SRM 1460 Series as a Thermal Diffusivity Standard for Laser Flash Instruments[1]

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ABSTRACT

The specific heat and thermal expansion ($\Rightarrow$ bulk density) of the SRM 1460 series austenitic stainless steel have been measured with the intent of converting this material to a thermal diffusivity standard. These data, along with the recommended NIST values of thermal conductivity, were used to compute the thermal diffusivity between -100 and 875°C. The estimated uncertainties in the computed thermal diffusivity values are 3.9% between -100 and 25°C and 5.6% above 25°C. In addition, the thermal diffusivity was measured directly and compared with the computed data. The deviations between the two data sets are generally less than 3.0% between 50 and 850°C.

KEY WORDS: bulk density; specific heat; SRM 1460 series; thermal diffusivity; thermal diffusivity standard

1. INTRODUCTION

The laser flash has become the most widely-used instrument for the measurement of the thermal diffusivity. It has been estimated that over 80% of the thermal diffusivity measurements conducted worldwide are done with laser flash instruments. The reasons are the accuracy, the relatively short measurement times, the easy sample preparation and the fact that such a wide range of materials can be tested.

In spite of the fact that the laser flash is so well accepted and widely used, almost no reliable reference standards exist for this method. This is especially surprising in light of the fact that there are so many excellent thermal conductivity standards. For example, graphite, stainless steel, electrolytic iron and sintered tungsten, designated RM 8424, SRM 1460, RM 8420 and SRM 8422, respectively, have been issued by NIST as thermal conductivity and electrical resistivity standards. Although thermal diffusivity, specific heat and thermal expansion measurements have been carried out on many of these materials over the years, to the best of the authors' knowledge there has been no concentrated effort to extend them to thermal diffusivity standards.

There has, however, been some work directed at establishing thermal diffusivity standards. For example, POCO AXM-5Q1 graphite has been well characterized using laser flash instruments, but its consistency is somewhat in question [1]. Glass ceramics, such as Pyroceram 9606, have also been used as laser flash standards, but these materials are translucent and must be coated on both sides to ensure that all of the laser energy is absorbed on the front surface and that the temperature rise is measured only on the rear surface. Coating materials such as graphite and silicon-carbide have been used for this purpose, but upon heating, they tend to diffuse into the sample. It is not entirely clear what effect this contamination has on the thermal diffusivity. Recently, Baba and Ono [2] carried out laser flash measurements on a glass-carbon material.

Based on the results of this work, it would appear that this material may be consistent and stable enough to use as a thermal diffusivity standard, but it is probably too early to pass judgment on this.

A viable alternative to developing new reference materials for the laser flash is to convert the SRM/RM thermal conductivity standards to thermal diffusivity standards. This may be the most pragmatic approach, since these materials are consistent, reliable, well-characterized and come complete with accurate thermal conductivity values. Further, since the materials are opaque, coating problems are not an issue. The conversion can be easily accomplished by measuring the specific heat and thermal expansion ($\Rightarrow$ bulk density) and calculating the thermal diffusivity using the combination of these data and the existing thermal conductivity data. The purpose of this work was to convert the SRM 1460 series austenitic stainless steel to a thermal diffusivity standard over the temperature range of -100 to 875°C as described above. In addition, the thermal diffusivity was measured between 20 and 900°C and compared with the calculated thermal diffusivity values.

2. EXPERIMENTAL

The specific heat measurements were carried out using a Netzsch DSC 404 differential scanning calorimeter capable of operation from -120 to 1500°C (using two furnaces). The low-temperature measurements were carried out over the temperature range of -100 to 100°C at a heating rate of 10 K/min in a dynamic helium atmosphere with a flow rate of 50 ml/min. A heating rate of 20 K/min and a dynamic argon atmosphere with a flow rate of 50 ml/min were used for the high-temperature measurements between 100 and 875°C. Sapphire was employed as the calibration material and the data were reduced using the well-known ratio method. The test sample

dimensions were nominally 6.0 mm diameter by 1.3 mm thick. The instrument and measurement techniques have been described by Henderson, et al. [3].

The thermal expansion was measured using a Netzsch model 402 C pushrod dilatometer capable of operation from -160 to 1600°C (using two furnaces). The measurements were conducted at 3 K/min in a dynamic helium atmosphere at a flow rate of 75 ml/min. The system was calibrated using platinum for the low-temperature measurements between -150 and 50°C and sapphire for the high-temperature measurements between 50 and 875°C. The nominal test sample dimensions were 6.0 mm diameter by 25.0 mm long.

The thermal diffusivity was measured over the temperature range of ≈20 to 900°C in an argon atmosphere at a flow rate of 150 ml/min using a Netzsch model 427 laser flash diffusivity apparatus. The unit used in this work was equipped with a high-temperature, water-cooled furnace capable of operation from 20 to 2000°C. The sample chamber is isolated from the graphite heating element by a protective tube, allowing samples to be tested under a vacuum or in an oxidizing, reducing, or inert atmosphere. The test samples had nominal dimensions of 12.5 mm diameter by 3.0 mm thick. The instrument and the laser flash method have been described in detail by Bräuer, et al. [4]. The reader is referred to this publication for further details.

