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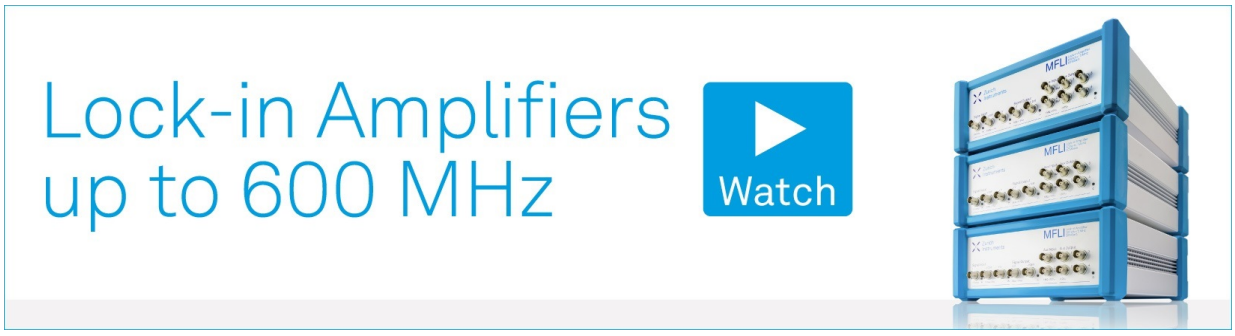
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6-H single-crystal silicon carbide thermo-optic coefficient measurements for ultrahigh temperatures up to 1273 K in the telecommunications infrared band

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6H single-crystal silicon carbide (SiC) is an excellent optical material for extremely high temperature applications. Furthermore, the telecommunication infrared band (e.g., 1500–1600 nm) is an eye safe and high commercial maturity optical technology. With this motivation, the thermo-optic coefficient $\partial n/\partial T$ for 6H single-crystal SiC is experimentally measured and analyzed from near room temperature to a high temperature of 1273 K with data taken at the 1550 nm wavelength. Specifically, the natural étalon behavior of 6-H single-crystal SiC is exploited within a simple polarization-insensitive hybrid fiber-free-space optical interferometric system to take accurate and rapid optical power measurements leading to $\partial n/\partial T$ data. The reported results are in agreement with the previously reported research at the lower <600 K temperatures. © *2005 American Institute of Physics*. [DOI: 10.1063/1.2133897]

I. INTRODUCTION

The proper design and performance of all-optical and optoelectronic devices requires knowledge of optical properties of materials. Some of these important optical properties include refractive index, reflectivity, and absorption. For proper device and system design, the measurement of refractive index is of prime importance. The refractive index of any material depends upon a large number of factors that includes temperature, pressure, wavelength, and other environmental conditions such as atmospheric gas concentrations and humidity. For key optical materials such as crystalline and amorphous silicon, the temperature dependence of refractive index n usually dominates the other mentioned factors.[1] The temperature T dependence of the refractive index is characterized by the thermo-optic coefficient (TOC) given by $\partial n/\partial T$. In particular, the TOC can change significantly as temperature is increased over a wide (e.g., 1000 K) range. Hence indirect measurement of the TOC using interpolation of lower temperature range data is not an accurate TOC measurement technique and direct TOC should be measured for higher temperatures.[2,3] A number of approaches have been used to measure the TOC of optical materials (e.g., GaAs, Si, and InP) using optics including direct techniques and indirect measurements.[4–7] Optical interferometry is an important tool in fast, reliable and accurate measurement of refractive indices.[8,9] In particular, the Fabry-Perot interferometric behavior of a test sample has been used to measure the refractive index and TOC. In this case, measuring the detuning of the detected multiple beam interference signal produced by a Fabry-Perot étalon test sample from its resonance position is used to determine the TOC.[10] It is important to note that the spectral resolution of the Fabry-Perot interferometer formed by the sample under test determines the TOC measurement resolution. In order to get clear resonance peaks (or dips) in the étalon spectrum, the optical reflectivities of the two sides of the étalon must be sufficient (e.g., $>20\%$). Hence care must be taken in preparing the étalon sample under test to enable the moderately high reflectivity for the two interfaces of the étalon.

