



Data Article

Experimental data showing the influence of different boron nitride particles on the silica network, the butyl stearate and the porogens in shape-stabilized phase change materials



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ABSTRACT

Shape-stabilized phase change materials (ss-PCMs) based on silica and butyl stearate were thermally enhanced via the addition of different hexagonal boron nitride particles (BN) to the *in situ* sol-gel synthesis. The dataset is used in conjunction with the experimental data of the influence of the particle size and surface area of BN on the thermal and mechanical properties of ss-PCMs discussed in Marske et al. (2021). To study the effect of the different BN particles on the hydrolysis degree of the silica network and on the chemical nature of the porogens sodium dodecyl sulfate and poly(vinyl alcohol) used for the ss-PCM synthesis, the ss-PCM samples are measured via High Power Decoupling (HPDEC) Magic Angle Spinning (MAS) ²⁹Si NMR and attenuated total reflection-Fourier transform infrared (ATR-FTIR) spectroscopy, respectively. Additionally, data of the influence of BN on the thermal properties is presented as thermogravimetric analysis

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(TG). The ^{29}Si MAS NMR spectra are referenced to tetramethylsilane and show the different silica species in ppm. The different value of wavenumber and intensity of each reference and ss-PCM sample is listed in the IR spectra. The decomposition points of the ss-PCMs are calculated from the TG data via OriginLab. The spectra and data can be used as a reference for other researchers and engineers to use in synthesizing ss-PCMs based on silica and other polymeric materials or as reference for pure BN, SDS, stabilized silica sol and PVA.

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Specifications Table

Subject	Thermal energy storage
Specific subject area	Shape-stabilized phase change materials, hexagonal boron nitride, thermal conductivity enhancement
Type of data	Figure, Table
How data were acquired	High Power Decoupling (HPDEC) Magic Angle Spinning (MAS) ^{29}Si NMR using a Bruker DRX-400 WB NMR spectrometer (Bruker Biospin, Karlsruhe, Germany) equipped with a 4 mm double-resonance MAS probe maintained at $-10\text{ }^{\circ}\text{C}$ by a temperature control unit. Attenuated total reflection-Fourier transform infrared (ATR-FTIR) spectroscopy using a Tensor 27 spectroscope equipped with a diamond ATR unit from Bruker. Thermogravimetry (TG) using a STA 409 C/CD thermal analyzer from Netzsch.
Data format	Raw, Analyzed
Parameters for data collection	ss-PCM samples were synthesized with silica, sodium dodecyl sulfate, poly(vinyl alcohol), butyl stearate, water, and BN of type HeBoFill with different particle sizes and specific surface areas via a specific sol-gel process
Description of data collection	For ^{29}Si MAS NMR, the ss-PCM sample scanning was carried at a radio-frequency field strength of 42 kHz as a $\pi/2$ pulse during an acquisition time of 25 ms (Larmor frequency of 79.49 MHz, spinning speed of 10 kHz, repetition delay of 40 s, number of scans between 2000 and 3500, and using tetramethylsilane as s reference). The ss-PCMs were scanned 60-times via ATR-FTIR instrument as pure substance at the range of $4000\text{--}400\text{ cm}^{-1}$ wavenumbers. An amount of 100 mg ss-PCM sample was heated from 25 to $400\text{ }^{\circ}\text{C}$ in thermogravimetric analysis (heating rate of $1\text{ }^{\circ}\text{C}/\text{min}$).
Data source location	Institution: Martin Luther University of Halle-Wittenberg City/Town/Region: Halle (Saale) / Saxony-Anhalt Country: Germany
Data accessibility	With the article
Related research article	F. Marske, T. Lindenberg, J. Martins de Souza e Silva, R. B. Wehrspohn, A. W. Maijenburg, T. Hahn and D. Enke, Size and surface effects of hexagonal boron nitrides on the physicochemical properties of monolithic phase change materials synthesized via sol-gel route, Appl. Therm. Eng. 196 (2021), 117325.

Value of the Data

- It is of great interest to find the boron nitride best suited to increase the mechanical stability, thermal conductivity, and durability of polymers, such as ss-PCMs, to the highest extent.
- The researcher can use the data to compare or develop ss-PCMs based on silica and boron nitride with higher decomposition points of PCMs and, thus, broaden the application range of different polymers.

- The data help to understand the relationship between boron nitride with different particle sizes and specific surface areas in sol-gel processes.
- The data of the pure substances before and after immobilization in the silica network enables a proper comparison with similar materials, such as graphite, carbon nanotubes and boron nitride nanosheets.

1. Data Description

The dataset is based on the analysis of ss-PCM samples synthesized with [1] and without BN particles [2]. The ss-PCM samples were measured via High Power Decoupling (HPDEC) Magic Angle Spinning (MAS) ^{29}Si NMR, Attenuated total reflection-Fourier transform infrared (ATR-FTIR) spectroscopy and thermogravimetry (TG). The thermally enhanced ss-PCM samples were synthesized with six types of hexagonal boron nitride platelets (BN). A total of four different amounts of BN of each BN type (5–20 wt%) was used for the ss-PCM synthesis, resulting in a total of 24 different ss-PCM samples. The particle size, particle thickness and specific surface area of each BN type is listed in our recent publication [1]. The raw data of all figures is given in the supplementary data of this paper.