3. RESULTS AND DISCUSSION

The temperature-dependent specific heat data are depicted in figure 1. The measurements were carried out on two samples and each sample was run twice. In order to eliminate bias due to any single measurement, a separate baseline and calibration standard run was performed for each sample measurement. As can be seen, there are no significant differences in the specific heat values between samples 1 and 2.

The scatter in the data is random and generally lies within the 2.5% accuracy of the instrument. The largest deviation in these data is ≈2.9% and occurs between sample 2, run 1 and sample 2, run 2 at 875°C. The maximum scatter lies generally in the range of 1.0 - 2.0%. The deviations from the average specific heat values are around 0.5 - 1.0%. Generally, the specific heat data show the expected behavior, increasing as a monotonic function of temperature, with the exception of the anomaly which occurs between ≈575 and 650°C. This anomaly is not atypical for austenitic stainless steel and has been reported, for example, by Binkele [5].

Shown in figure 2 are the linear thermal expansion data. As with the specific heat, two separate samples were tested, with two runs per sample. Also, a separate calibration was performed for each sample measurement, in order to reduce the chances of the data being biased. Inspection of figure 2 reveals that the scatter in the data is extremely low and that the data for samples 1 and 2 are indistinguishable. The maximum absolute scatter is only ≈0.014% at 875°C and occurs between sample 1, run 1 and sample 1, run 2. Generally, the relative deviations from the average linear thermal expansion are less than 0.5%. Clearly, the material expands as a monotonic function of temperature and, unlike the specific heat, displays no deviation from the trend between 575 and 650°C.

The average linear thermal expansion data were used to calculate the volumetric expansion, assuming isotropic behavior, and the bulk density. The results of the computations are presented in figure 3. The room temperature bulk density value of 8.007 g/cm^3 recommended by NIST was used in the computations. It should be pointed out, however, that the measured room temperature bulk density value of the test samples was ≈7.995 g/cm^3, which is ≈0.15% lower than that recommended by NIST.

The thermal diffusivity of the material was calculated using the average measured specific heat and bulk density values along with the thermal conductivity values recommended by NIST. Since the computations were made at 50°C intervals starting at -100°C, the NIST thermal conductivity data were interpolated to the desired temperatures using a spline function. The results of these computations are shown in figure 4. Obviously, the anomaly in the specific heat is also present in these data. The error bands were calculated using the standard error propagation equation, taking uncertainties of 2.5% and 0.5% in the specific heat and density data, respectively, over the entire temperature range. The uncertainties in the thermal conductivity data were taken as 3.0% between -100 and 25°C and 5.0% between 25 and 875°C, as recommended by NIST. The measured average specific heat and bulk density data, the interpolated NIST thermal conductivity data and the computed thermal diffusivity values are summarized in Table I.

Presented in figure 5 are the results of the thermal diffusivity measurements. Two separate samples were measured, with three runs per sample. Further, each data point shown is the average of 3 laser shots and all values were corrected for thermal expansion. As with the other measurements, the scatter in the data is quite low and the thermal diffusivity values of samples 1 and 2 display no significant differences. The maximum scatter in the data is ≈2.1%, occurring at ≈20°C between sample 2, run 1 and sample 2, run 3. As can be seen in figure 6, the deviations from the average thermal diffusivity values generally lie in the range of 1.0%. It is interesting to note that the anomaly seen in the specific heat data is also present in the thermal diffusivity, although the effect is much less apparent. In addition, it appears that the anomaly in the thermal diffusivity data occurs above 600°C.

In figure 7, the average measured and computed thermal diffusivities are compared over the temperature range of 50 to 850°C. The average measured thermal diffusivity values were interpolated to 50°C increments starting at 50°C, in order to compare these data directly with the computed thermal diffusivity. Two sets of error bands are shown in figure 7. The computed data carry the 5.6% error bands as shown in figure 4 (solid lines), while the measured values carry 5.0% bands (broken lines). The deviations be-tween the two sets of data are less than 5.0% over the entire temperature range, with the exception of the 50°C value, where the deviation is 5.5%. In general, the deviations be-tween the two data sets are in the range of 3.0%. The calculated and average measured thermal diffusivity values, along with the percent deviation are summarized in Table II.

4. CONCLUDING COMMENTS

The specific heat and thermal expansion ($\Rightarrow$ bulk density) of the SRM 1460 series austenitic stainless steel have been measured with the intent of converting this material to a thermal diffusivity standard. These data, along with the recommended NIST values of thermal conductivity, were used to compute the thermal diffusivity. The estimated uncertainties in the computed thermal diffusivity values are 3.9% between -100 and 25°C and 5.6% above 25°C. In addition, the thermal diffusivity was measured directly and compared with the computed data. The deviations between the two data sets are generally less than 3.0%.

It is the opinion of the authors that the SRM 1460 series is quite suitable as a thermal diffusivity standard. The deviations between the measured and calculated thermal diffusivity values lie within an acceptable range. In addition, the material

is very consistent and the thermophysical properties are clearly reproducible. Certainly, some effort should be made to explain the anomaly seen in the data. Further, additional measurements should be made by other laboratories in order to confirm and to raise the confidence in these results.

