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Research on Several Factors Affecting the Test of Thermal Expansion Coefficient of Glass Materials

Improving accuracy for high-performance applications

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Accurate measurement of glass transition temperature (T_g) and coefficient of thermal expansion (CTE) is of great significance in guiding the use and process performance of the materials. This manuscript measures the CTE and T_g of samples by the thermal expansion method and systematically researches the measurement factors affecting the T_g and the CTE, including the shape and size of the samples, the starting furnace temperature and the heat treatment process. The study shows that the sample shape and size, the starting furnace temperature and the heat treatment process all have an effect on the test results. At the same time, the placement of the sample and the data processing method will also make the test results deviate from the real value. Therefore, in order to accurately assess the thermal properties of the material, the size of the sample is specified to be 6 mm in diameter and 50 mm in length, while the initial temperature of the furnace during the CTE test should be lower than 35°C. In addition, test samples of the same glass grade should undergo the same heat treatment process to ensure the accuracy of the test results.

Keywords

glass; thermal expansion method; coefficient of thermal expansion; factors

1. Introduction

The application of glass materials in modern engineering structures requires them to adapt to extreme conditions. Therefore, it is of great importance for designers to test their performance in advance. T_g is the main index to measure the service temperature or heat resistance of polymer materials (1, 2). The thermal properties of the samples are tested and evaluated by thermal dilatometer (3, 4). The push rod expander has the advantages of high resolution, wide range and good linearity (5). It can contact the sample through the ejector rod before the test and automatically measure the initial length of the sample under the action of the contact force. It is easy for users to operate the push rod expander and obtain reliable data (6).

With continuous development, the accuracy requirements of parts in machinery, aviation and other fields are getting higher and higher. The temperature deformation of parts is generally explained and compensated by thermal expansion performance, so the accurate measurement of CTE will directly affect the accuracy of material compensation. Therefore, the CTE of materials has received extensive attention (7, 8). Lu *et al.* (9) studied the effect of heat treatment process on the thermal expansion behaviour of glass. The results show that the same glass sample has different thermal expansion properties after different heat treatment processes. Wang *et al.* (10) studied the effect of stress conditions on the CTE of IG-101 graphite. The results show that the absolute variation of the CTE increases with the increase of the applied load. Laplanche *et al.* (11) studied the temperature dependence of the CTE of cobalt-chromium-iron-manganese-nickel alloy. The results show that the CTE of the sample changes with temperature. However, few studies have been reported on the CTE of glass samples.

Based on the above reasons, this work systematically studied the influencing factors of glass thermal expansion behaviour by using a push rod dilatometer. The factors involved in the sample size and shape, heat treatment process and test process are analysed, which provides a reference for the reliability measurement of CTE.

2. Experimental Section

2.1 Sample Preparation

A specific glass material, named CPB (from China Building Materials Academy Co Ltd) (12), was used and the basic chemical composition of this glass is shown in **Table I**. Firstly, the glass material was annealed in a muffle furnace, heated to 600°C, with a constant temperature for 3 h; secondly, they were cooled to room temperature at a cooling rate of 2°C min^{-1} according to the standard requirements; then, cylinders with a diameter of 6 mm and a length of 50 mm were intercepted from the annealed glass material as the test specimens (**Figure 1**). Both ends of the test samples were ground to be parallel, the initial length (L_0) was measured by a numerically-displayed vernier callipers with the minimum index value of 0.01 mm, then they were left to stand in the test environment for more than 30 min.

2.2 Experimental Instruments

The CTE of the sample was tested by the thermal expansion method using the DIL 402 Expedis® Classic (NETZSCH Group, Germany) push rod horizontal thermal expansion instrument (**Figure 2(a)**) (13). In the experiment, the alumina standard sample was used to calibrate the

Table I Basic Chemical Composition of CPB Glass Material

Chemical composition	B_2O_3	Al_2O_3	CaO	BaO	SiO_2	CeO_2	Others
wt%	40.0	3.0	6.0	25.0	8.0	5.0	13.0

