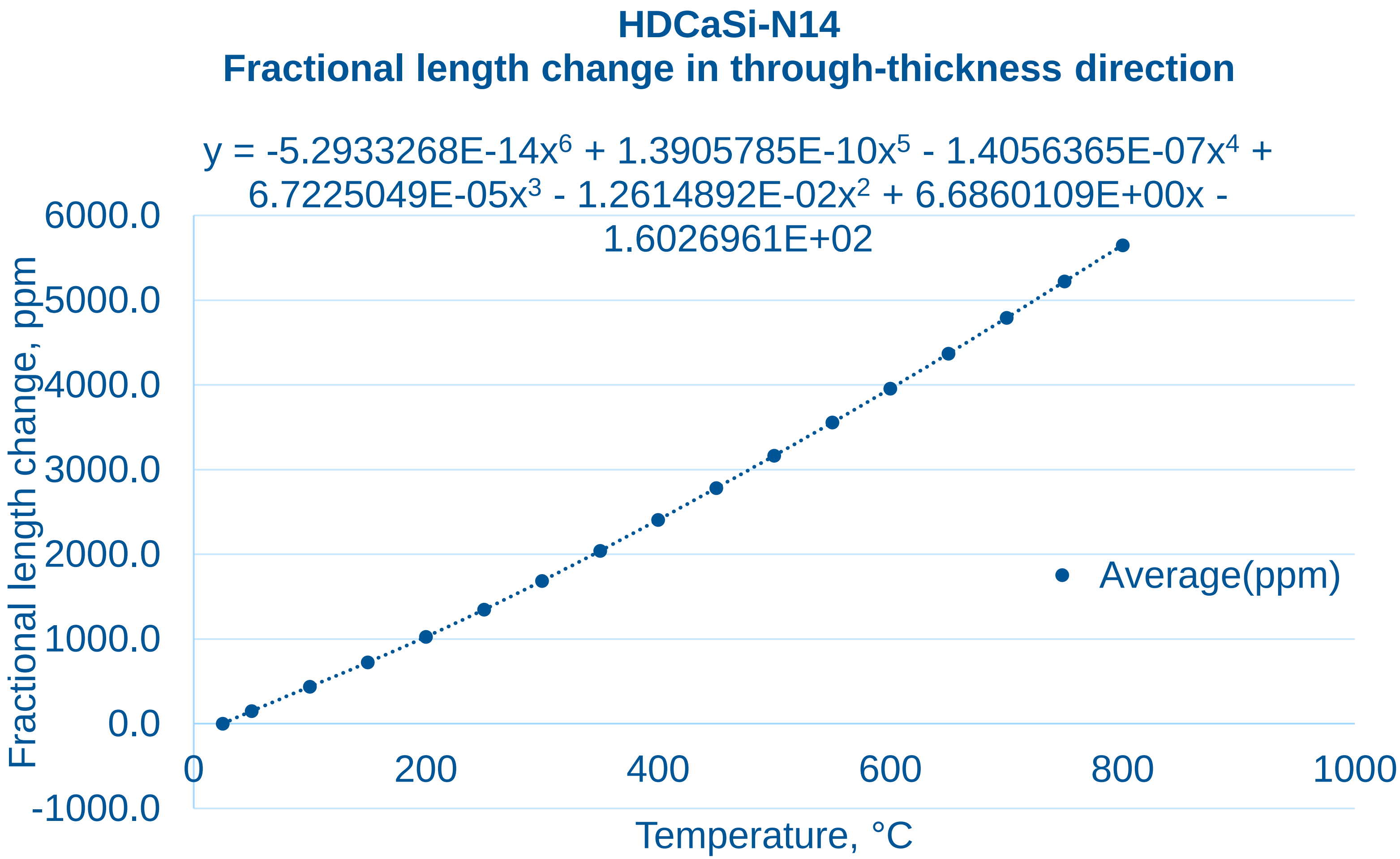
Specimen No.	Variation of measured thermal conductivity at 20°C	Density	Density variation	Owner
	(λ -λ_ave)*100/λ_ave (%)	kg/m3	(%)	
30635 0014 BL1	-0.3	811.8	0.2	RR
30635 0014 BLM 3	0.7	811.3	0.1	FIW
30635 0014 BLM 4	-0.6	805.5	-0.6	RR
30635 0015 CRM8	0.1	814.2	0.5	backup
30635 0016 DL 2	0.7	814.5	0.5	FIW
30635 0018 FLM4	-0.6	814.1	0.4	RR
30635 0019 GRM 9	0.5	812.4	0.2	LNE commercial
30635 0019 GR11	-0.5	803.3	-0.9	RR
30635 0020 HL2	0.4	815.2	0.6	LNE in house
30635 0020 HLM3	0.3	810.2	0.0	MKEH HFM
30635 0021 L5	0.1	808.4	-0.3	NPL HT
30635 0021 LLM7	0.1	810.5	0.0	MKEH HTTCMA
30635 0021 LM7	-0.6	812.8	0.3	PTB
30635 0021 RM5	-0.2	809.7	-0.1	CMI
30635 0021 RRM7	0.0	803.3	-0.9	NPL LT

