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Characterization of a High-Temperature Thermal Conductivity Reference Material

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Abstract The development of thermal conductivity reference materials for high-temperature insulation consists of three stages: provisional assessment of candidate reference materials, detailed assessment of candidate reference material(s) and corresponding inter-laboratory comparisons. This paper describes the detailed characterization of a candidate high-temperature thermal conductivity reference material, a high-density calcium silicate (HDCaSi-N). The selection criteria, assessments of uniformity and stability, the thermal expansion behavior and their effects on the thermal conductivity reference specimens are presented in the paper. The uniformity assessments include the thermal expansion variation in orthogonal orientations and different locations and from different boards, as well as thermal conductivity variation within the batch of the specimens. The dimensional stability assessment in terms of thermal expansion and the short-term stability in terms of thermal conductivity are also presented.

Keywords High-temperature guarded hot plate · Reference material · Technical insulation · Thermal conductivity · Thermal expansion

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List of Symbols

a	$a = T_\mathrm{m}$ is the mean specimen temperature (K)
b	$b = \frac{dT}{dz}$ is the linear temperature gradient through the thickness of the specimen (K·m^{-1})
E	Young's modulus (kg · m^{-1} · s^{-2})
h	Half the thickness of the specimen (m)
r	Radius (m)
R	Radius of the round disk (m)
T_m	Mean specimen temperature (K)
w	Displacement component for z direction (m)
Δw	Estimated bowing of the specimen (m)
x, y	The Cartesian coordinates in the plane perpendicular to z direction (m)
z	The Cartesian coordinate along the thickness of the specimen with the origin at the middle point of the thickness (m)

Greek Symbols

α	Mean thermal expansion coefficient in in-plane direction (K^{-1})
ν	Poisson's ratio

1 Introduction

To support the implementation of the new European regulations (EU NO. 305/2011) and to resolve the discrepancies between reference laboratories in thermal conductivity measurements, five European National Measurement Institutes, the National Physical Laboratory (NPL), the Laboratoire National de Métrologie et d'Essais (LNE), the Physikalisch-Technische Bundesanstalt (PTB), the Magyar Kereskedelmi Engedélyezési Hivatal (MKEH) and the Český Metrologický Institut (CMI), have been working together on a Joint Research Project, 'Thermo' funded by the European Metrology Research Program (EMRP). One of the aims of this project is to develop reference materials with thermal conductivity in the range 0.02 W · m^{-1} · K^{-1}–1 W · m^{-1} · K^{-1} and with a target maximum temperature of 650 °C. This is because up till now there has been no high-temperature reference material available with an appropriate level of thermal conductivity that could be used to aid in the investigation of disagreements in measurement results between laboratories. The development of the reference materials consists of three stages: provisional assessment of candidate reference materials, detailed characterization of the selected material(s) and corresponding inter-laboratory comparisons.

   This paper describes the detailed characterization of the proposed candidate high-temperature thermal conductivity reference material—a high-density calcium silicate (HDCaSi-N). Although the provisional assessment of candidate reference materials [1] had suggested that both HDCaSi-N and a low-density calcium silicate (LDCaSi) were the two promising candidates, it was found out later that the LDCaSi was not as robust as the HDCaSi-N, as there was a very high rate of damage during transportation

of the specimens. Since the reference materials for inter-laboratory comparisons and routine check of apparatus need to be robust, the detailed characterization was focused on the HDCaSi-N material.

The selection criteria, assessments of uniformity and stability, thermal expansion behavior and their effect on the thermal conductivity reference specimens are presented in the paper. Attempts were made to indicate the density variation within a specimen via a non-destructive examination (NDE) method. The uniformity assessments include the thermal expansion variation in orthogonal orientations and different locations, as well as thermal conductivity variation within the batch of the specimens. The mechanical stability assessment in terms of thermal expansion and short-term stability in terms of thermal conductivity are also presented.

After implementing a set of selection criteria including the variations of density and thermal conductivity at 20 °C within +/-1%, only a subset of the specimens from the batch of candidate reference material have met the acceptance criteria. These specimens will be used for the inter-laboratory comparisons in the third stage.