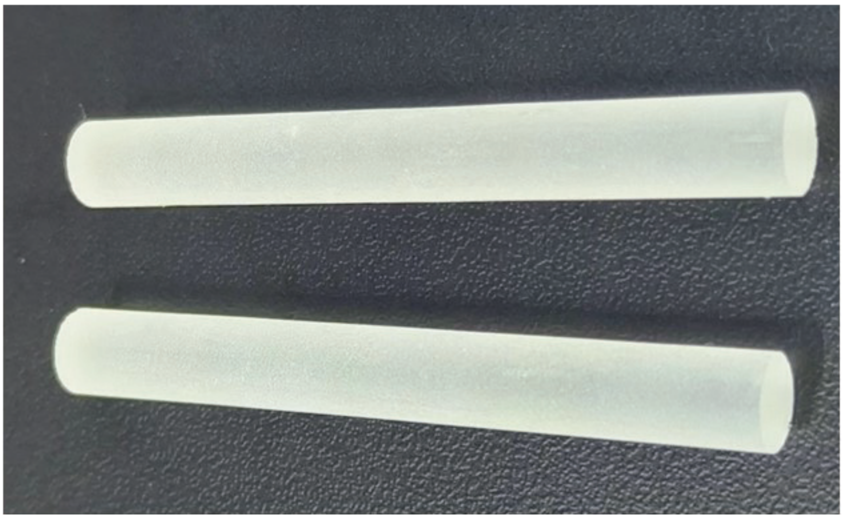

Fig. 1. CTE test sample

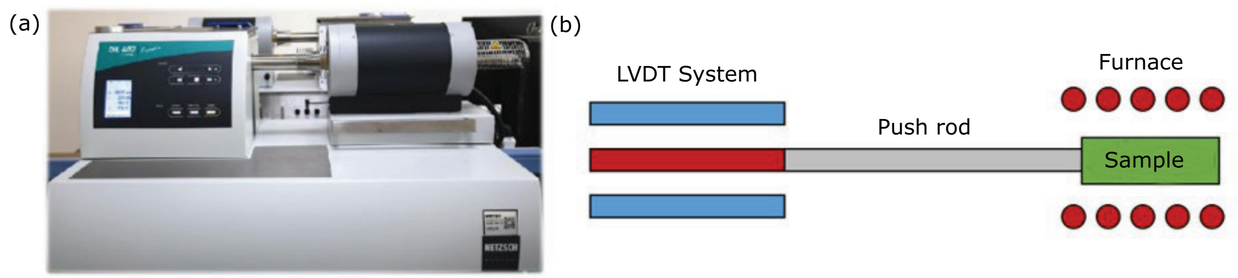

Fig. 2. (a) NICHI DIL 402PC photo, reprinted from (13). Copyright 2018, with permission from Elsevier; (b) the basic structure diagram of push rod thermal expansion instrument