colour coding	
Use 500 by 500 mm	lateral diamension: 500 by 500mm and can use the whole 500 by 500 mm
Use 320 by 320 mm	lateral diamension: 320 by 320 mm and can use the whole 320 by 320 mm
Use centre 320 by 320 mm only	lateral diamension: 500 by 500mm and can only use the centre 320 by 320 mm
At position A1 (edge) no visable cracks but high values. can only use center 320 by 320mm.	

LDCaSi – high damage rate during transportation

Thermal Expansion of the Candidate Reference Specimens



Predicted bowing of the HDCaSi-N14 specimens

Apparatus	LNE in-house HTGHP	NPL LTGHP	NPL HTGHP	CMI HTGHP	MKEH HTTCMA
Lateral dimensions of heater plates (mm)	318 by 318	305 by 305	Dia. 305	Dia. 306	Dia. 320
Metering area (mm)	150 by 150	152.5 by 152.5	Dia. 150	Dia. 150	Dia. 200
Radius for specimen discs or half the longest length of the centre octagon area (see Fig.2)(mm)	165.06	165.06	152.5	152.5 (Specimen 305mm dia.)	152.5 (Specimen 305mm dia.)
Specimen thickness of the 1st set (mm)	47.4	46.4	46.4	46.4	46.4
Temperature drop of the 1st set of specimens (K)	50	20	50	50	50
Specimen bowing for the 1st set of specimens(mm)	0.102	0.042	0.089	0.089	0.089 (Specimen 305mm dia.)
Specimen thickness of the 2nd set (mm)	40	40	40	40	40
Temperature drop of the 2nd set of specimens (K)	40	20	40	40	40
Specimen bowing for the 2nd set of specimens(mm)	0.097	0.048	0.083	0.083	0.083 (Specimen 305mm dia.)