References

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Table I. Specific heat, bulk density, thermal conductivity

and computed thermal diffusivity values

T (°C)	T (K)	λ* (W/m-K) literature	Specific Heat (J/g-K) measured (avg)	Bulk Density (g/cm^3) measured (avg)	a (cm^2/s) calculated
-150	123	10.20	---	8.067	---
-100	173	11.56	0.413	8.051	0.0348
-50	223	12.73	0.433	8.034	0.0366
0	273	13.78	0.445	8.015	0.0386
50	323	14.77	0.461	7.995	0.0401
100	373	15.69	0.476	7.975	0.0413
150	423	16.55	0.495	7.954	0.0420
200	473	17.36	0.510	7.933	0.0429
250	523	18.13	0.520	7.910	0.0441
300	573	18.85	0.530	7.889	0.0451
350	623	19.54	0.538	7.866	0.0462
400	673	20.20	0.544	7.844	0.0473
450	723	20.83	0.552	7.821	0.0482
500	773	21.43	0.556	7.798	0.0494
550	823	22.01	0.565	7.775	0.0501
575	848	22.29	0.573	7.764	0.0501
600	873	22.57	0.583	7.752	0.0499
625	898	22.84	0.582	7.740	0.0507
650	923	23.11	0.584	7.728	0.0512
700	973	23.63	0.589	7.704	0.0521
750	1023	24.13	0.593	7.681	0.0530
800	1073	24.61	0.598	7.657	0.0537
850	1123	25.07	0.601	7.633	0.0546
875	1148	25.30	0.603	7.620	0.0551

*Source: NIST Certificate for SRM 1461, 1984

Table II. Calculated and average measured thermal diffusivity values and percent deviation

T (°C)	T (K)	a (cm^2/s) calculated	a* (cm^2/s) measured (avg)	Percent Deviation
50	323	0.0401	0.0379	-5.5
100	373	0.0413	0.0393	-4.8
150	423	0.0420	0.0406	-3.3
200	473	0.0429	0.0419	-2.3
250	523	0.0441	0.0431	-2.3
300	573	0.0451	0.0443	-1.8
350	623	0.0462	0.0455	-1.5
400	673	0.0473	0.0467	-1.3
450	723	0.0482	0.0479	-0.6
500	773	0.0494	0.0490	-0.8
550	823	0.0501	0.0501	0.0
600	873	0.0499	0.0513	2.8
650	923	0.0512	0.0526	2.7
700	973	0.0521	0.0536	2.9
750	1023	0.0530	0.0544	2.6
800	1073	0.0537	0.0552	2.8
850	1123	0.0546	0.0560	2.5