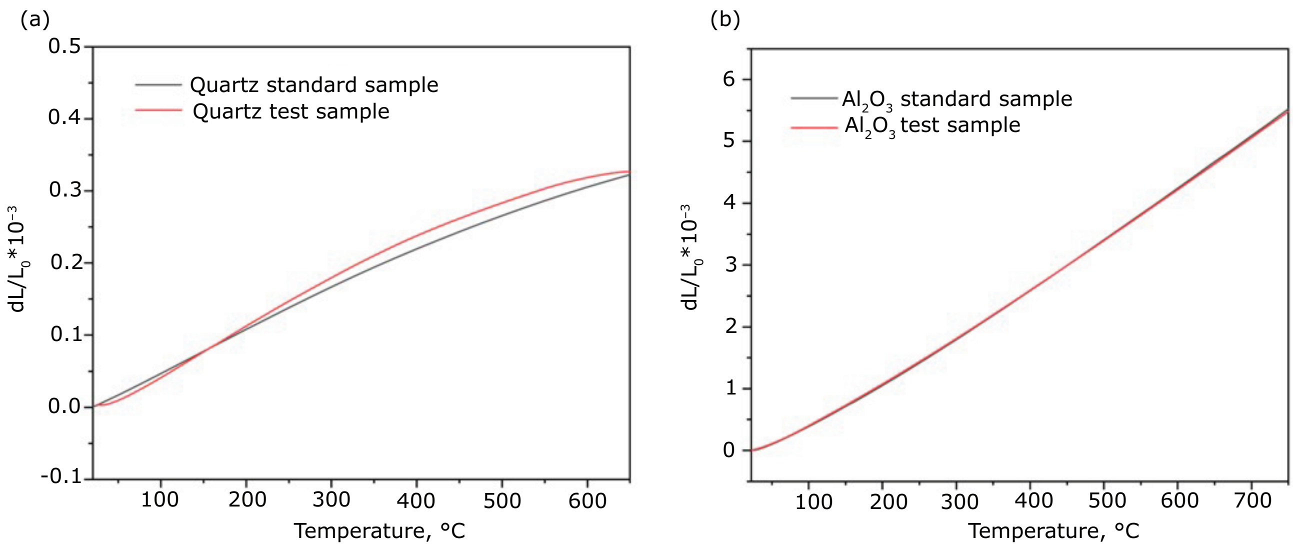

Fig. 3. Instrument quartz: (a) alumina; (b) baseline correction

expansion factors of the system and the expansion factors of the system were corrected and deducted during the sample test (14). The heating rate of the instrument was set to 5°C min^{-1} during the test. As shown in **Figure 2(b)**, the linear variable differential transformer (LVDT) is a displacement sensor, which is connected to the push rod. The change signal of the sample length is obtained by the contact between the push rod and the sample. The force of the push rod on the sample is very small and can be ignored.

2.3 Measurement Methods

The sample is in a temperature-controlled furnace. In the process of programmed temperature (linear, heating, cooling, constant temperature and its combination), the CTE test map of the sample can be obtained by using LVDT to continuously measure the length change of the sample, as shown in **Figure 3**. The CTE refers to the engineering expansion coefficient of the sample relative to the room temperature (15), which is defined as the average length change rate of the unit length sample in a certain temperature range (T_1, T_2). The calculation formula is as follows:

$$\alpha_{(T1-T2)} = [(\varepsilon_X)_{(T2)} - (\varepsilon_X)_{(T1)}]/(T_2-T_1) \qquad \text{(i)}$$

$$\varepsilon_X = dL/L_0 = \Delta L/L_0 \qquad \text{(ii)}$$

T_1 is the reference temperature, generally at room temperature; T_2 is the target temperature.

3. Results and Discussion

3.1 Sample Shape and Size

In the past, before the development of nano level precision in engineering applications, the influence of sample shape and size was often ignored. With the rapid development of modern science and technology, the influence of sample shape and size factors on the CTE testing of materials has received widespread attention (16). In this experiment, the effect of sample shape and size on the CTE test results was systematically investigated. The experimental data are shown in **Tables II and III**.

From **Tables II and III**, it can be seen that the shape and size of the samples affect the test results of the CTE. This may be due to the uneven distribution of the internal temperature of samples of different sizes during the heating process, which leads to the formation of a variable temperature field and the generation of thermal stresses, which in turn affects the results of the CTE test. The experimental results show that if the diameter and length of the sample are slightly larger than the standard value, the experimental data will be closer to the true value. Therefore, in order to improve the accuracy of the CTE, a standard test method should be established that specifies the shape and size of the test specimens.

3.2 Starting Temperature of the Furnace

The CTE of the same sample at different furnace temperatures was obtained by thermal expansion method. The specific results are listed in **Table IV**. It can be seen from **Table IV** that for the same sample, the higher the initial furnace temperature, the smaller the CTE at 30–300°C.

In order to draw the above conclusions, the following assumptions can be made: the furnace temperature at the first measurement is 34°C and the furnace temperature at the second measurement is 50°C. **Figures 4 and 5** are the structural diagrams of the CTE before and during the CTE test. Thermocouple 1 measures the temperature near the sample, that is, the abscissa temperature of the expansion curve. Thermocouple 2 measures the temperature of the furnace body. It should be noted that the thermal conductivity of the thermocouple is better than that of the glass sample, so the temperature of Thermocouple 1 (temperature near the sample) will

Table II Comparison of CPB's Measured Coefficient of Thermal Expansion with Different Lengths