Specimen cut for the Round-Robin

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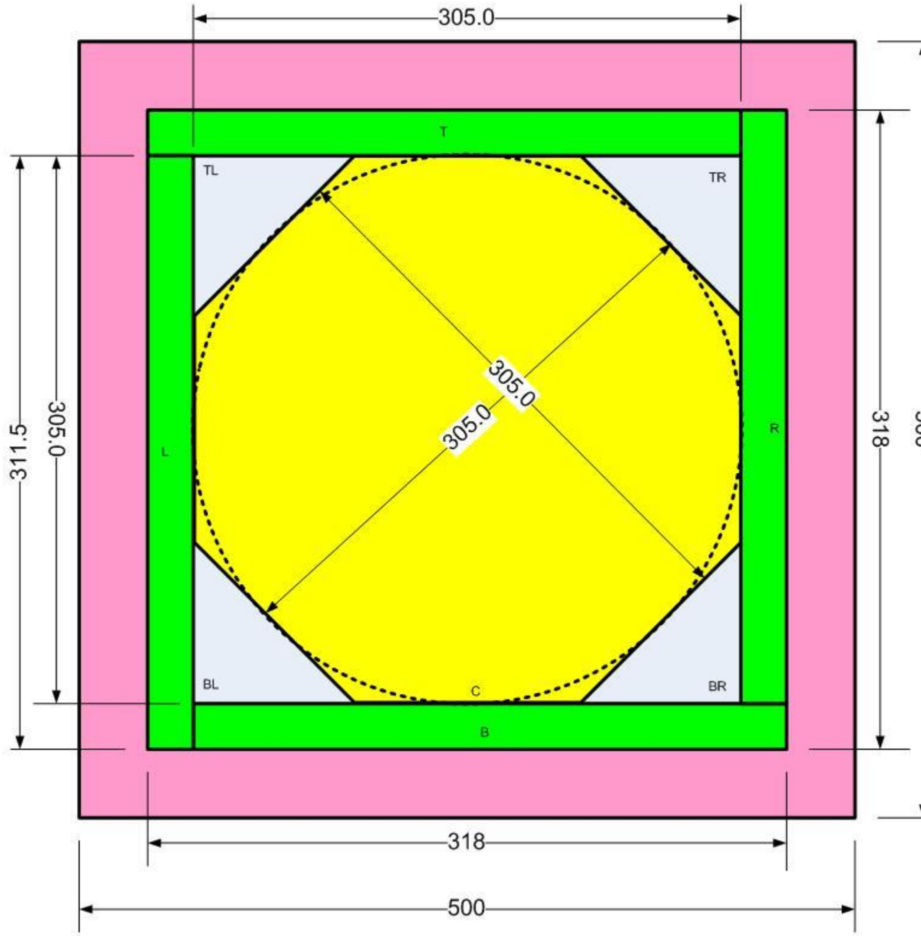