*corrected for thermal expansion

FIGURE CAPTIONS

Fig. 1. Specific heat

Fig. 2. Linear thermal expansion

Fig. 3. Volumetric expansion and bulk density

Fig. 4. Computed thermal diffusivity

Fig. 5. Measured thermal diffusivity

Fig. 6. Deviation of measured thermal diffusivity data from average values

Fig. 7. Calculated and average measured thermal diffusivity

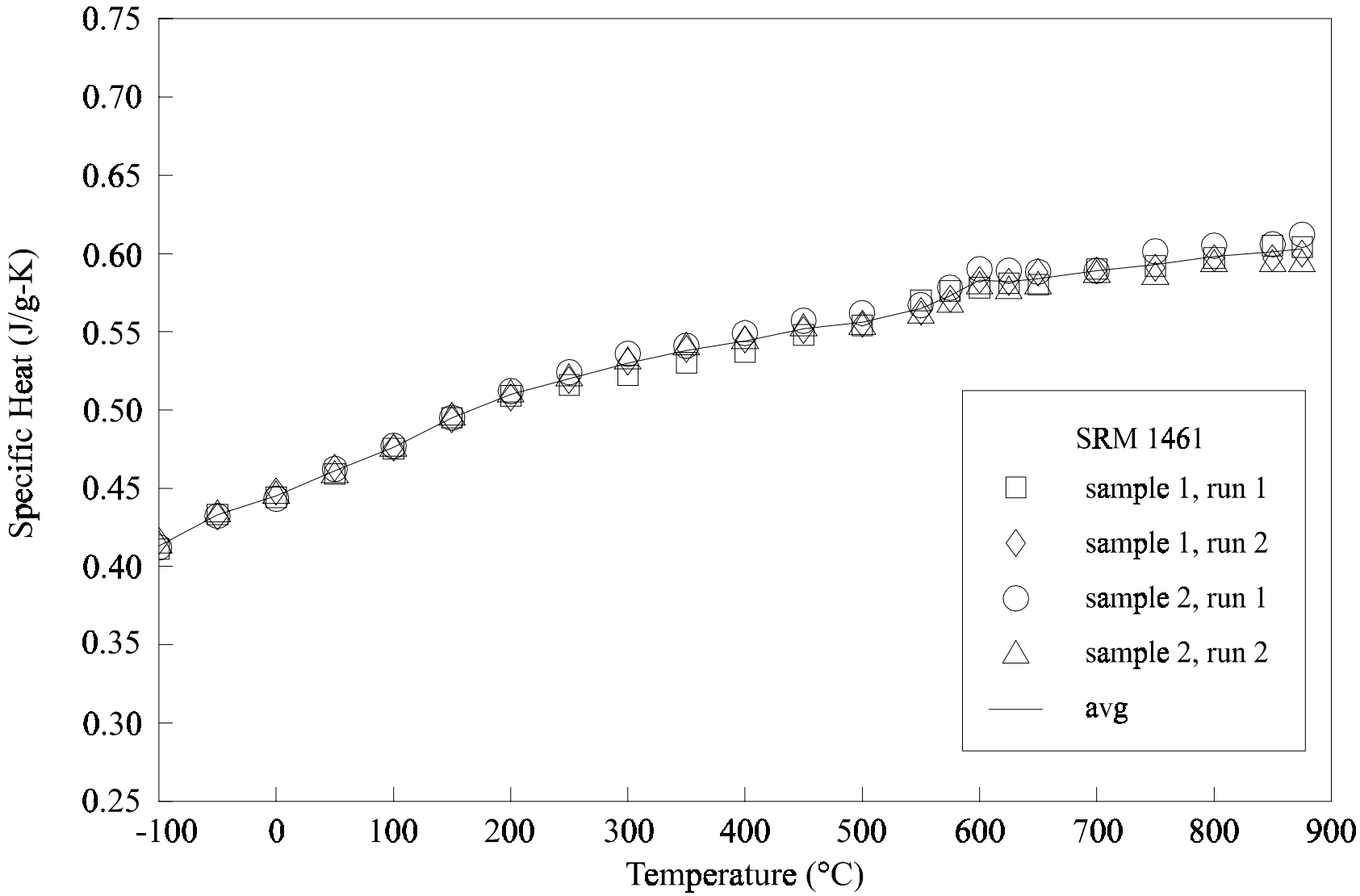