Because a well-interconnected silica network is a key property of mechanical stable ss-PCMs, we analyzed the influence of BN with different particle sizes and specific surface areas on the hydrolysis degree of the pure silica sol and the silica network in our ss-PCM samples via ^{29}Si MAS NMR in Fig. 1. The ^{29}Si MAS NMR spectra of the stabilized silica sol show three peaks in Fig. 1 (A). In Fig. 1 (B–H), the ^{29}Si MAS NMR spectra of the ss-PCMs BN0 (B), BN4501_20 (C), BN2503_20 (D), BN1207_20 (E), BN0312_20 (F), BN0315_20 (G) and BN0320_20 (H) show three partially overlapping peaks with a decreased peak intensity of peak 2 in comparison to Fig. 1 (A).

Physical and chemical interactions between the surfactant sodium dodecyl sulfate (SDS), the co-polymer poly(vinyl alcohol) (PVA), silica, butyl stearate and BN were analyzed via attenuated total reflection-Fourier transform infrared (ATR-FTIR) spectroscopy. Fig. 2 shows the ATR-IR spectra of the educts stabilized silica sol (A), butyl stearate (B), SDS (C) and PVA (D). Fig. 3 shows the combination of six ATR-IR spectra of the pure BN particles of the type HeBoFill501 (BN4501), HeBoFill 491 (BN2503), HeBoFill64 (BN1207), HeBoFill111 (BN0312), HeBoFill230 (BN0315) and HeBoFill205 (BN0320). Fig. 4 shows the sample row BN2503 (A1) and BN0312 (B1) with four different BN amounts used in the ss-PCM synthesis after drying at 100°C for 24 h (A1, B1) and calcination at 600°C for 6 h (A2, B2). Fig. 5 shows a combination of the ATR-IR spectra of all ss-PCM samples containing 20 wt% BN after drying at 100 °C for 24 h (A1) and calcination at 600 °C for 6 h (A2).

The effect of the particle size and specific surface area of BN on the decomposition points of the ss-PCMs was analyzed via thermogravimetry (TG). The TG curves of the sample rows BN4501 (A), BN2503 (B), BN1207 (C), BN0312 (D), BN0315 (E) and BN0320 (F) are shown in Figs. 6–8. For the determination of the decomposition points, the maximum of the first derivative of the TG curves (Figs. 6–8) was calculated via OriginLab 8.1. All decomposition points in dependence of the BN type and the BN mass fraction are listed in Table 1 and shown in Fig. 9.

2. Experimental Design, Materials and Methods

2.1. Materials

The stabilized silica sol of type Köstrosol 0730 is a suspension of 30 wt% colloidal silica particles with a 7 nm average particle diameter in water, and was obtained by Chemiewerk Bad Köstritz, Germany. Butyl stearate (BS) is a technical grade product (40–60%) and was purchased by Alfa Aesar. The co-polymer poly(vinyl alcohol) (PVA) has an average molecular weight of 22,000 and is hydrolyzed by 88% (PVA, 88 % hydrolyzed, average M.W. 22000). PVA was