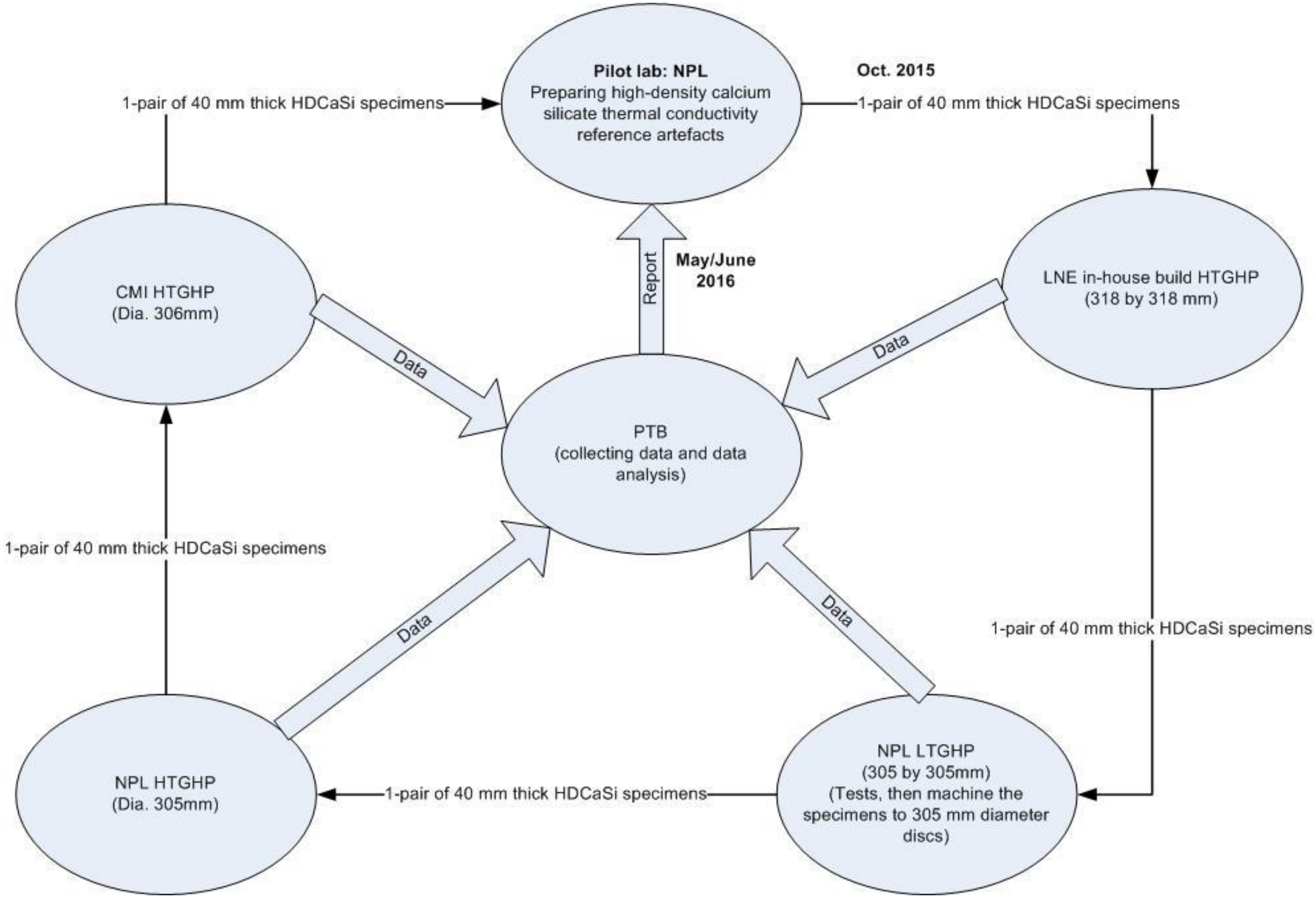
Cutting plan for the lateral dimensions of the RR specimens