2 The Material and Preparation of Specimens

During the provisional assessment of candidate reference materials, the chemical composition and microstructure of the candidate material HDCaSi-N (batch No. 30639), in particular its stability under thermal cycling had been analyzed [1]. The X-ray fluorescence spectrometry (XRF) and X-ray diffraction (XRD) techniques had been applied to reveal the composition of each of the four materials. The microstructure of the candidate reference material, HDCaSi-N (before and after heat treatment), had been illustrated in the micrographs obtained using a scanning electron microscope (SEM). The XRD measurements performed on HDCaSi-N samples had revealed the major phases: hydrated calcium silicate ($Ca_2SiO_4 \cdot H_2O$) and quartz had been identified in as-received specimens; calcium silicate and quartz had been identified in specimens that had been held 24 h in a thermogravimetric analysis apparatus (TGA-Cahn TG-171) at 850 °C for three repeated thermal cycles [1].

During the detailed assessment of HDCaSi-N, nine consecutive boards numbered 0013–0021, from a new batch (batch No. 30635), were purchased from a supplier. The dimensions of each of the board were 2570 mm by 1270 mm by 50.8 mm. Board number 0017 was reserved as a backup. The specimens for XRF and thermal expansion tests were prepared from several of the boards and at different locations before any heat treatment of the specimens. The specimens for density and thermal conductivity measurements were machined to 320 mm by 320 mm or 502 mm by 502 mm in lateral dimensions. These specimens were then heat-treated at 850 °C for 24 hours, and the top and bottom surfaces of each specimen were machined flat and parallel to within 0.1 mm. These heat-treated and machined specimens were used for the uniformity assessments in terms of density and thermal conductivity (see Sect. 4).

To verify the consistency of this newly purchased batch of HDCaSi-N for detailed assessment, chemical analysis using XRF was carried out on four specimens: one specimen prepared from board No. 0013, one from board No. 0021 and two specimens from board No. 0016. The two specimens from board No. 0016 were cut from the

Table 1 The oxide composition of four HDCaSi-N specimens from the board No. 0013, 0016 and 0021 of the batch No. 30635 and the HDCaSi-N specimen from the preliminary batch No. 30639 [1]

Result(s)		30635-0013	30635-0016-T	30635-0016-B	30635-0021	30639
Sample basis		Dried 110°C	Dried 110°C	Dried 110°C	Dried 110°C	Dried 110°C
Silicon dioxide	SiO_2	48.67%	48.79%	48.56%	48.30%	48.53%
Titanium dioxide	TiO_2	0.18%	0.18%	0.18%	0.17%	0.18%
Aluminum oxide	Al_2O_3	0.60%	0.60%	0.59%	0.59%	0.62%
Iron (lll) oxide	Fe_2O_3	0.22%	0.22%	0.22%	0.22%	0.22%
Calcium oxide	CaO	44.12%	44.31%	44.20%	44.03%	44.12%
Magnesium oxide	MgO	0.35%	0.34%	0.34%	0.34%	0.33%
Potassium oxide	K_2O	0.28%	0.28%	0.28%	0.27%	0.28%
Sodium oxide	Na_2O	0.36%	0.34%	0.33%	0.31%	0.34%
Phosphorus pentoxide	P_2O_5	0.03%	0.03%	0.03%	0.03%	0.03%
Chromium (lll) oxide	Cr_2O_3	<0.01%	<0.01%	<0.01%	<0.01%	<0.01%
Manganese (ll,lll) oxide	Mn_3O_4	0.04%	0.04%	0.05%	0.04%	0.04%
Zirconium oxide	ZrO_2	0.61%	0.62%	0.60%	0.57%	0.59%
Hafnium (lV) oxide	HfO_2	0.01%	0.01%	0.01%	0.01%	0.01%
Lead oxide	PbO	<0.02%	<0.02%	<0.02%	<0.02%	<0.02%
Zinc oxide	ZnO	0.01%	0.01%	0.01%	0.01%	0.01%
Barium oxide	BaO	0.01%	0.01%	0.01%	0.01%	<0.01%
Strontium (ll) oxide	SrO	<0.01%	<0.01%	<0.01%	<0.01%	<0.01%
Tin (lV) oxide	SnO_2	<0.01%	<0.01%	<0.01%	<0.01%	<0.01%
Copper oxide	CuO	<0.01%	<0.01%	<0.01%	<0.01%	<0.01%
Loss on ignition		4.75%	4.22%	4.38%	4.57%	4.38%
Loss on ignition temperature °C		1025	1025	1025	1025	1025
Total		100.24%	100.00%	99.79%	99.47%	99.68%
Sulfur trioxide	SO_3	0.11%	0.12%	0.11%	0.12%	0.10%
SiO_2/CaO ratio		1.10	1.10	1.10	1.10	1.10