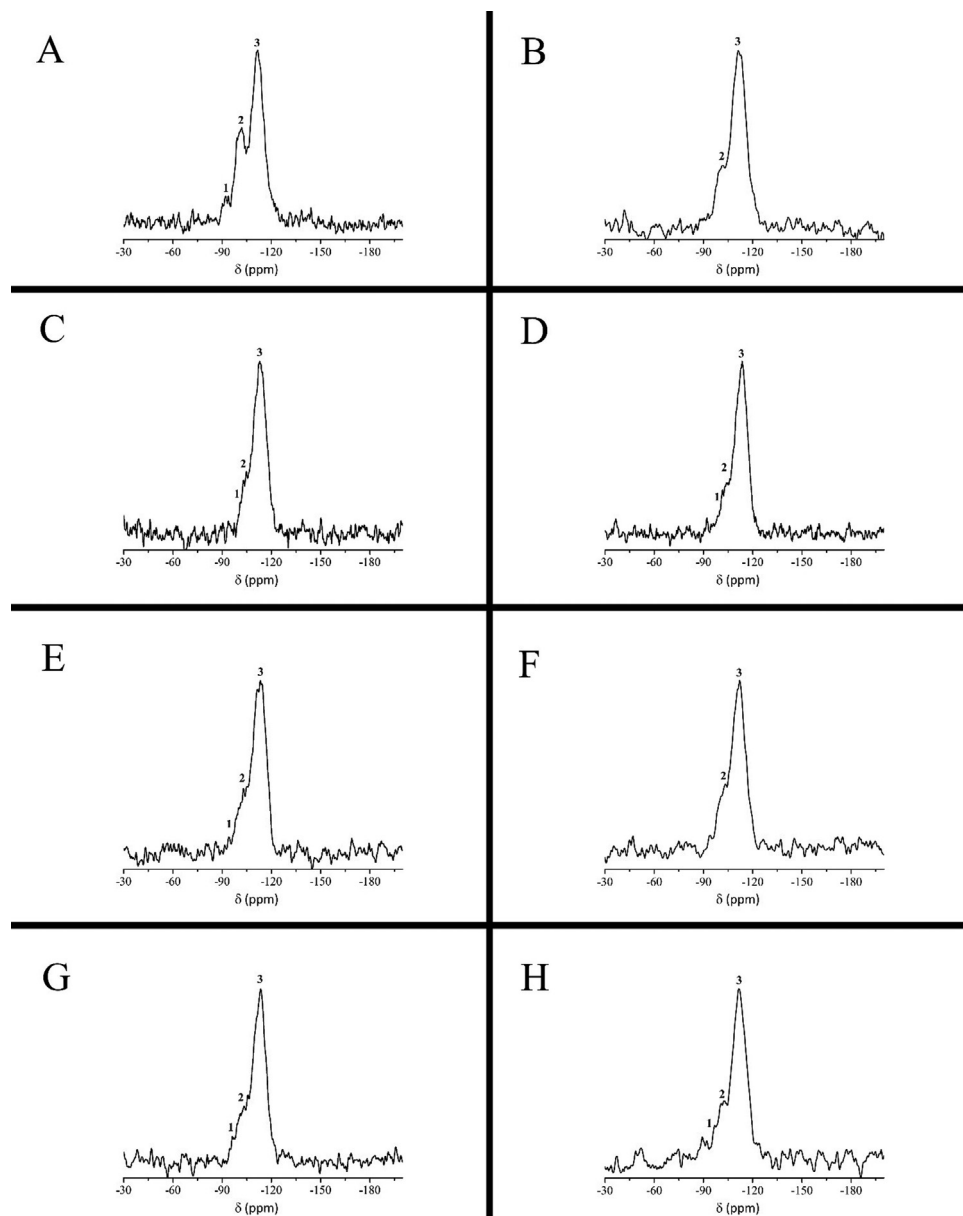


Fig. 1. ^{29}Si MAS NMR spectra of stabilized silica sol (A), ss-PCM BNO (B) synthesized without BN and ss-PCM samples BN4501_20 (C), BN2503_20 (D), BN1207_20 (E), BN0312_20 (F), BN0315_20 (G) and BN0320_20 (H). 1 = $(\text{Si-O})_2\text{Si}(\text{OH})_2$ (Q^2); 2 = $(\text{Si-O})_2\text{Si}(\text{O-Si})(\text{OH})$ (Q^3); 3 = $(\text{Si-O})_2\text{Si}(\text{O-Si})_2$ (Q^4).

purchased by ACROS Organics. The surfactant sodium dodecyl sulfate (SDS) was purchased as dust-free pellets with a purity over 99% (GC) by Sigma-Aldrich. The different hexagonal boron nitride platelets (BN) of HeBoFill type (510, 490, 641, 110, 230, 205) were obtained by Henze Boron Nitride Products AG, Germany. The BN of HeBoFill type have the following particle sizes and specific surface areas: 45 μm , 1 m^2/g (HeBoFill 510); 25 μm , 3 m^2/g (HeBoFill 490); 12 μm ,

