Thermal expansion and long term stability of ceramics NEXCERA studied by absolute length measurements using multiple wavelengths imaging interferometry

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Abstract

NEXCERA has been developed by Krosaki Harima Corporation as a low thermal expansion cordierite ceramic for commercial use. In order to study the response of the material's dimension to temperature changes three 200 mm gauge block shaped NEXCERA samples have been investigated by precise absolute length measurements using multiple wavelengths imaging interferometry under ambient conditions. The applied temperature cycle involves a series of stable temperatures from 18°C to 25°C followed by temperature steps. As a result of this study length relaxation and hysteresis can be concluded to be absent on the level of the measurement uncertainty (2.5 nm). Accordingly, the length of samples can be clearly assigned to the sample temperature allowing the definite extraction of the CTE. In each case of NEXCERA samples, in the range from 20°C to 22°C the CTE is well within $\pm 10^{-8}$ K^{-1} at an uncertainty level of less than 3×10^{-9} K^{-1}.

1 Introduction

Materials with an ultra low coefficient of thermal expansion (CTE) become more and more important in many industrial applications. E.g., information of the CTE is required for accurate mechanical measurements and increasing demands in manufacturing processes require a better knowledge of the CTE. A very important aspect is also the dimensional stability of materials (e.g. see [1]). Using high accuracy absolute length measurements these effects can be studied at the same time.

2 Design of NEXERA material and sample

The materials used in this study were cordierite, $Mg_2Al_4Si_5O_{18}$ with a few kinds of sintering additives. The mixed powders were wet-milled together with organic binder and water. The resulting slurry was dried to granulated powder, and then isostatically pressed into chalk-like blocks. The compressed blocks were once annealed at 350 °C in air, and then sintered at 1360 °C in an Ar-gas flow atmosphere. The fired bodies were ground using diamond wheels. Finally a number of three NEXCERA gauge blocks (200 mm in length, cross section: 9 mm×35 mm) were finished by Mitutoyo Corporation. As the samples themselves, the platens onto which the gauge blocks are contacted by wringing, are made of ultra low CTE material (ULE-glass from CORNING): In this way temperature induced strains between sample and platen are minimized.

3 Measurement methods

The absolute length measurements were performed at PTB's interference comparator INKO6 which primary purpose is the calibration of gauge blocks under ambient conditions as described in [2]. In the present state three stabilized lasers are available as light source: a J2-stabilized laser at 532 nm, a Zeeman-stabilized laser at 633 nm and a Rb-stabilized laser working at 780 nm. Absolute length evaluation from the interferograms is performed by specialized software, originally designed for PTB's Precision Interferometer [3]. Besides usage of the enhanced pixel resolution of the camera, this software applies accurate corrections for remaining non-parallelism of sample faces which otherwise generates a sensitivity of the measured length with respect to the lateral position along the surfaces. Each length measurement involves the phase topographies measured at the three wavelengths and each phase topography is calculated from four interferograms. The optics error of the interferometer at the actual pixel position of each sample is considered explicitly by applying an additional correction. Regions of interest (ROIs) are generated at well defined pixel positions and related sub-pixel corrections are extracted for the length evaluation. The phase topographies within the ROIs are unwrapped, averaged and further processed resulting in fractional orders of interference. The standard uncertainty of the length measurements concerning the present thermal expansion analysis is estimated to be

$u(l)$ = 2.5 nm. This value is dominated by the influence of the air refractive index which is sensitive to air parameters and thus on their measurement uncertainties.
All temperature sensors are linked to fix-point calibrations according to ITS-90. The overall standard uncertainty of the temperature measurement is estimated to be 10 mK. Temperature stability during each length measurement and homogeneity is better than 2 mK.

4 Measurement results

Figure 1, left, shows an example of a phase topography measured at the wavelength λ = 532.290008382 nm. Rectangles indicate ROIs at the platen and at the front face of the sample.

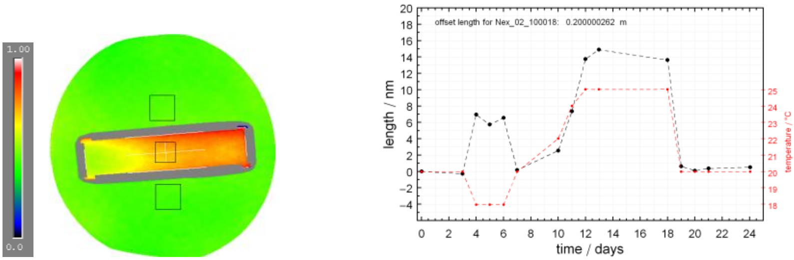

Figure 1: left: Example of an interference phase topography obtained for 532 nm wavelength for a single NEXCERA gauge block.
Right: Lengths of a NEXCERA gauge block (black data points) together with the temperature course (red data points).