same lateral location, with one of them close to the top surface and another close to the bottom surface.

The XRF elements analysis (Table 1) includes: SiO_2, TiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, K_2O, Na_2O, P_2O_5, Cr_2O_3, Mn_3O_4, ZrO_2, HfO_2, PbO, ZnO, BaO, SrO, SnO_2 and CuO. Please note that SO_3 remaining in the test sample after loss and fusion is also reported, but this is not a total SO_3 figure. A result for loss on ignition is also reported. To determine the loss on ignition, a sample is heated at 1025°C to constant mass and the loss or gain of mass is measured using a thermogravimetric instrument. The comparisons between the four HDCaSi-N specimens from the board No. 0013, 0016 and 0021 of batch No. 30635 and the specimen from the preliminary batch No. 30639 show that the compositions are consistent between the boards from the same

batch and also between the two batches. The ratio of SiO_2/CaO remains the same, at 1.10 for all five specimens shown in Table 1. The stoichiometry ratio for SiO_2/CaO in the stable form of calcium silicate $(CaSiO_3)$ is 1.07. The difference is due to the small amount of glass fiber in the material [1].

3 Thermal Expansion Behavior of HDCaSi-N

To check the mechanical stability under thermal cycling and uniformity of the newly purchased batch of candidate reference material, HDCaSi-N, thermal expansion tests were performed on eighteen specimens prepared from board Nos. 0013, 0016 and 0021. From each board six specimens were prepared from two lateral locations: three of them from the left-hand side and another three of them from the right-hand side of the board. Within each set of three specimens prepared from the same location, one specimen was prepared for thermal expansion test in the through-thickness direction, and two specimens were prepared with axis in plane, one of them is parallel to the sheet/board length and the other is transverse to the sheet/board length.

3.1 Measurement Method

Thermal expansion measurements were taken using a Linseis twin push-rod alumina dilatometer calibrated using platinum and alumina reference materials. Test pieces approximately 30 mm in length and 5–6 mm square were carefully cut manually using a small saw with a fine abrasive blade. In the test apparatus, a small axial force of 150 mN was applied. They were then subjected to three thermal cycles to 850 °C at a heating and cooling rate of $2\,°C \cdot min^{-1}$ with a 1 h hold at peak temperature.

3.2 Thermal Expansion Characterization

The typical thermal expansion curves in three orthogonal directions for the candidate reference material HDCaSi-N (batch No. 30635) are shown in Fig. 1. The fractional length change over the first thermal cycle shows that the material underwent significant shrinkages both in plane (parallel and transverse to sheet length) and through thickness in the first thermal cycle and the latter direction showed greater changes. The sharp change in fractional length at around 700 °C was due to the dehydroxylation of the material. The XRD measurements that had been taken on HDCaSi-N samples in the provisional assessments revealed that one of the major phases, hydrated calcium silicate $(Ca_2SiO_4 \cdot H_2O)$ in fresh samples, became calcium silicate $(CaSiO_3)$ after the specimen had been heat-treated at 850 °C [1]. The fractional length changes recorded for the second and third thermal cycles showed that the HDCaSi-N material was mechanically stable.