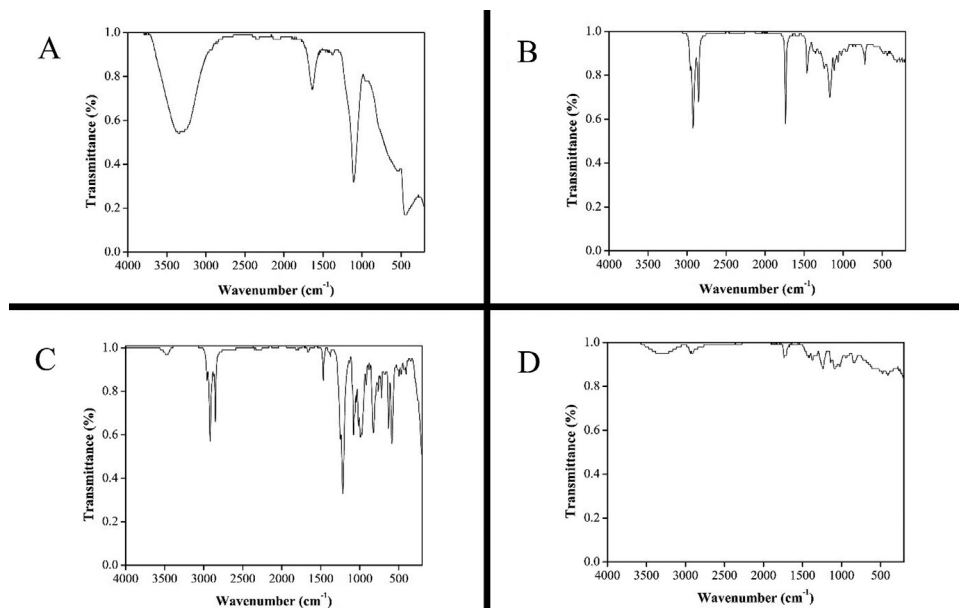


Fig. 2. ATR-IR spectra of educts silica sol (A), butyl stearate (B), sodium dodecyl sulfate (C) and poly(vinyl alcohol) (D). ATR-IR peaks: (A) 3700–2900, 1647, 1110, 456 cm^{-1} ; (B) 2922, 2854, 1738, 1465, 1174, 721 cm^{-1} ; (C) 2958, 2917, 2849, 1468, 1218, 1081, 1017, 994, 9973, 828, 633, 591 cm^{-1} ; (D) 3350–3000, 2942, 1741, 1424, 1244, 1096, 853 cm^{-1} .

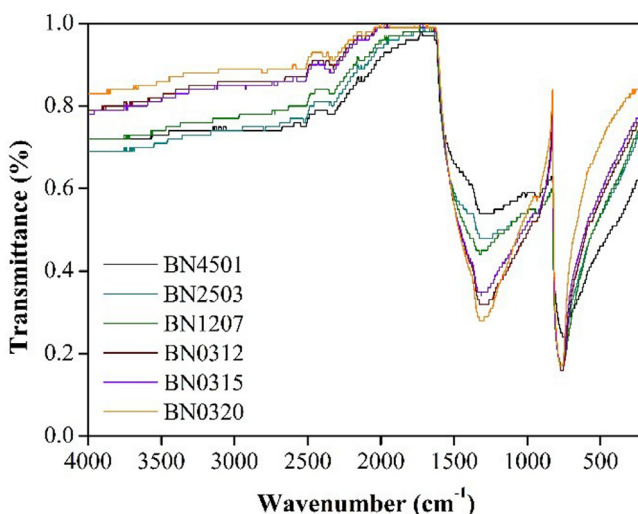


Fig. 3. ATR-IR spectra of pure hBN HeBoFill501 (BN4501), HeBoFill 491 (BN2503), HeBoFill64 (BN1207), HeBoFill111 (BN0312), HeBoFill230 (BN0315) and HeBoFill205 (BN0320). ATR-IR peaks of BN2503: 1316, 770 cm^{-1} .

7 m^2/g (HeBoFill 641); 3 μm , 12 m^2/g (HeBoFill 110); 3 μm , 15 m^2/g (HeBoFill 230); 3 μm , 20 m^2/g (HeBoFill 205). Tetramethylsilane was purchased by Sigma-Aldrich. Deionized water was in our study.

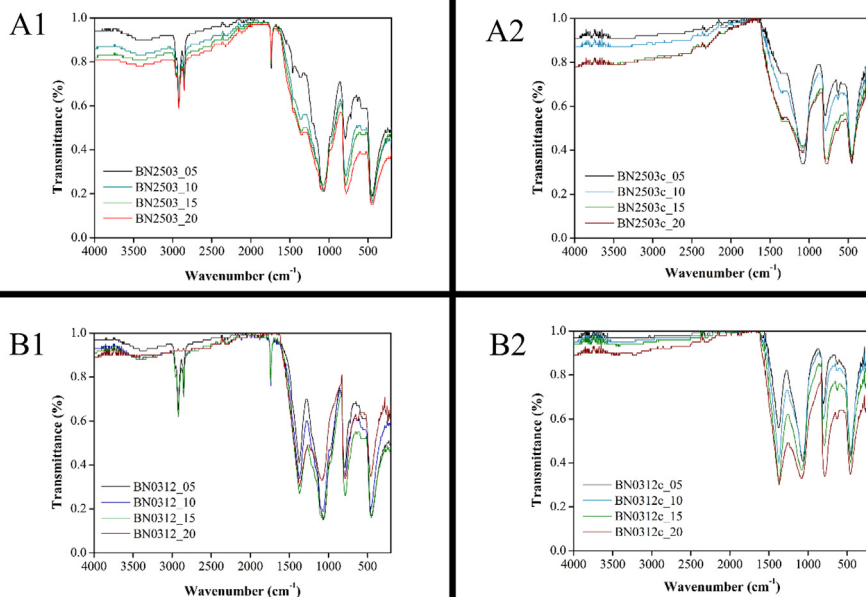


Fig. 4. ATR-IR spectra of ss-PCMs BN2503 (A1), BN2503c (A2), BN0312 (B1) and BN0312c (B2). The ss-PCMs marked with a “c” are calcined at 600 °C for 6 h and represent the pure silica network with boron nitride in the ss-PCM. ATR-IR peaks: (A1, BN2503_20) 2924, 2854, 1383, 1071, 789, 461 cm^{-1} ; (A2, BN2503c_20) 1381, 1098, 790, 464 cm^{-1} ; (B1, BN0312_20) 2923, 2854, 1740, 1378, 1092, 792, 457 cm^{-1} ; (B2, BN0312c_20) 1370, 1095, 793, 462 cm^{-1} .