4-off HDCaSi-N14 blocks supplied with lateral dimensions 500 by 500mm and 50mm thick.
Requirements for machining:
Thickness: 47.4mm+/-0.1mm for 2-off specimens;
40mm +/-0.1mm for 2-off specimens.
Surface flatness: within 0.05 mm
Surface parallelism: within 0.05 mm
The centre piece and each of the corners and strips shall be numbered using a pencil for its position (C, T, B, L, R, TL, TR, BL and BR).

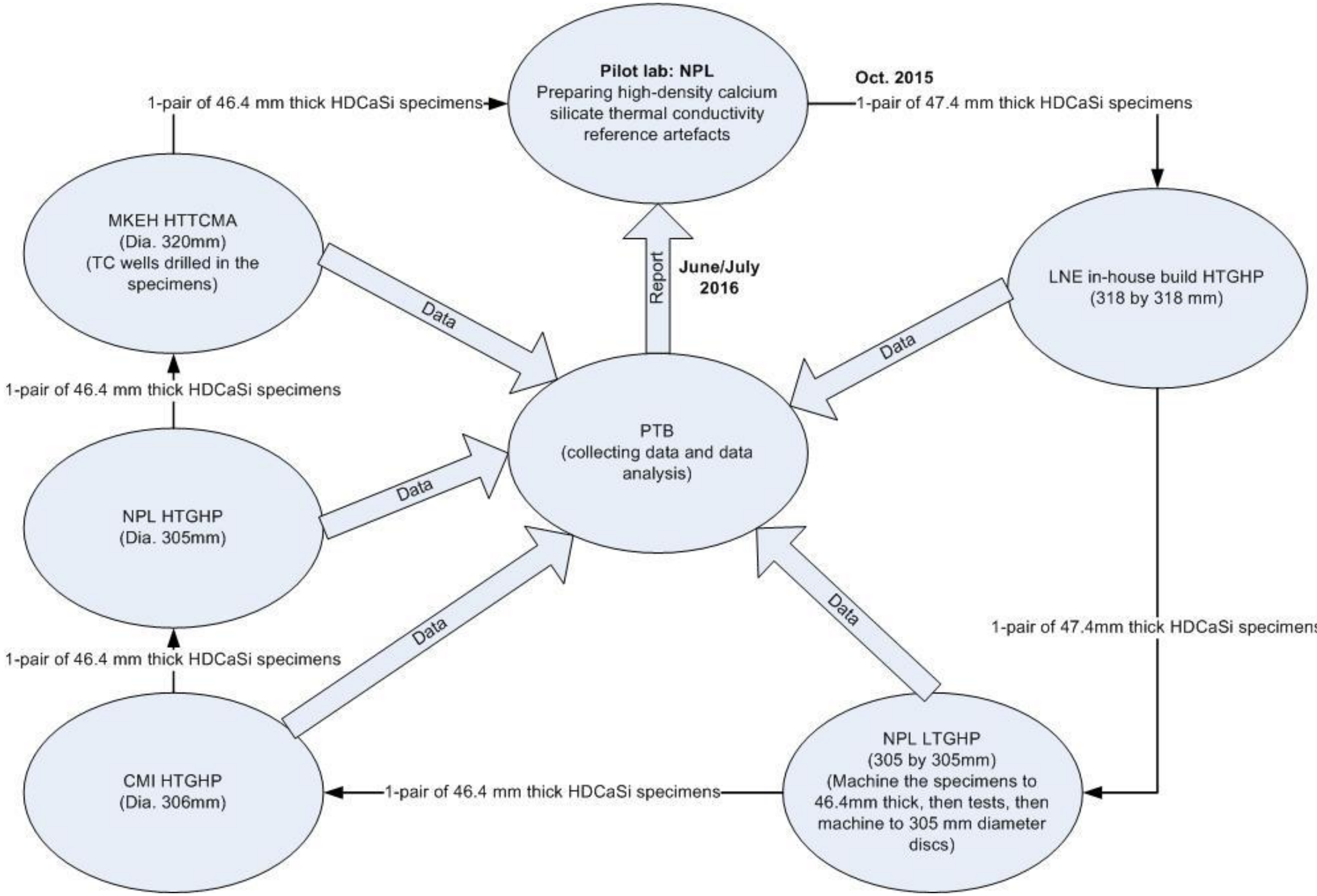
Unit: mm
Contact: Jiyu Wu Ext.6045

Thermo Round-Robin: 40mm thick specimens



Ready for submission to BIPM KCDB as European Regional Comparison

Thermo Round-Robin: 47 mm thick specimen



Ready for submission to BIPM KCDB as European Regional Comparison

Thermo Round-Robin Progress

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HDCaSi	46.4 mm
HDCaSi	40 mm

Year	Month	Week	Week number	LNE In-house HTGHP	NPL LTGHP	NPL HTGHP	CMI HTGHP	MKEH HTTCMA
2015	Oct	40	1	HD 47.4 mm				
		41	2					
		42	3					
		43	4					
		44	5					
	Nov	45	6					
		46	7	47.4mm from LNE to NPL				
		47	8					
		48	9		Re-Machined to 46.4mm			
	Dec	49	10	HD 40mm				
		50	11					
		51	12					
		52	13	Holidays				
2016	Jan.	1	14					
		2	15	40mm from LNE to NPL				
		3	16					
		4	17		HD 46.4 mm			
		5	18					
	Feb	6	19		HD 40mm			
		7	20			Machine to 305 disc, 46.4 mm		
		8	21					
		9	22			Machine to 305 disc, 40mm	46.4mm from NPL to CMI	
	March	10	23			environmental chamber repair		
		11	24			HD 40mm	**HD 46.4mm**	
		12	25					
		13	26					
		14	27				46.4mm from CMI to NPL	
	April	15	28					
		16	29					
		17	30			**HD 46.4 mm**	40mm from NPL to CMI	
		18	31				HD 40 mm	
	May	19	32					
		20	33					
		21	34				40mm from CMI to NPL	
		22	35					
	June	23	36			46.4mm from NPL to MKEH		
		24	37					
		25	38					HD46.4 mm
		26	39					
		27	40					