Silicon carbide (SiC) is an excellent optical material because of its superior thermal, mechanical, electrical, and chemical inertness properties. Single-crystal 6H-SiC is in particular an optical quality (i.e., flat on an optical wavelength scale) large band gap planar material that can be fabricated in large sizes (e.g., >2 cm diameter wafers). In addition, single-crystal 6H-SiC can withstand high temperatures (e.g., 2500 K) and high pressures (50 atm). Recently, a single-crystal 6H-SiC based temperature sensor using optics has been designed and demonstrated for a room temperature to 1273 K temperature range.[11] Therefore, it is important to measure optical properties of 6H SiC for a wide range of temperature conditions. To date, to the best of the authors' knowledge, the TOC of 6H-SiC has been measured up-to 600 K with no extreme temperature data available as needed for high temperature sensing applications.[12] In addition, it is well known that 6H-SiC has some optical birefringence.[13] Previously reported 6H-SiC measurements did not taken into account the effect of material birefringence in their measurements. In addition, the 6H-SiC samples used had poor étalon reflectivities that led to poor spectral resolution for the étalon and hence the low quality of previous 6H-SiC TOC data plots.

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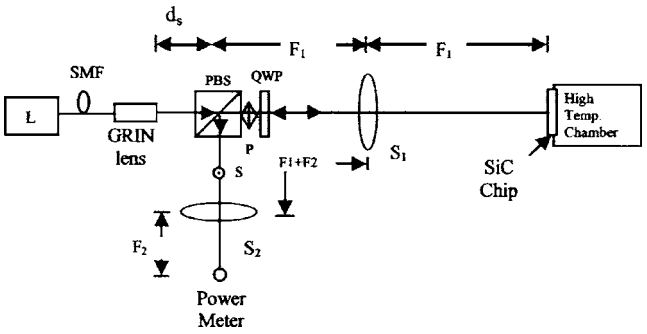


FIG. 1. Fiber-free-space hybrid thermo-optic coefficient (TOC) measurement system that exploits the natural étalon behavior of the single-crystal 6H-SiC chip placed in air. L: laser, PBS: polarization beam splitter, QWP: quarter wave plate, SMF: single mode fiber.

This paper reports the experimentally measured TOC for 6H-SiC from near room temperature to the extreme 1273 K temperature. In particular, the TOC measurement system takes into account the birefringent nature of the 6H-SiC sample and provides high resolution TOC data plots using the natural high quality étalon behavior of the single-crystal 6H-SiC bulk sample. In addition, the free-space-fiber hybrid TOC measurement system operates at the attractive eye safe telecommunications band centered around 1550 nm. The rest of the paper describes the TOC measurement system and the acquired high accuracy data.

II. FIBER-FREE-SPACE HYBRID TOC MEASUREMENT SYSTEM EXPERIMENT

Figure 1 shows the fiber-free-space hybrid TOC measurement system that exploits the natural étalon behavior of the single-crystal 6H-SiC chip placed in air. Specifically, single-crystal 6H-SiC has a refractive index near 2.57 for the 1550 nm band,[13] giving a sufficiently high near 20% Fresnel optical power reflection coefficient for the two air-SiC interfaces that form the étalon reflection mirrors. Hence, single-crystal 6H-SiC forms a natural high optical quality étalon that when placed in the Fig. 1 system forms a high modulation depth Fabry-Perot interferometer leading to accurate TOC measurements for the SiC test sample. Light from a Single Mode Fiber (SMF) coupled 1550 nm band laser is connected to a self-imaging type fiber collimation Graded-Index (GRIN) lens with a half-self imaging distance of d_s. Here d_s is the distance from the fiber lens to where the Gaussian beam forms its minimum beam waist.[14] Polarized light of mostly horizontal or p polarization is produced using a fiber-optic polarization controller (PC) that strikes the cube polarization beamsplitter (PBS). Hence the PBS also acts as a polarizer allowing only p-polarized light to enter the quarter-wave plate (QWP), thus producing circularly polarized light that passes through the SiC chip under test. Note that the QWP axis is oriented at 45° to the p-polarization direction to enable generation of circularly polarized light. Because circularly polarized light contains equal amounts of both p and vertical (or s) polarizations, the birefringence of the SiC sample irrespective of the SiC crystal ordinary and extraordinary axes is in effect averaged out with the double passage of the circularly polarized beam through the sample. Hence the Fig. 1 TOC measurement system in effect provides a polarization independent operation and a TOC measurement that takes into account the birefringence of the sample without requiring alignment of index axes with a given polarization. Thus, index averaged TOC data are measured for the birefringent test sample, in this case, 6H-SiC. Do note that prior room temperature infrared band birefringence data for 6H-SiC indicates it to be only weakly birefringent with a refractive index change of 0.04.[15] Hence, the Fig. 1 system also well approximates the polarization dependent TOC measurements for 6H-SiC.