   The mean thermal expansion coefficients of the candidate reference material HDCaSi-N (batch No. 30635) at 400 °C are presented in Fig. 2 for all eighteen specimens measured. The variation (standard deviation) of the mean thermal expansion coefficient in the through-thickness direction is 1 % for the six specimens from board
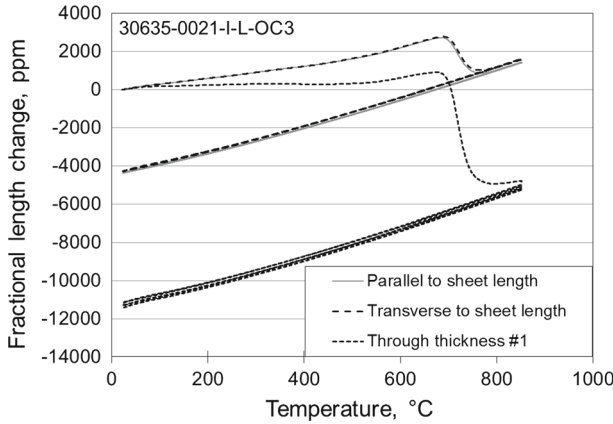
Fig. 1 Typical thermal expansion curves for the HDCaSi-N (batch No. 30635) during the three thermal cycling up to 850 °C
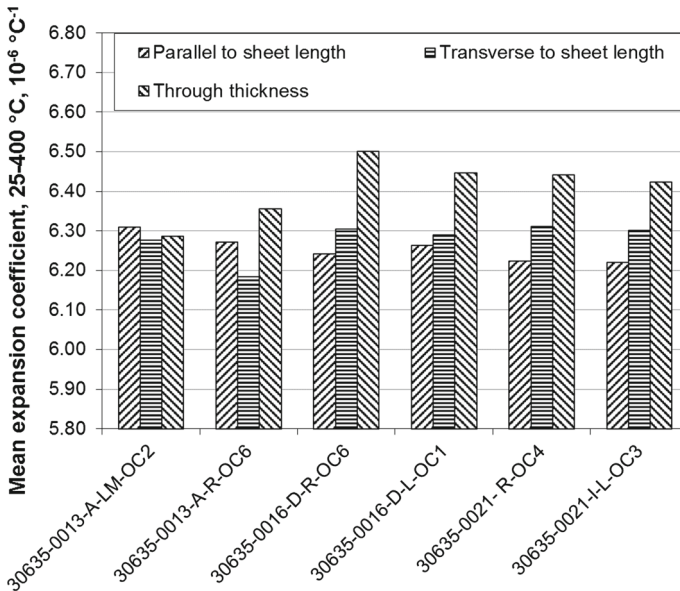

Fig. 2 Mean thermal expansion coefficient at 400 °C for all eighteen HDCaSi-N specimens

Nos. 0013, 006 and 0021. The variation (standard deviation) of the mean thermal expansion coefficient in the in-plane directions (both parallel and transverse to the sheet length) is 0.7 % for twelve out of the eighteen specimens, six in each direction, from the three boards. The results indicate that the material has reproducible thermal expansion behavior, with through-thickness results being significantly greater than in-plane results.

Figure 3 shows the mean thermal expansion coefficients averaged over the six specimens measured from 150 °C to 850 °C for each direction. As the thermal expansion
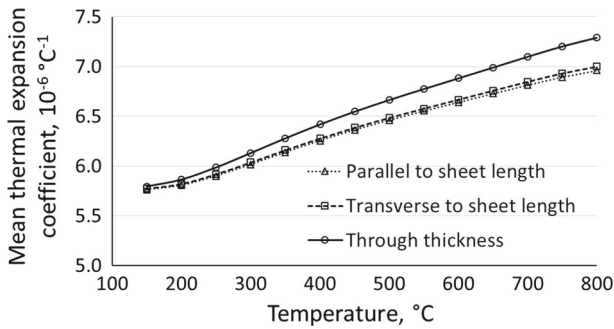
Fig. 3 Mean thermal expansion coefficient of HDCaSi-N (batch No. 30635)

coefficients in the two in-plane directions, parallel and transverse to the sheet length, are the same (well within the uncertainty of 2 %), a single in-plane value for thermal expansion can be used. The values of mean thermal expansion coefficients in Fig. 3 are used in Sect. 5 to estimate the specimen bowing due to differential thermal expansion.