Sample length, mm	Sample diameter, mm	CTE at 30–300°C, K^{-1}
46	6.0	88.2×10^{-7}
48	6.0	89.6×10^{-7}
50	6.0	91.3×10^{-7}
52	6.0	91.7×10^{-7}
54	6.0	92.0×10^{-7}

Table III Comparison of CPB's Measured Coefficient of Thermal Expansion with Different Diameters

Sample length, mm	Sample diameter, mm	CTE at 30–300°C, K^{-1}
50	5.6	87.3×10^{-7}
50	5.8	88.7×10^{-7}
50	6.0	91.3×10^{-7}
50	6.2	92.1×10^{-7}
50	6.4	93.5×10^{-7}

Table IV Thermal Expansion Coefficient of Samples at Different Furnace Temperatures

Starting temperature, °C	CTE at 30–300°C, K^{-1}	T_g, $°C^{-1}$	T_f, $°C^{-1}$
31–32	91.0×10^{-7}	654.1	702.2
33–34	91.2×10^{-7}	652.6	700.3
35–40	91.4×10^{-7}	657.9	703.4
50	84.1×10^{-7}	631.1	676.5

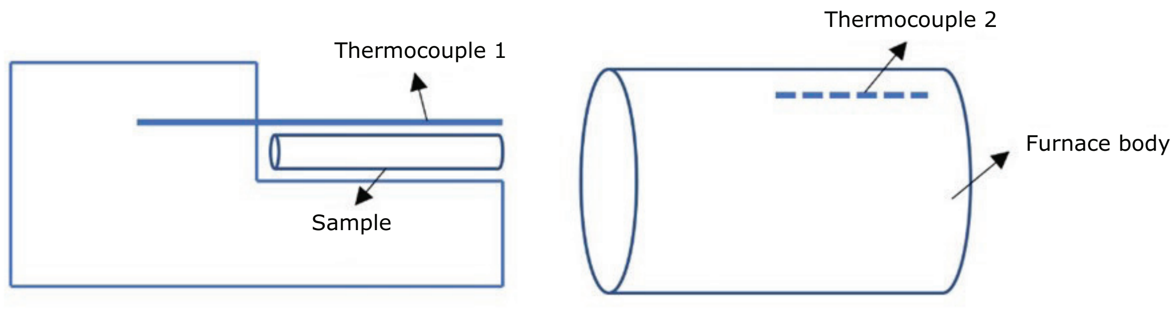

Fig. 4. Structure diagram before CTE test

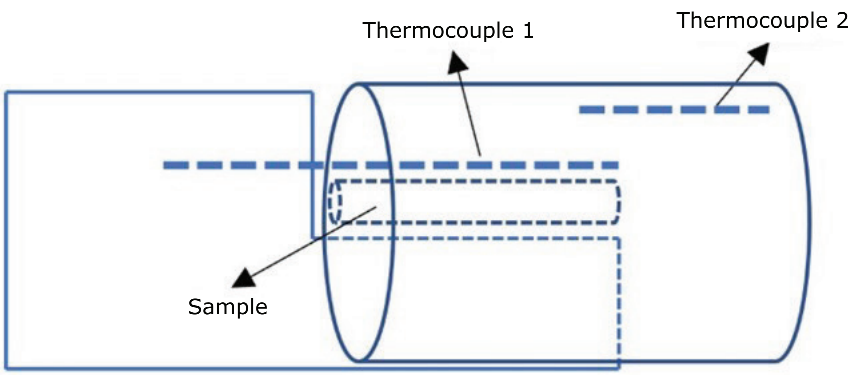

Fig. 5. Structure diagram in CTE test

be slightly higher than the actual temperature of the glass sample. When the display temperature of the two measured samples is 30°C, the time for the display temperature of the second measurement to rise from room temperature to 30°C is shorter than that of the first measurement. This is because the temperature of the furnace body at the second measurement is higher than that at the first measurement. At this time, the actual temperature of the sample at the second measurement is smaller than that at the first measurement. The following reasonable assumptions can be made: the second measurement is actually the expansion of the sample at 25°C when the display temperature is 30°C and the first measurement is actually the expansion of the sample at 28°C when the display temperature is 30°C. The CTE of 25–300°C is smaller than that of 28–300°C. Therefore, it can be concluded from the above analysis that the higher the initial furnace temperature, the smaller the CTE at 30–300°C.