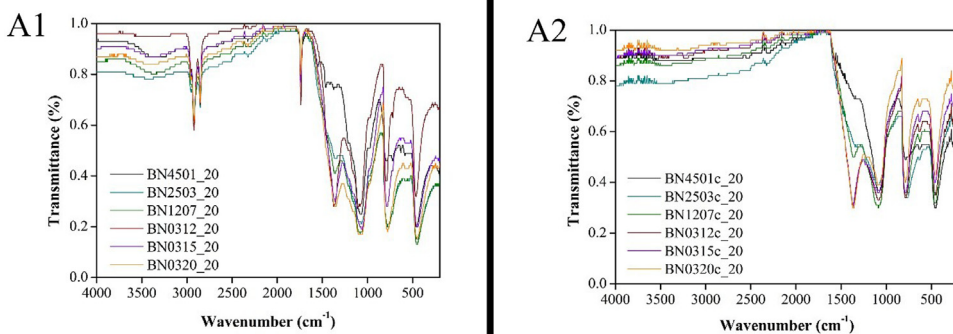


Fig. 5. ATR-IR spectra of all samples from BN4501_20 to BN0320_20 (A1) and all calcined samples from BN4501c_20 to BN0320c_20 (A2). The ss-PCMs marked with a “c” are calcined at 600 °C for 6 h and represent the pure silica network with boron nitride in the ss-PCM. ATR-IR peaks: (A1, BN2503_20) 2924, 2854, 1383, 1071, 789, 461 cm^{-1} ; (A2, BN2503c_20) 1381, 1098, 790, 464 cm^{-1} .

2.2. Methods

High Power Decoupling (HPDEC) Magic Angle Spinning (MAS) ^{29}Si NMR was analyzed on a Bruker DRX-400 WB NMR spectrometer by Bruker Biospin (Karlsruhe, Germany). The spectrometer was equipped with a 4 mm double-resonance MAS probe maintained at -10 °C by a temperature control unit. The Larmor frequency was set to 79.49 MHz and the spinning speed was set to 10 kHz. It was worked with a repetition delay of 40 s. A total of 2000 to 3500 scans