The lens S_1 of focal length F_1 is placed at distance of d_s+F_1 from the GRIN lens. S_1 images the beam waist position that is at a distance d_s from the GRIN lens to the SiC plane placed at a distance of F_1 from S_1. Hence S_1 produces a perfectly collimated Gaussian beam on the SiC sample, allowing a low loss design for the final optical power measurement via the power meter. The reflected light from the SiC étalon returns via S_1 and the QWP to form a polarized beam with an s-polarization component. This s-polarized beam component is reflected by the PBS towards another lens S_2 of focal length F_2 that images the light on to the power meter. This deflected light is then focused onto a power meter by S_2 placed at a distance of F_1+F_2 from lens S_1. This optical design establishes a self-imaging type system to ensure optimal power transmission[14] and ensures a minimum beam waist position at the detector. Hence, if there is any vibration, the power meter will still capture the complete light beam given a sufficiently large photodetector area size. The SiC sample is placed in a high temperature chamber that causes the sample refractive index to change with temperature. Light reflected from the SiC étalon goes through optical power variations of several cycles as the temperature is swept over a large range. By measuring these power cycles, the TOC of SiC can be calculated as explained next.

The optical light power P_{FP_m} measured by the power meter is given by $P_{\mathrm{FP}_m}=KR_{\mathrm{FP}_m}$, where K is a constant that depends upon the experimental specifications like input power, power meter response, losses due to other optics, and the noise present in the system. As the noise in the system varies with time, this constant K keeps changing. R_{FP_m} is the SiC étalon reflectance given by

$$R_{\mathrm{FP}}=\frac{R_1+R_2+2\sqrt{R_1R_2}\cos\phi}{1+R_1R_2+2\sqrt{R_1R_2}\cos\phi}, \tag{1}$$

where R_1 and R_2 are the Fabry-Perot étalon front and back mirror reflectances, respectively. ϕ is the round-trip propagation phase accumulated by an optical beam while passing through the SiC étalon of thickness t and refractive index n at a wavelength of λ, and is given by $\phi=4\pi nt/\lambda$ at normal incidence. For the SiC sample in air, $R_1=R_2=R=r^2$, where $r=(n-1)/(n+1)$ is the Fresnel amplitude reflection coefficient of an air-SiC interface. Equation (1) indicates that the maximum reading of the power meter are taken when $\cos(\phi)=1$ or $\phi=2\pi m$, where $m=0,1,2,3,\ldots$, while the minimum reading of the power meter are taken when $\cos(\phi)=-1$ or $\phi=(2m-1)\pi$. As the temperature T of the SiC sample changes by an amount dT, the optical path length in the chip

changes, causing ϕ to change by $d\phi=2\pi$ cycles leading to consecutive optical peak (or null) measurements. Since ϕ is related to the SiC sample refractive index n, measuring the dT values that give the consecutive $d\phi=2\pi$ cycles over a large temperature range will allow the calculation of the change of refractive index ∂n with temperature change ∂T or the desired SiC TOC. Starting with

$$\phi = \frac{4\pi nt}{\lambda}, \tag{2}$$

and taking the derivative on both sides of Eq. (2) with respect to the temperature gives

$$\frac{d}{dT}\{\phi(T)\} = \frac{4\pi}{\lambda}\left[t(T)\frac{\partial}{\partial T}n(T) + n(T)\frac{\partial}{\partial T}t(T) \right]. \tag{3}$$