In order to reduce the influence of furnace temperature on measurement of CTE, the same sample was tested at different initial furnace temperatures. The test results show that when the furnace temperature is lower than 35°C, it has little effect on the CTE (shown in **Table V**). This is because at low temperatures, the heat transfer coefficient of the glass is relatively low. For it to reach the temperature equilibrium will take a relatively long time for the samples to reach the initial temperature required for the test so that it can be stabilised to characterise the expansion characteristics of the glass material. Therefore, in order to reduce the influence of furnace temperature on the test results of the CTE and ensure the accuracy of the test results, the furnace temperature should be kept below 35°C at the beginning of the test.

Table V Coefficient of Thermal Expansion Test

Initial furnace body temperature, °C	CTE at 30–300°C, K^{-1}
31	91.3×10^{-7}
33	91.5×10^{-7}
35	91.4×10^{-7}
40	85.8×10^{-7}
50	84.1×10^{-7}

3.3 Heat Treatment Process

Samples of the same material, same shape and same size can have variations in the CTE of the sample due to different processing techniques (17–19). In this experiment, the CTEs of the samples at different annealing temperatures were studied under the same annealing time. The experimental results are shown in **Table VI**. The results show that the coefficients of expansion of the samples are different at different annealing temperatures, mainly due to the changes in the structure and stress conditions of the internal components of the samples caused by the different annealing temperatures (**Figure 6**).

3.4 Other Influencing Factors

The position of the sample in the furnace also affects the results of the CTE test. The same sample was not clamped with a push rod during the first test, but was clamped during the second measurement. The experimental results are shown in **Figure 7**. There is a mutation point and a peak on the curve. The mutation point represents the transition temperature of the glass, the transition temperature refers to the point at which the physical properties of the glass material change significantly at a specific temperature. Specifically, when the temperature of the glass reaches the transition point, the refractive index, specific heat capacity, CTE and other properties of the glass will change; the structure of the glass will also change. The peak value refers to the softening point of the glass, which indicates that the glass begins to enter the softening state, during which it is easy to carry out various processing and forming operations. From the diagram, we can find that the length of the sample measured for the first time (49.988 mm) is larger than that measured for the second time (49.880 mm). This is because the test system obtains the change signal of the sample length through the contact between the push rod and the sample. In the first testing round, the push rod is not clamped when contacting the sample, resulting in a gap between the sample and the push rod. A larger length in sample can be measured and a deviation appears between the first measured CTE curve and the second measured curve, which will lead to a wrong interpretation of the thermal properties of the material and affect its later practical application. Therefore, it is necessary to carefully check the placement of the sample during the test to reduce the error of the test results.

Table VI Coefficient of Thermal Expansion of Samples at Different Annealing Temperatures

Annealing temperature, °C	CTE at 30–300°C, K^{-1}
590	92.1×10^{-7}
600	91.4×10^{-7}
610	90.9×10^{-7}

Figure 8 shows the impact of improper test data processing on the accuracy of test results. **Figure 8(a)** is the result of deviation caused by the intersection point mark error, **Figure 8(b)** is