4 Assessments of Uniformity and Short-Term Stability

4.1 The Selection of Reference Specimens HDCaSi-N

The provisional assessment [1] of the candidate thermal conductivity reference material had suggested that the HDCaSi-N remained stable dimensionally, mechanically and chemically after the first heat treatment up to 850 °C for 24 h. Although the flat slab specimens had been found slightly warped after the first heat treatment, after grinding the surfaces of the heat-treated specimens flat and parallel, they stayed flat after being through two following repeated thermal cycling up to 850 °C. The provisional assessment also suggested the need for stringent process and criteria for selecting the reference specimens of HDCaSi-N [1].

In the detailed characterization stage reported in this paper, firstly the HDCaSi-N specimens were heat-treated up to 850 °C for 24 h and then the top and bottom surfaces of each specimen were machined flat and parallel (see Sect. 2), before the characterization of thermal conductivity and density. The following criteria were applied in the selection process:

1. Bulk density variation is within ±1 %;
2. The NDE method [2] indicates that the density variation in a specimen is within ±1 %;
3. The variation of thermal conductivity/resistance values measured at 20 °C is within ±1 %.

First, the heat-treated and machined specimens that met the criteria of bulk density variation within +/-1 % and within the density band (800–816 kg · m^{-3}) were selected for the next stage. This density band was chosen because it has the maximum number of specimens. In the second step, attempts were made using a NDE method to indicate the density variation within each of the specimens selected in the first step. The NDE

method uses a Portable Ultrasonic Non-Destructive Digital Indicating Tester (PUN-DIT), an ultrasonic pulse velocity (U.P.V.) test instrument to measure the time taken by a pulse of ultrasound to pass through the material under that test. Before using the NDE instrument, the limit of the instrument signal variation corresponding to the 1 % density variation of the HDCaSi-N was determined using small sacrificial specimens. For the specimens with nominal lateral dimensions of 500 mm by 500 mm, the NDE tests were carried out at 25 points on each of the specimens, with 100 mm interval between each test point. For the specimens with nominal lateral dimensions of 320 mm by 320 mm, the NDE tests were carried out at 9 points on each of the specimens, with 100 mm interval between each test point. Specimens that met the following criteria were then selected for thermal conductivity measurements at 20 °C:

(1) Those 500 mm square specimen with indicated density variation within ±1% at all 25 points;
(2) Those 500 mm square specimen with indicated density variation within ±1% at the center 9 points (covering the 300 mm by 300 mm central area) and with indicated average density in the surrounding area agreeing with the average density in the center area within +/-1%; and
(3) Those 320 mm square specimens with indicated density variation within ±1% at all 9 points.

Afterward, the selected specimens from the second step were measured at 20 °C mean specimen temperature in a Guarded Heat Flow Meter apparatus at NPL. Only the specimens with thermal conductivity/resistance agreement within ±1 % were selected for further detailed characterization at high temperatures. In total, fifteen HDCaSi-N specimens fulfilled all the criteria and passed the three-step selection process.

4.2 Uniformity in Terms of Thermal Conductivity up to 650 °C

To check the uniformity of the specimens that met the selection criteria above, thermal conductivity measurements were taken on five out of the fifteen specimens using the NPL high-temperature guarded hot plate (HTGHP) at mean specimen temperatures from 150 °C to 650 °C. The five specimens were from board Nos. 0014, 0018, 0019 and 0021, with two of them from the board No. 0014. The NPL HTGHP [3,4] is a single-specimen apparatus with 305 mm diameter heater plates. The measurement uncertainty of the NPL HTGHP is normally ±5 % ($k = 2$).