Here $\partial/\partial T\, n(T)$ is the TOC that has to be measured. Therefore Eq. (3) can be rearranged as

$$\mathrm{TOC} = \frac{\lambda}{4\pi t(T)}\frac{d}{dT}\{\phi(T)\} - \frac{n(T)}{t(T)}\frac{\partial}{\partial T}t(T). \tag{4}$$

Fundamentally, the thickness t of the SiC chip also changes with temperature. Specifically, the temperature dependence $t\,(T)$ of the thickness is characterized by the SiC thermal coefficient of linear expansion $\alpha=4.56\times10^{-6}$ K^{-1},[12,13] and is given by

$$t(T) = [1 + \alpha\Delta T]t(T_i), \tag{5}$$

where T_i is the initial (e.g., room) temperature and $\Delta T=T-T_i$ is the temperature difference. Differentiating Eq. (5) with respect to temperature (T) gives

$$\frac{\partial}{\partial T}t(T) = \alpha\, t(T_i). \tag{6}$$

Substituting Eq. (6) into Eq. (4) gives the desired TOC to be:

$$\mathrm{TOC} = \frac{1}{t(T)}\left(\frac{\lambda}{4\pi}\frac{d}{dT}[\phi(T)] - n(T)\alpha t(T_i) \right). \tag{7}$$

Here $d\phi(T)/dT$ is determined by measuring dT that gives the desired 2π phase shift corresponding to the adjacent detected optical power peaks (or nulls), while α is a known material constant. However based on Eq. (7), $n(T)$ and $t(T)$ are required for calculating a value for TOC. For the measurement of refractive index, one can write

$$\begin{aligned}\Delta\phi(\Delta T) &= \phi(T) - \phi(T_i) \\ &= \left(\frac{4\pi n(T)t(T)}{\lambda}\right) - \left(\frac{4\pi n(T_i)t(T_i)}{\lambda}\right).\end{aligned} \tag{8}$$

Rearranging the terms in Eq. (8):

$$n(T) = \frac{\Delta\phi(\Delta T)\lambda}{4\pi t(T)} + \frac{n(T_i)t(T_i)}{t(T)}. \tag{9}$$

Equation (9) shows that the refractive index of the étalon material $n(T)$, in this case, 6H-SiC can be calculated at higher temperatures (e.g., T) if the initial state, i.e., the initial thickness $t(T_i)$ and the refractive index at room temperature $n(T_i)$ are known, in addition to the elevated temperature thickness $t(T)$, the wavelength λ and the optical phase shift $\Delta\phi(\Delta T)$ change. The first term in Eq. (7) contains the term $d\phi(T)/dT$ that for a 300 μm 6H-SiC chip changes relatively fast compared to the second term containing the SiC instantaneous refractive index $n(T)$ that varies by a small relative amount with the same temperature change. Based on Ref. 11 data, a 300 μm 6H-SiC chip starting at room temperature produces a phase change of 2π for a 35 °C temperature change. The equivalent refractive index at a temperature of room+35 °C calculated by $n(T)=n(T_i)+\mathrm{TOC}\times\Delta T$ gives a value of $2.57+3.0\times10^{-5}\times35=2.5711$, where $n(T_i)=2.57$ is given in Ref. 12. Note that here $\mathrm{TOC}=3.0\times10^{-5}$ at room temperature has been used as cited in Ref. 12. Using a 1550 nm wavelength, the Eq. (7) first term$\lambda/4\pi\, d/dT[\phi(T)]$ is calculated to be 2.58×10^{-8} m/K while the Eq. (7) second term $n(T)\alpha t(T_i)$ provides a contribution of 3.5185×10^{-9} m/K. Hence, the contribution from the second term is smaller by a factor of 7.33 as compared to the first term for the temperature change of 35 °C from room temperature to ~335 K. A similar calculation done at 600 K where a smaller 25 °C change provides a 2π change[11] gives a value of 3.10×10^{-8} m/K for the first factor in Eq. (7), while the contribution from the second factor is 3.5175×10^{-9} m/K. This calculation shows that a near 300 °C temperature change produces a 20% change in the first factor in Eq. (7) while the second term is changed by only 0.01%. Hence, it can be inferred that the second term in Eq. (7) provides approximately an order of magnitude smaller and constant contribution to TOC calculations. Therefore the measured 6H-SiC TOC can be approximated as

$$\mathrm{TOC} \approx \frac{\lambda}{4\pi t(T)}\frac{d}{dT}\{\phi(T)\}. \tag{10}$$