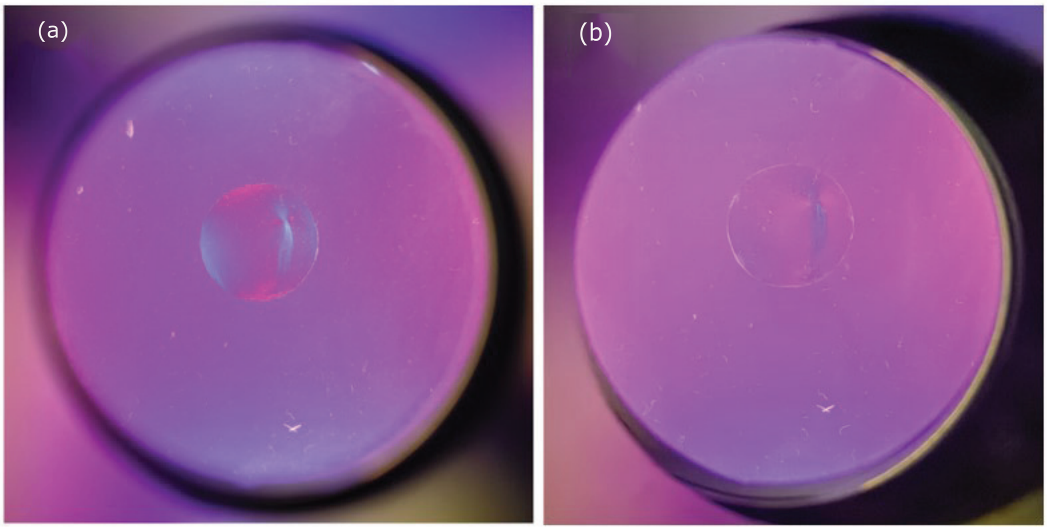

Fig. 6. The stress distribution at different annealing temperatures: (a) 590°C; (b) 610°C

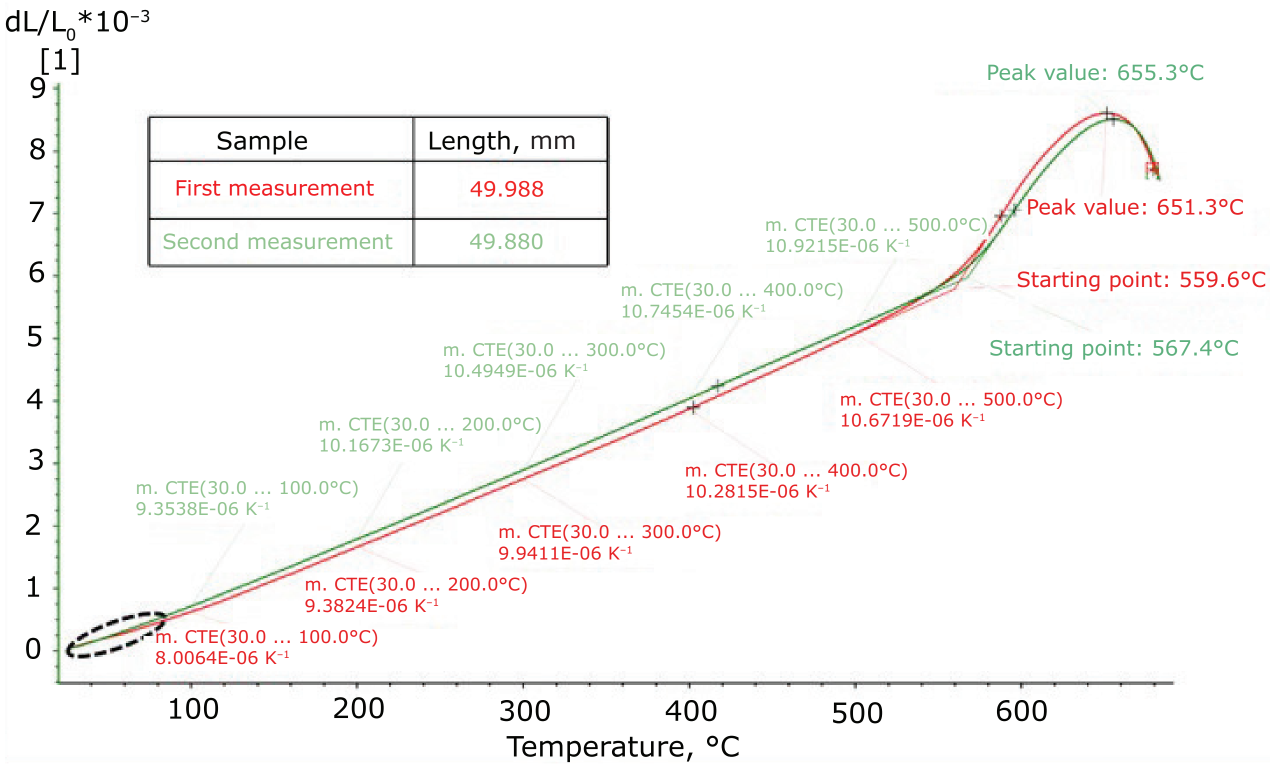

Fig. 7. The measurement results when the sample is in different positions in the furnace

the normal data processing and **Figure 8(c)** is the error caused by the subtraction definition error. The test results show that data processing is an important part of the test. Each step in the data processing should be accurate and appropriate to ensure the accuracy of the test results.