Figure 4 shows the results of the thermal conductivity measurements of the five HDCaSi-N specimens as relative difference to the mean thermal conductivity values. The thermal conductivity values of each specimen differed by less than 2 % from the average values of the five specimens measured from 150 °C to 650 °C. As the 2 % difference is less than half of the measurement uncertainty of the NPL HTGHP, these specimens are considered to be identical by the NPL HTGHP; in other words, they are consistent in terms of thermal conductivity up to 650 °C. Although the bulk density of the specimens varied from 800 kg · m^{-3} to 813 kg · m^{-3}, no thermal conductivity dependence on density was detected using the NPL HTGHP. The results indicate that the criteria used in the selection process were sufficient to ensure uniformity across the candidate reference specimens.
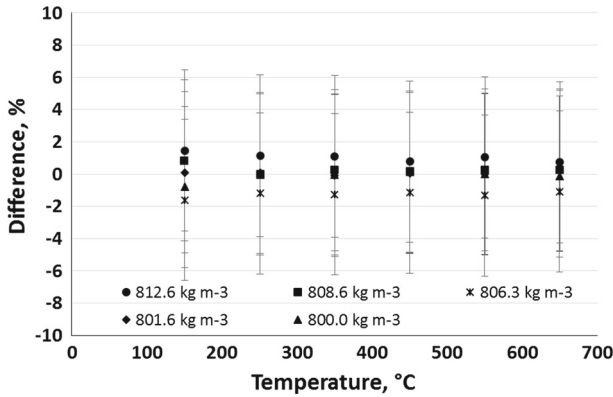
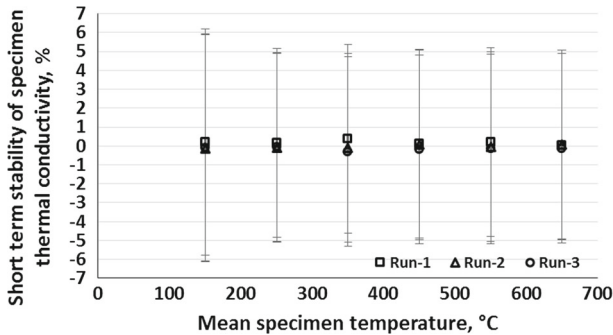**Fig. 4** Check of uniformity in terms of thermal conductivity up to 650 °C

Fig. 5 Short-term stability of the candidate reference material HDCaSi-N in terms of thermal conductivity

4.3 Stability in Terms of Thermal Conductivity up to 650 °C

The short-term stability of the candidate reference material HDCaSi-N was assessed up to 650 °C using the NPL HTGHP. The thermal conductivity of a specimen machined from the board No. 21 of the batch numbered 306035, one of the fifteen specimens selected, was measured in three independent runs over two months. In an independent thermal conductivity measurement, the test specimen was installed in the NPL HTGHP, then the thermal conductivity was measured at mean specimen temperatures up to 650 °C, afterward the apparatus was cooled down at 1 °C/min rate to room temperature, and then the specimen was taken out of the apparatus for storing at 23 °C and 50 % relative humidity environment until next measurement. It is worth mentioning that as the material absorbs moisture, during transportation or storage it shall not be exposed to temperatures below 0 °C to avoid damages from freeze-thaw and shall be kept in a sealed container and/or dry environment. The assessment of the short-term stability of the candidate reference material HDCaSi-N is shown in Fig. 5. The error bars are the measurement uncertainty of the NPL HTGHP. The difference of the measured thermal conductivity between three independent runs was well within 1 %, showing the material is stable and usable in repeated regime.

5 Discussion

When a flat slab specimen of the candidate reference material HDCaSi-N is used in high-temperature guarded hot plates, there will be a temperature gradient, 30 K–70 K, established through the thickness of the specimen. Due to the temperature difference between the cold surface and the hot surface of the specimen, these surfaces expand different amounts—the cold surface expands less than the hot surface. As a result, the specimen bows toward the cold side.

The bowing of specimen could lead to a small air gap forming between the contacting surfaces of the specimen and the plate and therefore could increase the thermal contact resistance at the interfaces and cause the measured thermal conductivity value deviate (less than) from the true value. In addition, the increase of the bowing of specimen could increase the distortion of the one-dimensional uniform heat flux that need to be established through the thickness of the specimen and could cause measurement errors, even invalid measurements in some extreme cases. Therefore, it is essential to understand the key factors that affect the specimen bowing before the measures to minimize it could be proposed.