However, for a general material and for higher precision measurements, Eq. (7) should be used that requires prior knowledge of the refractive index at room temperature at the operating wavelength.

Data from the Fig. 1 system provides the temperature locations over an entire test range that correspond to optical power peaks (or nulls), where $d\phi(T)$ has changed by 2π due to temperature change dT. Using this measured temperature data set corresponding to the optical power peaks (or nulls), a numerical function representing $d\phi(T)/dT$ is found. Next using Eq. (10) with the numerical function, the TOC of the test sample is calculated. To implement this procedure, an experiment is performed using the Fig. 1 system to measure the TOC of 6H-SiC at the telecom 1550 nm wavelength and extreme temperature ranges from near room temperature to 1273 K.

III. RESULTS AND DISCUSSION

The Fig. 1 system is setup in the laboratory using a single-crystal 6H:SiC with a thickness $t(T_i)$ of 300 μm (at room temperature) and a laser source at 1550 nm wavelength. The temperature of the SiC chip is raised from room temperature to 1273 K (1000 °C) by using an electronically controlled induction heater that heats a steel cylinder on which the SiC chip is placed. The temperature is recorded by a k-type thermocouple in contact with the SiC chip. The Fig.

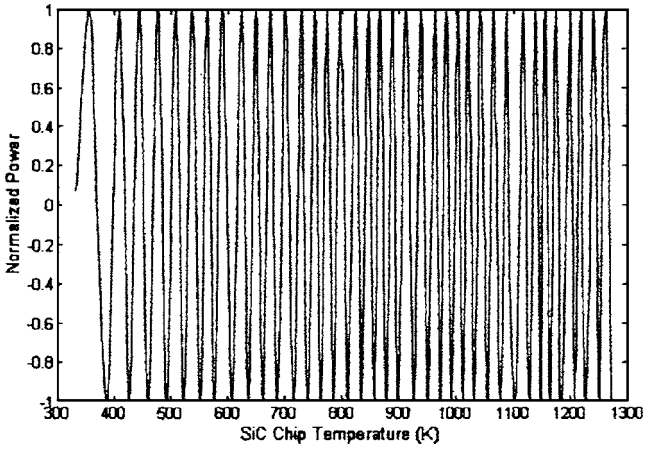


FIG. 2. Normalized power measured by the power meter in Fig. 1 as the SiC chip temperature is increased from near room temperature to 1273 K.

1 optical design is a high optical throughput setup with polarizing multiplexing used to separate the transmit and receive light channels. Given all antireflection (AR) coated components, the majority of light lost in the system comes mainly from the Fresnel loss of the 6H-SiC étalon, with at most 30% light returned by the étalon to the detector. As the temperature of the SiC chip is changed, the optical path length in the étalon changes causing the detected optical power to cycle between maxima and minima as seen by Fig. 2 where the power is shown after normalization using localized maximum and minimum values. From this normalized data, the locations of consecutive dT values can be determined that produce the consecutive $d\phi(T)=2\pi$ phase shifts leading to generation of a plot of unwrapped phase shift $\phi(T)$ versus temperature T. This plot is shown in Fig. 3 and is given by the expression $\phi(T)=9.4\times10^{-8}T^3-0.00031T^2+0.0046T+18$. Note that a numerical cubic fit on the Fig. 3 data is used to calculate $d/dT\{\phi(T)\}$ for the chosen 6H-SiC sample. In Fig. 3, note that the variation between the data points and the cubic curve fit is very minimal. Another aspect of the demonstrated experiment is the availability of a large number of data points from near room temperature to 1273 K (1000 °C). Specifically, there are 36 full peak-to-peak power