4. Conclusion

With the development of science and technology, the requirements for material accuracy are increasing and the influence of thermal deformation cannot be ignored. Comprehensive consideration of the influencing factors of thermal expansion is an effective way to improve the accuracy of thermal expansion calculation. In this study, the effects of sample shape and size, initial furnace temperature and heat treatment process on the test results of expansion coefficient were discussed. From the current work, the following conclusions are drawn. The sample shape parameter is a factor that cannot be ignored in the expansion coefficient test. The test should be processed with reference to the standard sample size. Based on the current experimental results, the sample size is slightly larger than the standard sample size, which has little effect on the expansion coefficient test results. Therefore, it is necessary to study the relationship between them. It is preliminarily concluded that when the initial furnace temperature is lower than 35°C, it has little effect on the test results of expansion coefficient. The heat treatment process was found to influence the expansion coefficient, it is therefore necessary to study its relationship in a follow-up study. Many other factors, such as sample placement and data processing, should be considered to obtain fully accurate results.

The thermal expansion performance of materials has obvious scientific significance and important engineering application value in high-tech fields such as aerospace, microelectronics, efficient energy utilisation, nuclear energy, new material development, as well as industrial fields such as the petrochemical industry, iron and steel metallurgy, building energy conservation, refrigeration and air conditioning. It is the basis of engineering design, technological innovation, application field and scientific research. This work systematically studies the influencing factors of expansion coefficient and provides guidance for subsequent research work.

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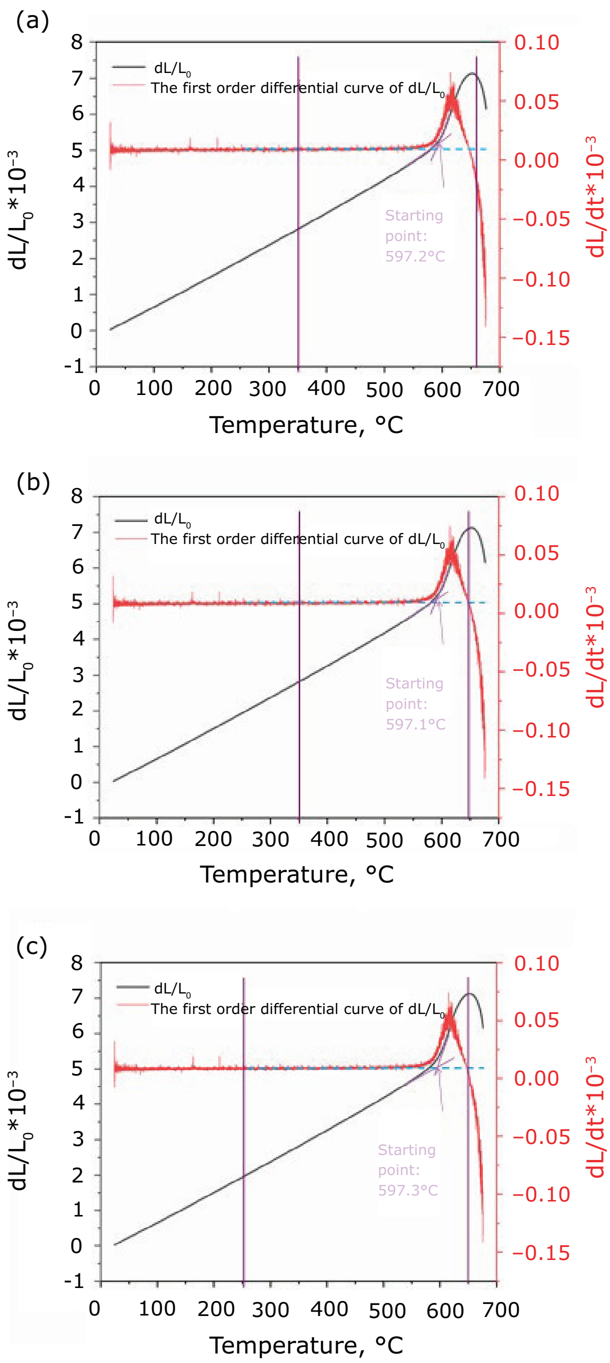