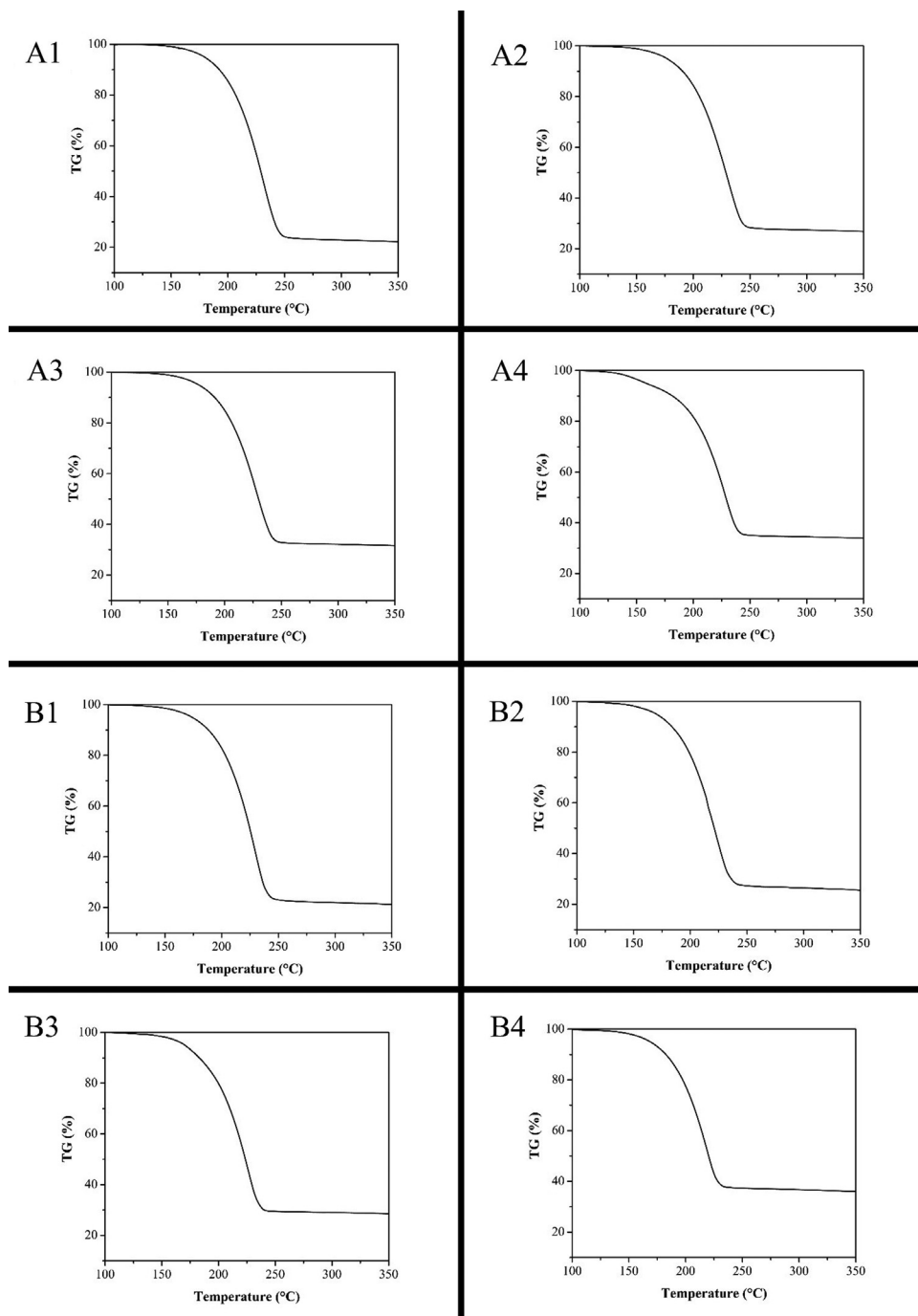


Fig. 6. TG curves of ss-PCMs BN04501_05 (A1), BN4501_10 (A2), BN4501_15 (A3), BN4501_20 (A4), BN2503_05 (B1), BN2503_10 (B2), BN2503_15 (B3) and BN2503_20 (B4).

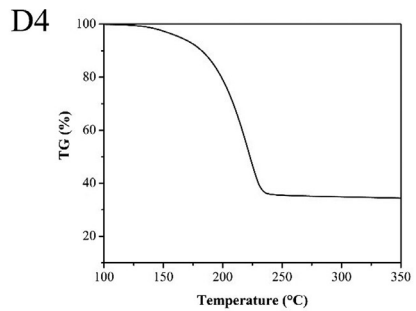
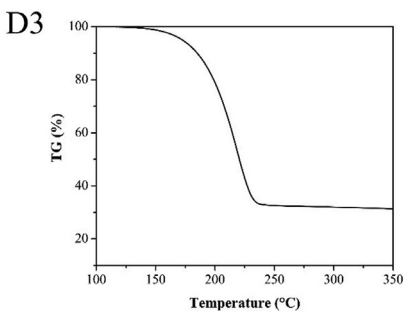
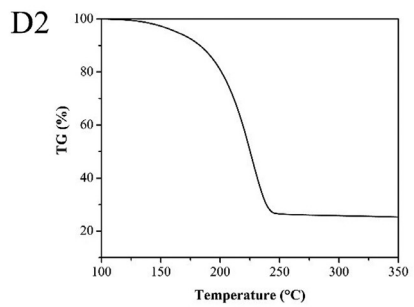
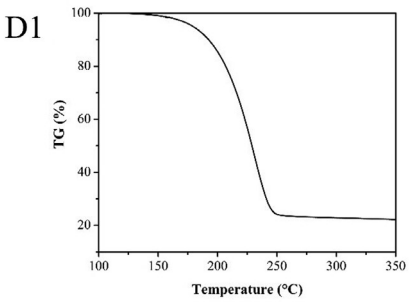
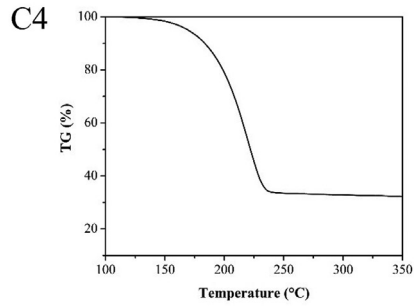
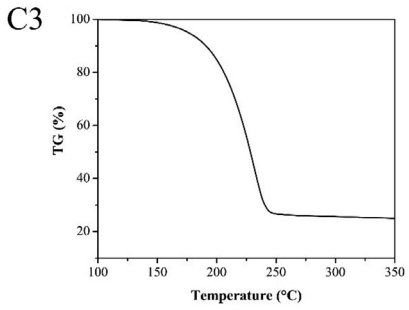
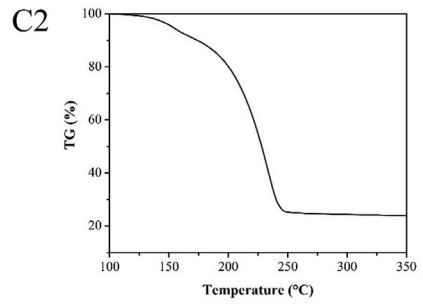
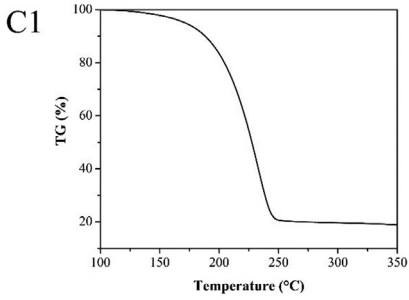


Fig. 7. TG curves of ss-PCMs BN1207_05 (C1), BN1207_10 (C2), BN1207_15 (C3), BN1207_20 (C4), BN0312_05 (D1), BN0312_10 (D2), BN0312_15 (D3) and BN0312_20 (D4).