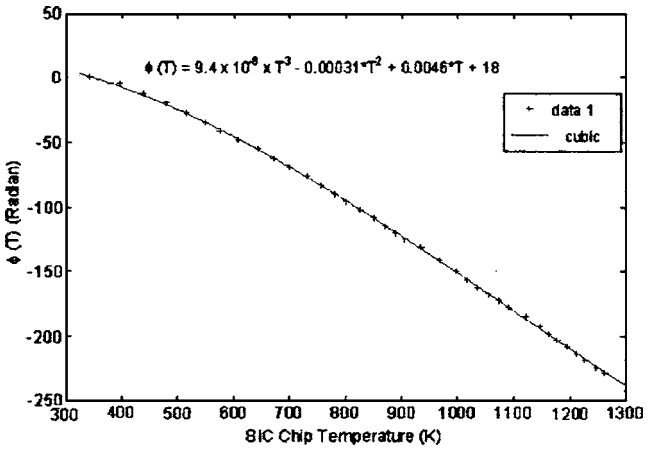


FIG. 3. Plot of measured modulo-2π values of phase $\phi(T)$ vs SiC chip temperature. The dots are the 37 data points while the solid line is the cubic fit to the data.

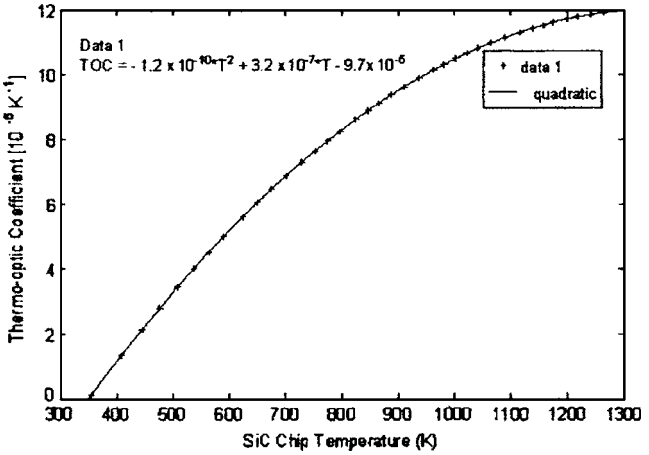


FIG. 4. Calculated 6H-SiC thermo-optic coefficient (TOC) versus SiC temperature at 1550 nm using the Fig. 3 measured cubic fit data for the phase $\phi(T)$ change with temperature. The TOC expression shows a quadratic dependence on the temperature.

cycles leading to a densely filled improved curve fitting. Based upon the $d/dT\{\phi(T)\}$ curve fit data in Fig. 3, the 6H-SiC TOC is calculated by using Eq. (10) and Eq. (5) and the result is shown in Fig. 4. This TOC measurement can be expressed as

$$\mathrm{TOC}=-1.2\times10^{-10}T^2+3.2\times10^{-7}T-9.7\times10^{-5}\ \mathrm{K}^{-1}. \tag{11}$$

To check the validity of the conducted experiment represented by Eq. (10), the refractive index of SiC is calculated using Eq. (9) and Eq. (5) and by using the prior room temperature SiC refractive index value $n(T_i)$ of 2.57 along with a SiC thermal expansion coefficient of α of 4.56×10^{-6} K^{-1}.[12,13] Note that $\Delta\phi(\Delta T)$ needed for evaluating Eq. (9) is calculated via Eq. (8), i.e., by using room temperature phase as the reference phase and subtracting this phase value from the subsequent unwrapped phase values obtained from the Fig. 3 measured expression of $\phi(T)=9.4\times10^{-8}T^3-0.00031T^2+0.0046T+18$ at the elevated temperature T. Hence, the resultant refractive index $n(T)$ is calcu-