The estimation of the amount of free bowing has been carried out in the EMRP 'Thermo' project using thermal stress analysis [5]. The compressive load applied on the test specimen in high-temperature guarded hot plates is assumed negligible, and the specimen is assumed to expand freely. The temperature through the thickness of the specimen $2h$ at T_m is assumed to be linear, and the hot surface and the cold surface are at $z = h$ and $z = -h$.

$$T = a + bz = T_\mathrm{m} + \frac{\mathrm{d}T}{\mathrm{d}z}z \tag{1}$$

where $a = T_\mathrm{m}$ is the mean specimen temperature; $b = \frac{\mathrm{d}T}{\mathrm{d}z}$ is the linear temperature gradient through the thickness of the specimen.

Substituting Eq. (1) into equation (9.5.5) in Ref. [5], $N_T = \alpha E \int\limits_{-h}^{h} T \mathrm{d}z; \quad M_T = \alpha E \int\limits_{-h}^{h} T z \mathrm{d}z$ (9.5.5) [5]

$$N_T = 2\alpha E a h; \quad M_T = \frac{2}{3}\alpha E b h^3 \tag{2}$$

where E is Young's modulus; α is the mean thermal expansion coefficient in in-plane direction.

Substituting Eq. (2) into equation (9.5.6c) in Ref. [5] for the displacement component w for z direction, $w = -\frac{3M_T}{4h^3 E}(x^2 + y^2) + \frac{1}{(1-\nu)E}\left\{(1+\nu)\alpha E \int\limits_{0}^{z} T \mathrm{d}z - \frac{\nu z}{h}N_T \right.$

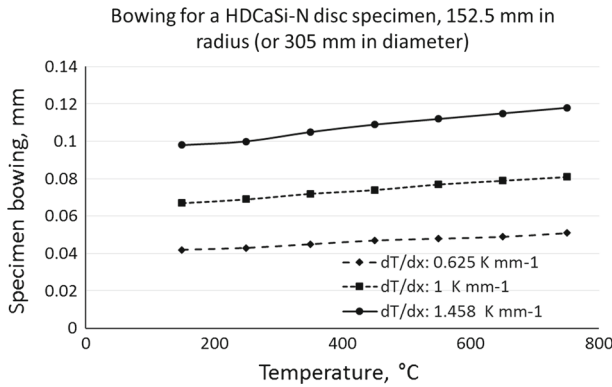$\left. -\frac{3\nu z^2}{2h^3}M_T \right\}$ (9.5.6c) [5]

Fig. 6 Specimen bowing versus temperature gradient

where ν is Poisson's ratio. After simplification,

$$w = \frac{-\alpha b}{2}(x^2 + y^2) + \alpha a z + \frac{\alpha}{2} b z^2 \tag{3}$$

For specimens in the shape of round disk, $x^2 + y^2 = r^2$ where r is the radius, then Eq. (3) becomes

$$w = \frac{-\alpha b}{2} r^2 + \alpha a z + \frac{\alpha}{2} b z^2$$

The estimated bowing, Δw, of the specimen can be calculated from subtracting the displacement at center $r = 0$, from the displacement at the radius of the round disk, $r = R$

$$\Delta w = -\frac{\alpha b}{2} R^2 \tag{4}$$

Equation (4) suggests that the thermal expansion of the material, temperature gradient through the thickness of the specimen and specimen radius (or diameter) affect the amount of bowing. The thermal expansion of the HDCaSi-N was characterized and is reported in Sect. 3. In current section, Eq. (4) was used to estimate the amounts of bowing of HDCaSi-N specimens with different lateral dimensions and different temperature gradient through the thickness of the specimen when used in HTGHPs. The results are shown in Figs. 6 and 7.