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Fig. 1

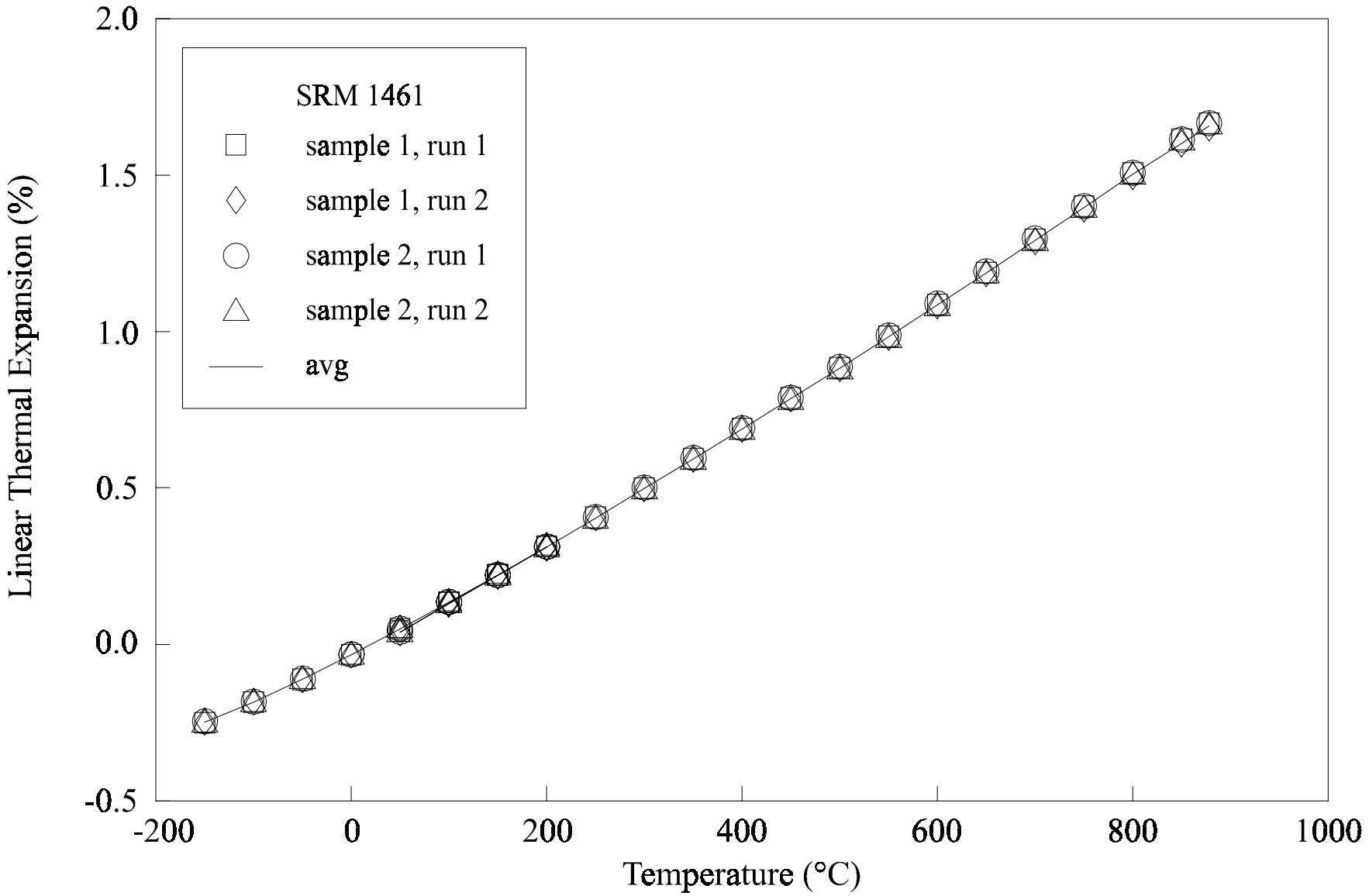

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Fig. 2

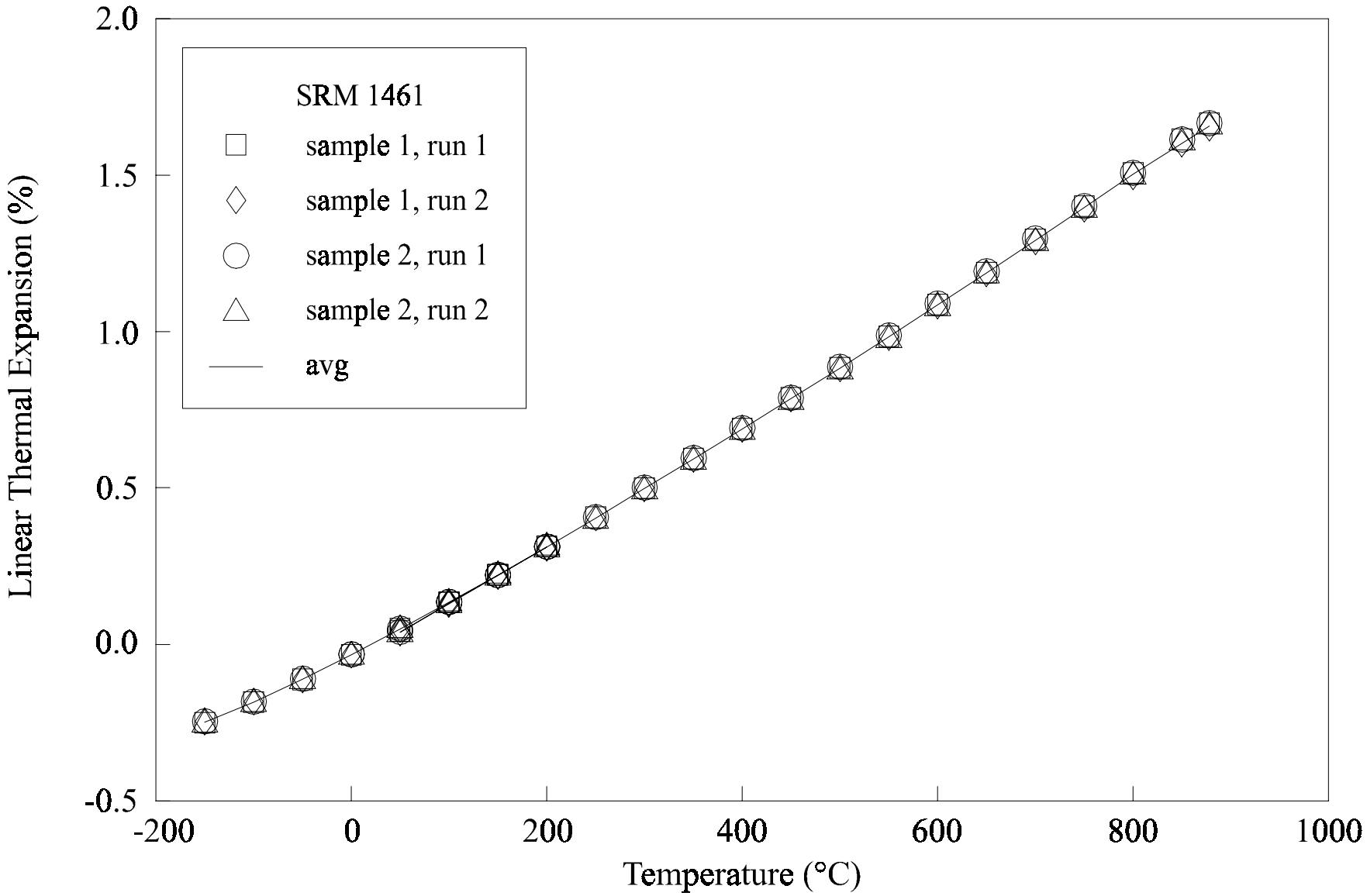

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Fig. 3