Fig. 8. The error diagram caused by data processing: (a) red dotted line and differential curve intersection mark error; (b) correctly handle starting point data; (c) the starting point minus different values

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References

1. M. Hadipeykani, F. Aghadavoudi, D. Toghraie, *Phys. A: Stat. Mech. Appl.*, 2020, **546**, 123995
2. M. Zhang, Z. Cao, S. Yang, Y. Zhang, Y. Han, F. Qiu, J. Zheng, H. Liu, J. Jia, *J. Chin. Ceram. Soc.*, 2024, **52**, (10), 3301
3. J. Kinast, E. Hilpert, R.-R. Rohloff, A. Gebhardt, A. Tünnermann, *Surf. Coatings Technol.*, 2014, **259**, (C), 500
4. A. T. Fadhil, G. Washer, A. Poudel, *Mater. Eval.*, 2024, **82**, (1), 79
5. M. Torabi Milani, J. Del Fatti, K. Orna, Y. Zhang, A. N. Sinclair, *Mater. Eval.*, 2023, **81**, (3), 38
6. H. Hayashi, M. Watanabe, H. Inaba, *Thermochim. Acta*, 2000, **359**, (1), 77
7. Y. Hua, 'Analysis of Influencing Factors of Thermal Expansion Coefficient', The 5th Baosteel Academic Annual Conference, Shanghai, China, 1st June, 2013
8. Y. Meng, *Ind. Metrol.*, 2005, **15,** (3), 6
9. A. X. Lu, Z. B. Ke, Z. H. Xiao, X. F. Zhang, X. Y. Li, *J. Non-Cryst. Solids*, 2007, **353**, (28), 2692
10. H. Wang, X. Zhou, L. Sun, J. Dong, S. Yu, *Nucl. Eng. Des.*, 2009, **239**, (3), 484
11. G. Laplanche, P. Gadaud, O. Horst, F. Otto, G. Eggeler, E. P. George, *J. Alloys Compd.*, 2015, **623**, 348
12. Z. Cao, J. Jia, China Building Mat Academy, 'Micro Channel Plate Borate Core Glass and Preparation Method Thereof', *Chinese Patent* 105293903, 2017
13. M. Hunkel, H. Surm, M. Steinbacher, 'Dilatometry' in "Handbook of Thermal Analysis and Calorimetry", eds. S. Vyazovkin, N. Koga, C. Schick, ch. 3, Vol. 6, Elsevier, Amsterdam, The Netherlands, 2018, pp. 103–129
14. 'Standard Test Method for Linear Thermal Expansion of Solid Materials with a Push-Rod Dilatometer', ASTM E0228-17, ASTM International, West Conshohocken, USA, 2017
15. N. Taibi, Z. Belabed, B. Boucham, M. Bennguediab, A. Tounsi, K. M. Khedher, M. A. Salem, *J. Appl. Comput. Mech.,* 2024, **10,** (2), 224
16. K. Lichtenberg, K. A. Weidenmann, *Thermochim. Acta*, 2017, **654**, 85
17. A. X. Lu, Z. B. Ke, Z. H. Xiao, X. F. Zhang, X. Y. Li, *J. Non.-Cryst. Solids*, 2007, **353**, (28), 2692
18. Z. Meilun, C. Zhenbo, Y. Shengyun, H. Yu, W. Ke, Z. Yang, Z. You, M. Jing, B. Tiezhu, L. Hui, J. Jinsheng, *J. Alloys Compd.*, 2024, **1005**, 175991
19. A. Q. Tool, *J. Am. Ceram. Soc.*, 1946, **29**, (9), 240

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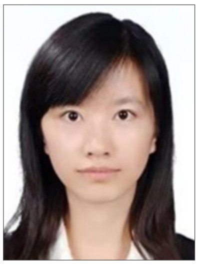

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