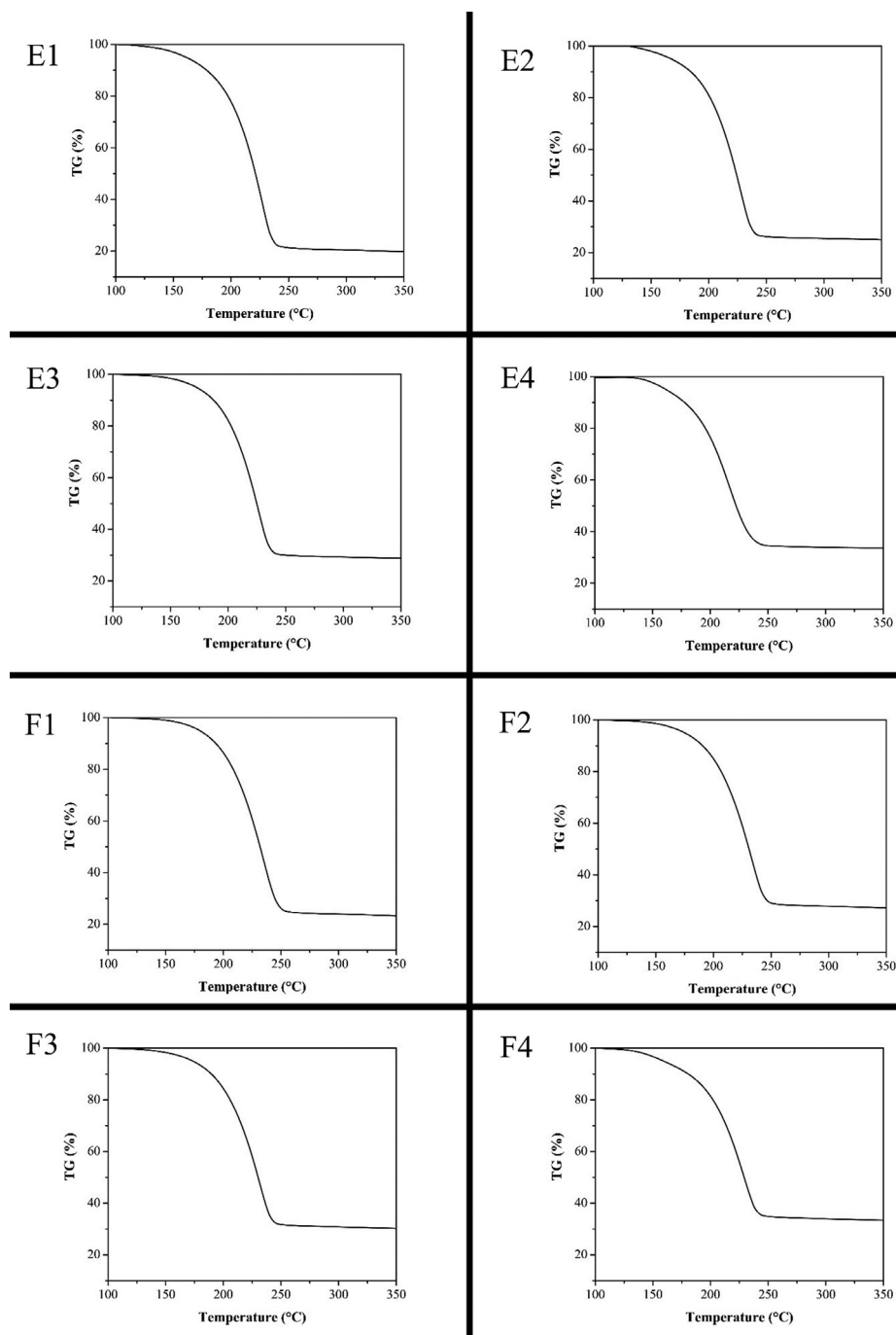


Fig. 8. TG curves of ss-PCMs BN0315_05 (E1), BN0315_10 (E2), BN0315_15 (E3), BN0315_20 (E4), BN0320_05 (F1), BN0320_10 (F2), BN0320_15 (F3) and BN0320_20 (F4).

Table 1

Decomposition points of pure BS, sample BN0 and ss-PCMs BN4501–BN0320 measured via TG. TG data was processed via OriginLab 8.1 to calculate the decomposition points. Data of the decomposition points of BS, BN0, ss-PCM sample row BN2503 and ss-PCMs of the sample row BN4501–BN0320 containing 10 wt% and 20 wt% BN are taken from our related research article [1].