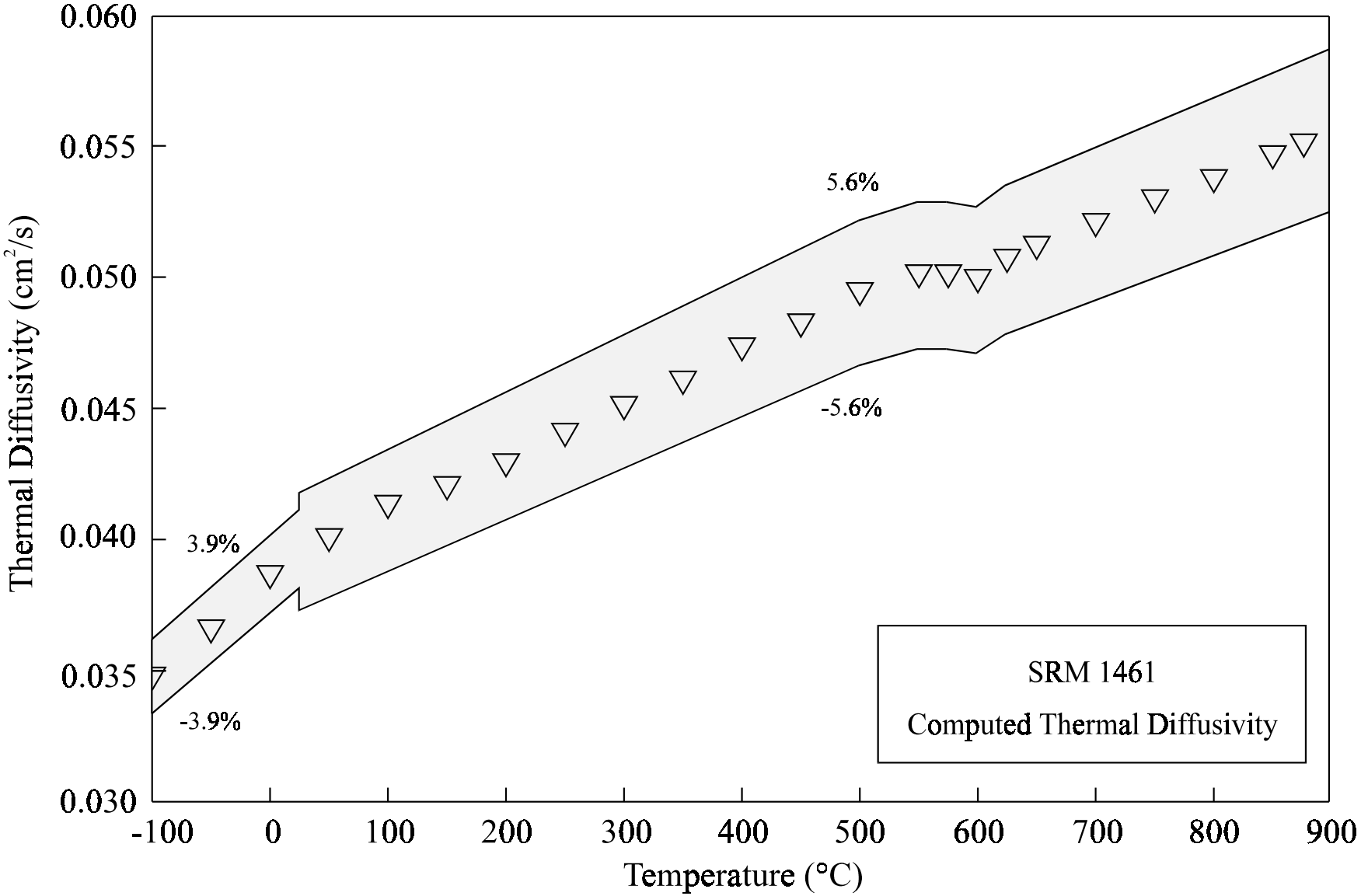

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Fig. 4

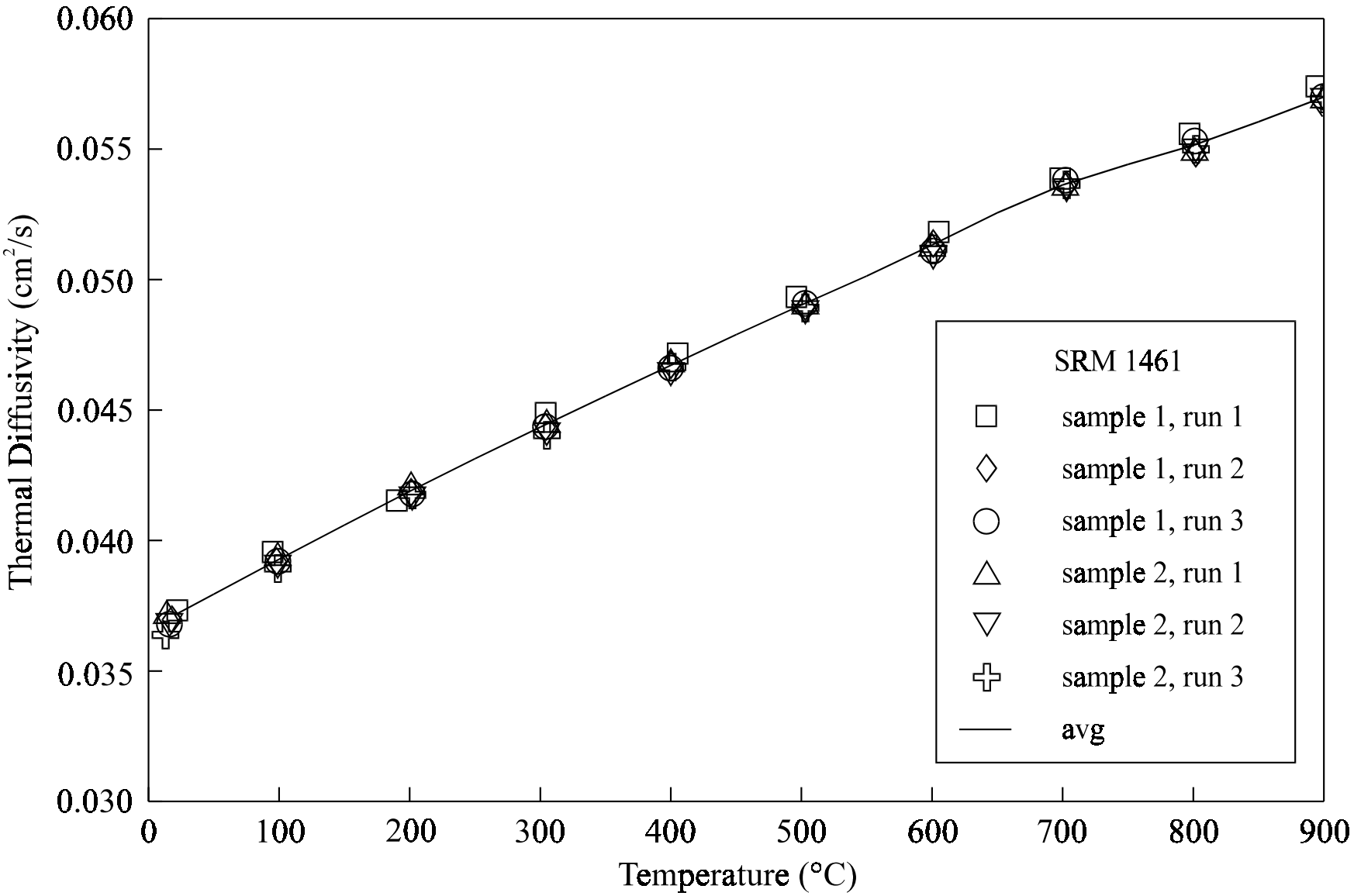

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Fig. 5