Figure 1, right, shows the measured lengths together with the temperature course. The absolute length at 20°C is indicated as offset length which is subtracted from the actual lengths so that length changes are displayed. In Figure 2 the same data points are shown as a function of temperature. In the left part of the figure the measurement sequence is indicated by arrows and the number of the day of measurement is drawn. The right part of Figure 2 shows the data together with a parabolic fit polynomial. The upper part shows the deviation from the fit. The CTE (indicated as α) extracted from the polynomial fit is plotted as solid line in Figure 3.

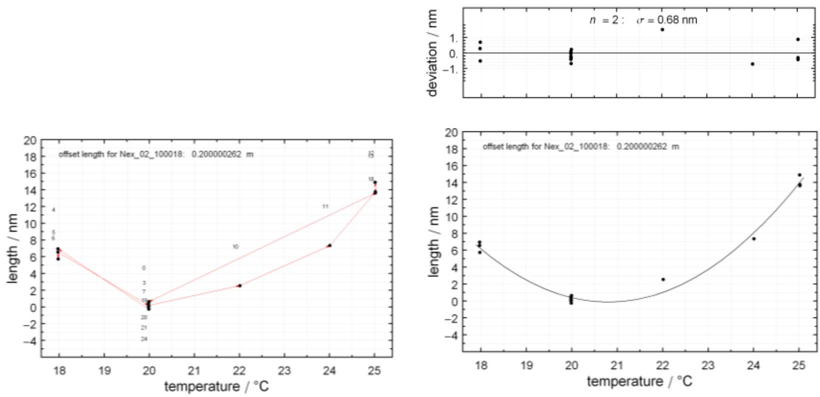

Figure 2: Data of Figure 1 displayed as a function of the sample temperature

The grey regions in the figure show the CTE uncertainties which are based on the measurement uncertainties of the input data. The light grey regions represent the amount of additional uncertainty due to the arbitrariness of the fit polynomial (see [4] for details). The fact that this contribution is very small indicates that the usage of $n = 2$ is highly appropriate. In each case of NEXCERA sample the resulting CTE ranges from about -2.3×10^{-8} K^{-1} (18°C) to 3.2×10^{-8} K^{-1} (25°C). From 20°C to 22°C the CTE is well within $\pm10^{-8}$ K^{-1} at an uncertainty level of less than 3×10^{-9} K^{-1}.

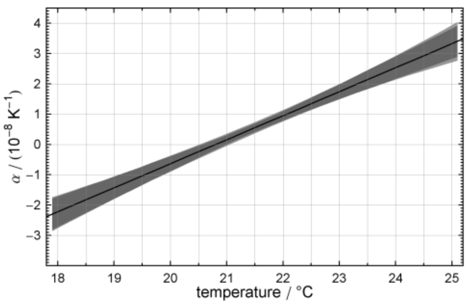

Figure 3:
CTE extracted from the data fit shown in Figure 2, right.

5 Conclusions

For all three samples, length relaxation and hysteresis can be concluded to be absent (at least on the level of the stated measurement uncertainty of 2.5 nm). This conclusion is also supported by length measurements repeated after two years (data not shown) indicating superiour long-term stability. The first data points measured at 20°C (day 0) agree with the last data points at 20°C (day 24) within ±1 nm.

The size of the temperature induced length changes has been found to be very small leading to ultra-low CTEs around room temperature.

References:

[1] S. F. Jakobs. Expansion hysteresis upon thermal cycling of Zerodur. Appl. Opt. 23, 3014-3016 (1984)

[2] G. Bönsch. Automatic gauge block calibrations by phase stepping interferometry with three laser wavelengths. Proc. SPIE 4401, 1-10 (2001).

[3] R. Schödel. Ultra high accuracy thermal expansion measurements with PTB’s Precision Interferometer, Meas. Sci. Technol. 19 084003 (2008).

[4] R. Schödel. Accurate extraction of thermal expansion coefficients and their uncertainties from high precision interferometric length measurements. Proc SPIE 5879, 1-11 (2005).