Figure 6 is the estimated bowing of an HDCaSi-N specimen that is 305 mm in diameter, under different temperature gradient. When the temperature gradient through the thickness of the specimen increases from 0.625 K mm^{-1} (e.g., 30 K over a 48 mm thick specimen) to 1.458 K mm^{-1} (e.g., 70 K over a 48 mm specimen), it is estimated that the amount of specimen bowing is more than doubled. The bowing of the specimen increases slightly with the increase of the mean specimen temperature. The values of 30 K and 70 K are the limits of the temperature difference between the hot and cold faces specified in EN/TS 15548-1 in order that the assumption of linear temperature gradient through the thickness of the specimen remains valid and also
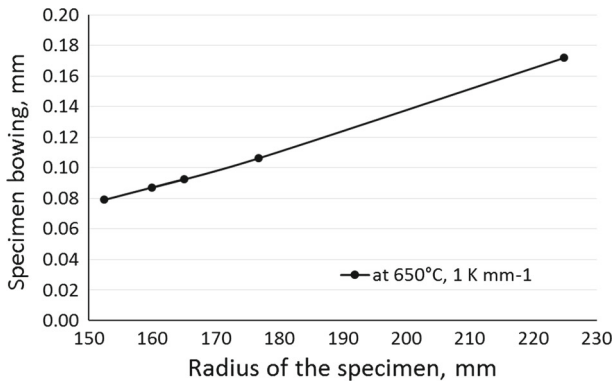
Fig. 7 Specimen bowing versus radius of specimen (650 °C)

for rigid specimen to limit the amount of the specimen bowing and its effect on the thermal conductivity measurements.

Figure 7 is the estimated effect of the lateral dimension of the HDCaSi-N specimen on the amount of bowing. At the mean specimen temperature 650 °C and temperature gradient of 1 K mm^{-1} through the thickness of the specimen, the amount of bowing is more than doubled when the radius of the specimen increases from 152.5 mm to 225 mm.

The results in Figs. 6 and 7 show that the effect of specimen bowing on thermal conductivity measurements can be minimized by using a smaller temperature gradient through the thickness of the specimen and reducing the specimen diameter. However, the temperature gradient shall not be too small to cause a significant increase in the measurement uncertainty of temperature difference between the hot and cold faces of the specimen. In addition, the thermal contact resistance at the interfaces could be minimized by using contact media and applying high compressive load. However, for rigid HDCaSi-N specimens it is recommended to minimize the bowing of specimens, because using high compressive load to flatten the specimens with large amount of bowing can cause the fracture of specimens.

Therefore, it is recommended that the HDCaSi-N specimen is used with smaller lateral dimensions and a smaller temperature gradient through the thickness of the specimen to minimize the amount of specimen bowing. For example, for a HTGHP with 500 mm by 500 mm plates, it is recommended to surround a smaller central specimen (larger than the metering area) with separate small pieces of the same material.

6 Conclusions

The XRF analysis on the candidate reference material HDCaSi-N has shown that the compositions are consistent between the boards from the same batch and also between the two batches. The ratio of SiO_2/CaO is the same within analysis uncertainty for all five specimens shown in Table 1. The characterization of the thermal expansion behavior of the material indicates that the material has reproducible thermal expan-

sion, with through-thickness results being significantly greater than in-plane results. It is mechanically stable after the first thermal cycle up to 850 °C. The uniformity assessment results have shown that the selected reference specimens are uniform in terms of thermal conductivity up to 650 °C, and the criteria used in the selection process are sufficient to ensure the uniformity across the candidate reference specimens. The assessment of short-term stability has shown that material is stable in terms of thermal conductivity up to 650 °C and is usable in repeated regime. Based on the estimated bowing of specimen due to the effect of differential thermal expansion caused by the temperature difference across the specimen, it is recommended that smaller lateral dimension specimens are used, with a smaller temperature drop through the thickness of the specimen, or between the hot and cold plate to minimize the amount of specimen bowing. In summary, the selected reference specimens are reproducible, mechanically stable and uniform and stable in terms of thermal conductivity up to 650 °C. However, it is worth mentioning that during transportation or storage this material shall not be exposed to temperatures below 0 °C to avoid damages from freeze-thaw and shall be kept in a sealed container and/or dry environment.

7 Future Work

The long-term stability of the candidate reference material still needs to be monitored. The 15 selected HDCaSi-N specimens have now been used in inter-comparisons between European reference laboratories. The details of the inter-comparisons and the results will be reported in a publication in the future.

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