Sample	T _G (°C)
BS	218
BN0	216
BN4501_05	233
BN4501_10	230
BN4501_15	228
BN4501_20	226
BN2503_05	228
BN2503_10	226
BN2503_15	223
BN2503_20	220
BN1207_05	233
BN1207_10	232
BN1207_15	229
BN1207_20	221
BN0312_05	231
BN0312_10	225
BN0312_15	220
BN0312_20	219
BN0315_05	232
BN0315_10	228
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BN0315_20	225
BN0320_05	235
BN0320_10	233
BN0320_15	231
BN0320_20	228

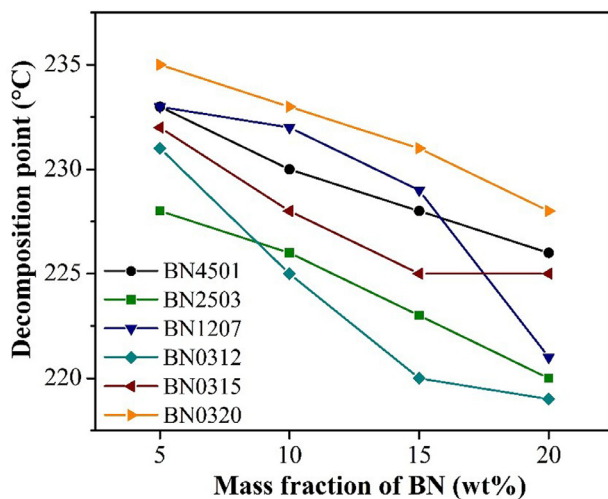


Fig. 9. Decomposition points of ss-PCM sample row BN4501, BN2503, BN1207, BN0312, BN0315 and BN0320 in dependence of the BN mass fraction. Data of the decomposition points of ss-PCM sample row BN2503 and ss-PCMs of the sample row BN4501–BN0320 containing 10 wt% and 20 wt% BN are taken from our related research article [1].

were performed for each sample. Tetramethylsilane (TMS) was used as external reference and the ss-PCM sample scanning was carried at a radio-frequency field strength of 42 kHz as a $\pi/2$ pulse during an acquisition time of 25 ms. The liquid stabilized sol was measured via ^{29}Si NMR without MAS at 20 °C and the measurement settings were changed as followed: repetition delay of 15 s, number of scans of 12,000, acquisition time during a $\pi/2$ pulse of 30 μs .

Attenuated total reflection infrared spectroscopy (ATR-IR) was carried out via a Tensor 27 spectroscope equipped with a diamond ATR unit from Bruker at room temperature (25 °C). The samples were put directly on the diamond ATR unit and were scanned for a total of 60-times. The peaks of the signals were determined via the peak analyzer option in OriginLab 8.1.

The thermal stability was analyzed via thermogravimetry (TG) using a STA 409 C/CD thermal analyzer from Netzsch. An average amount of 100 mg of every ss-PCM sample was measured and heated from 25 °C to 400 °C at a heating rate of 1 °C/min. The raw data was exported and processed via OriginLab 8.1. For the calculation of the decomposition points, the first derivative of the TG curve was calculated and the value of the maximum of the first derivative curve was determined.

Ethics Statement

The authors have both collected the presented data and written this article according to generally standards of ethical behavior in scientific publishing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships which have or could be perceived to have influenced the work reported in this article.

CRedit Author Statement

Felix Marske: Conceptualization, Methodology, Validation, Formal analysis, Investigation, Data curation, Writing – original draft, Visualization; **Titus Lindenberg:** Formal analysis, Resources; **Juliana Martins de Souza e Silva:** Formal analysis, Resources, Writing – review & editing; **Ralf B. Wehrspohn:** Resources, Writing – review & editing; **A. Wouter Maijenburg:** Writing – review & editing, Resources; **Thomas Hahn:** Supervision, Project administration, Funding acquisition; **Dirk Enke:** Writing – review & editing, Supervision.

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Supplementary Materials

Supplementary material associated with this article can be found in the online version at doi:[10.1016/j.dib.2021.107428](https://doi.org/10.1016/j.dib.2021.107428).

References

- [1] F. Marske, T. Lindenberg, J. Martins de Souza e Silva, R.B. Wehrspohn, A.W. Maijenburg, T. Hahn, D. Enke, Size and surface effects of hexagonal boron nitrides on the physicochemical properties of monolithic phase change materials synthesized via sol–gel route, *Appl. Therm. Eng.* 196 (2021) 117325, doi:[10.1016/j.applthermaleng.2021.117325](https://doi.org/10.1016/j.applthermaleng.2021.117325).
- [2] F. Marske, J. Martins de Souza e Silva, R.B. Wehrspohn, T. Hahn, D. Enke, Synthesis of monolithic shape-stabilized phase change materials with high mechanical stability via a porogen-assisted *in situ* sol–gel process, *RSC Adv.* 10 (2020) 3072–3083, doi:[10.1039/c9ra10631f](https://doi.org/10.1039/c9ra10631f).