Thermo Star-shape Comparison

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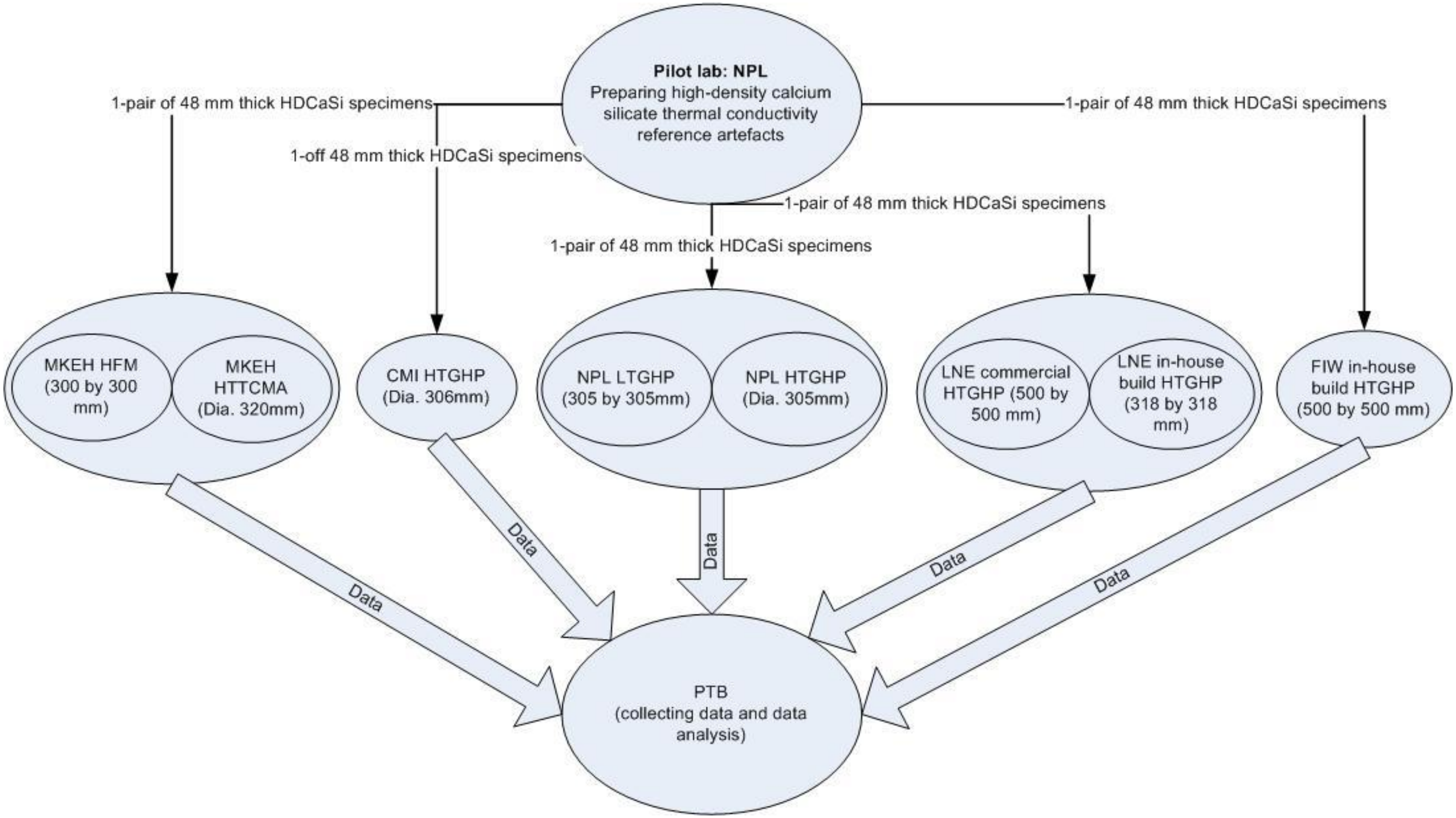

Register the Star-shape comparison with EURAMET TC-T

Repeat 3 times, Centre-guard imbalance at 150° C and 550° C, Short time repeatability tests

Early Indication of Results by PTB

Q & A

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The National Physical Laboratory is operated by NPL Management Ltd, a wholly-owned company of the Department for Business, Innovation and Skills (BIS).

Thermal Contact Resistance

- Estimation of the effect in uncertainty budget
  - Limit of the LNE model: radiation, conduction, estimate gap thickness, small dT
  - CMI will present a numerical model for estimation
  - Convection?

    Critical Rayleigh number Ra=1708, above which -> Benard cell; above Ra=50000 then turbulent free convection

    Air properties evaluated at $T_f = T_c$.

$$Ra = \frac{g\beta\delta^3(Th - Tc)}{v^2} P_r$$

- Determine thermal contact resistance (MKEH)