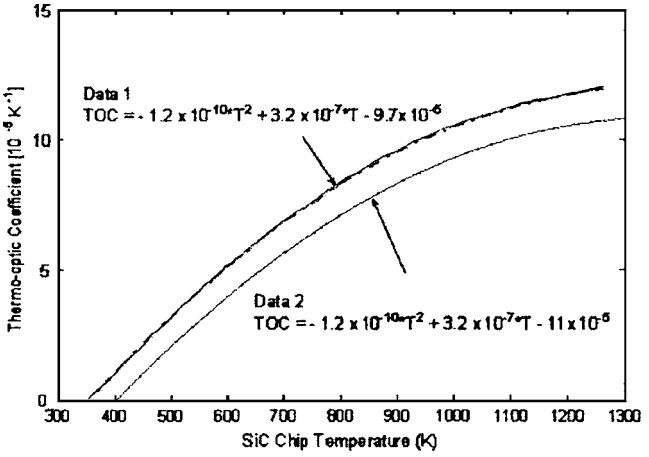


FIG. 5. Calculated 6H-SiC thermo-optic coefficient (TOC) versus SiC temperature at 1550 nm. Data 1 curve is from Fig. 4 using the approximate expression of Eq. (10) for TOC. Data 2 curve is using the exact expression of Eq. (7) for the TOC.

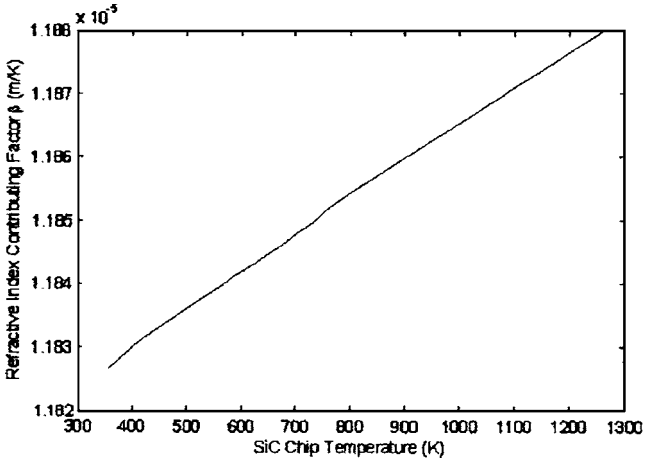


FIG. 6. Plot of the factor $\beta=n(T)\alpha t(T_i)/t(T)$ in Eq. (7), i.e., the difference between Data 1 and Data 2 in Fig. 5 showing the small error introduced if TOC of 6H-SiC chip is calculated without prior knowledge of 6H-SiC refractive index.

lated using Eq. (9) and used in Eq. (7) to calculate the TOC as shown by the data 2 curve in Fig. 5. This TOC quadratic curve fit expression is given by

$$\mathrm{TOC} = -1.2\times10^{-10}T^2 + 3.2\times10^{-7}T - 11\times10^{-5}\ \mathrm{K}^{-1}. \tag{12}$$

Note that Eqs. (11) and (12) are in good agreement with a slight difference only in the constant term (i.e., 11 instead of 9.7) indicating that the approximation in Eq. (10) is justified for a 300 μm thick 6H-SiC chip. Note that the difference between the expressions in Eq. (7) and Eq. (10) is given by $\beta=n(T)\alpha t(T_i)/t(T)$ that is plotted in Fig. 6 for the experiment conducted in this paper. Results show that ignoring β introduces a relatively small error (1.182×10^{-5} to 1.188×10^{-5} m/K over a 1000 K temperature change) in calculating the TOC of the used 6H-SiC chip using Eq. (10). A key advantage of using the Eq. (10) approximation to calculate the SiC chip TOC is that it eliminates the need for any prior knowledge of the SiC refractive index $n(T)$.

IV. CONCLUSION

In conclusion, implemented is a high optical efficiency fiber-free-space hybrid interferometer to measure the TOC of single-crystal 6H-SiC. Theoretical analysis is carried out to include refractive index and thickness change with temperature change. The TOC for 6H-SiC is reported at 1550 nm over the wide range of near room temperature to 1273 K. Results are in agreement with the previous lower temperature range results, e.g., $\mathrm{TOC}=6\times10^{-5}\ \mathrm{K}^{-1}$ at 600 K.

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