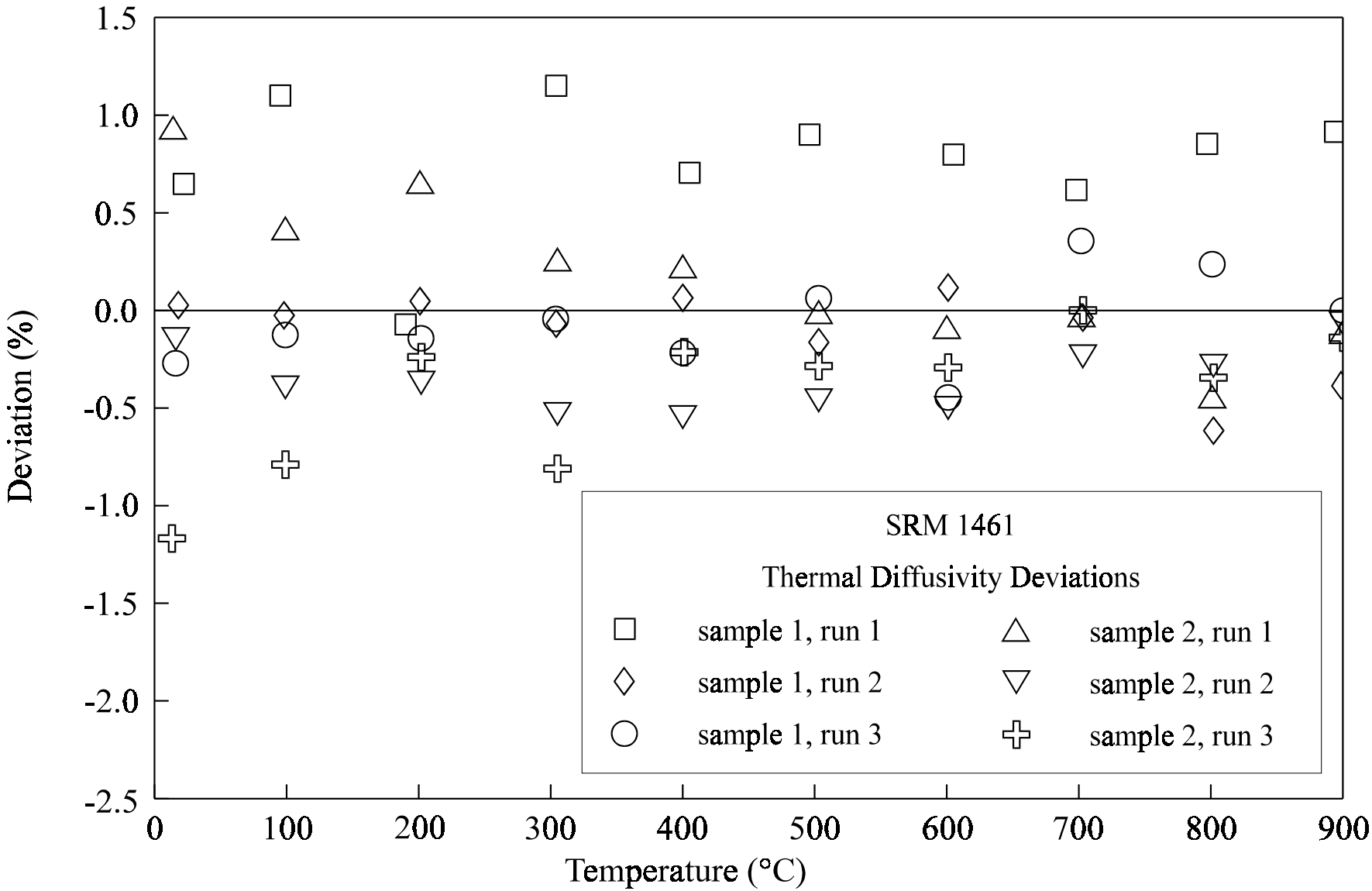

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Fig. 6

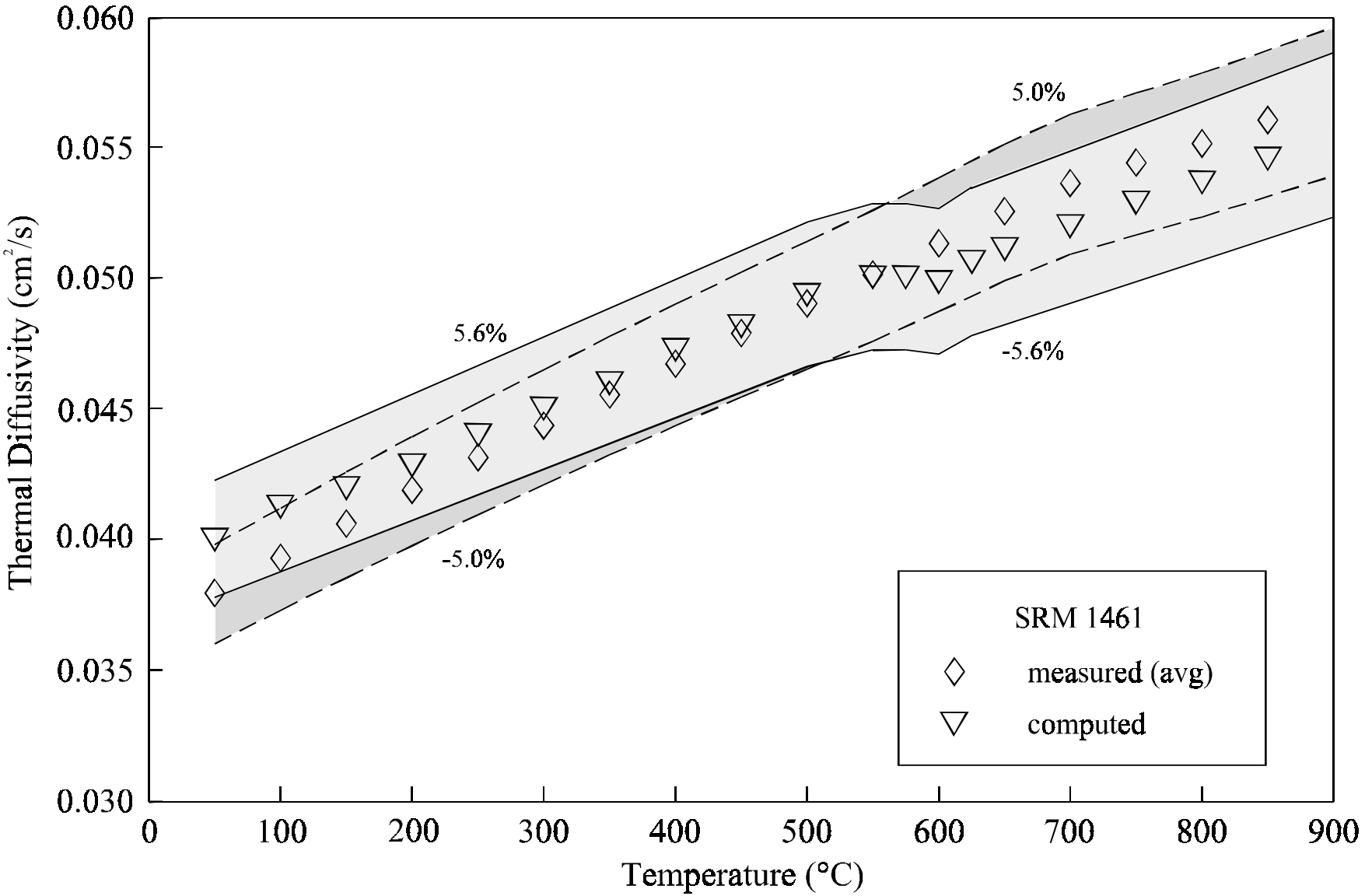

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Fig. 7