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Research Article

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Specific Heat of Zirclaoy-4

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Abstract

This study investigates the specific heat of Zircaloy-4 using differential scanning calorimetry within the temperature range of 293–663 K. The calorimeter was calibrated with a certified standard material (SRM 720, Sapphire, NIST) to ensure proper operation and determine the correction value. The correction value for the measurement data ranged from 0.99 to 1.01, with a maximum uncertainty of 2.39%. Specific heat increased with increasing temperature. As the temperature increased from 293 K to 663 K, the specific heat of Zircaloy-4 increased from 0.291 J/(g-K) to 0.321 J/(g-K), slightly surpassing that of pure zirconium. The uncertainty of the specific heat ranged from $6.86\times10^{-}3$ J/(g-K) to $1.1610^{-}2$ J/(g-K), equivalent to 2.14%–3.93%.

Keywords: Specific Heat, Zircaloy-4, Differential Scanning Calorimetry, Calibration, Uncertainty.

Introduction

Nuclear power harnesses energy derived from high temperatures and pressures. In nuclear reactors, fuel rods are typically subjected to extremely severe conditions. The stability of the nuclear industry largely depends on the integrity of these fuel rods under such conditions. Ensuring the soundness of nuclear fuel rods involves meeting.

Several Critical Design Requirements:

1. Maintaining the nuclear fuel temperature below its melting point.
2. Preventing an increase in rod internal pressure (RIP).
3. Limiting the total tangential strain of the cladding tube to 1% or less.
4. Keeping the equivalent strain below 2.5 %.
5. Restricting the thickness of the cladding tube oxide layer to 100 μm or less.

The temperature of the nuclear fuel rods significantly influences all of these requirements and thus directly impacts the design specifications. Key thermophysical properties such as specific heat, thermal expansion, density, and thermal diffusivity of the nuclear fuel and cladding materials, play a critical role in determining the temperature distribution in the fuel rods and assessing valuating the reliability and safety of reactor systems. Among these, the heat capacity is a crucial parameter for calculating temperature distribution using the governing heat transfer equation. Various types of measurement data [1-4] and review and correlation data for computer codes [5-12] are available. However, these measurement data have not been evaluated for uncertainty, which is essential for ensuring the reliability of these data used in design and safety assessments.

In this study, the specific heat of Zircaloy-4 is measured using differential scanning calorimetry (DSC). Self-calibration of the DSC was conducted using a standard reference material (SRM 720, Sapphire, NIST), enabling the determination of a correction factor for the measurement data. The uncertainty of the measured specific heat data for Zircaloy-4 was evaluated following the Guide to the Expression of Uncertainty in Measurements (GUM) [13], thereby enhancing the reliability of the measurement data.

Experimental

To calibrate the DSC in this study, we defined the correction factor as the ratio of the specific heat value from the standard material certificate to the specific heat value measured directly using the DSC:

$$f_{cal} = \frac{c_{pref-c}}{c_{pref-m}} \quad (1)$$

where fcal is the correction factor, f_{cal} is the correction factor, c_{pref-c} is the specific heat value of the SRM as provided in the certificate, and c_{pref-m} is the specific heat value measured directly using DSC.

The specimen used for measuring the specific heat of Zircaloy-4 was obtained from the Korea Nuclear Fuel Corporation. The diameter and thickness of the specimen were 5 mm and 1 mm, respectively. The chemical composition of the specimen is listed in TABLE 1, alongside the chemical compositions of other zircaloys for comparison.

Table 1: Chemical Composition of Zircaloy

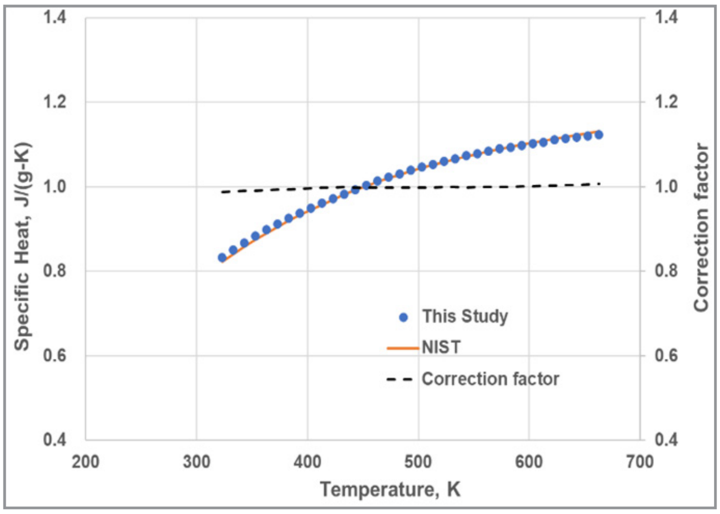
Element	Concentration (wt%)			
	This study	Terai [1]	Brooks and Stansbery [2]	Murabayashi[3]
Sn	1.31	1.558	1.29	1.51
Fe	0.25	0.220	0.037	0.23
Cr	0.14	0.117	0.0095	0.10
O	0.125	0.132	0.012	
Ni			0.0496	
Al			0.066	
Zr	Balance	Balance	Balance	Balance

The specific heat of Zircaloy-4 was measured in the temperature range of 293-673 K using DSC (DSC200F3, Netzsch). The measurements were conducted at a constant heating rate of 5 K/min under an argon gas flow.

Result and Discussion

Self-Calibration

Self-calibration was carried out using a standard reference material (SRM 720, NIST) to verify the correct operation of DSC. The certified and measured specific heat values of the SRM, along with the DSC correction factor, are plotted against temperature in Fig. 1. These values range between 0.99–1.01 with uncertainty ranging from 1.38 to 2.39%. The specific heat measurement data of the SRM archives a correction factor of 1%, and the uncertainty up to 2.39% confirms the proper operation of the device.



Figtre 1: Certified and measured values of the specific heat of the SRM, along with the correction factor of DSC.

Specific Heat of Zircaloy-4

Specific heat is the amount of heat absorbed per unit mass of the material when its temperature increases by 1 K. This property can be characterized using the following expression:

$$c_x = \frac{1}{m}\frac{\Delta Q}{\Delta T} \quad (2)$$

where c_x, m, $\triangle Q$, and $\triangle T$ represent specific heat at constant x (x is volume or pressure), mass of the specimen, quantity of heat absorbed and temperature increase, respectively. Fig. 2 shows the measured specific heat of Zircaloy-4 with respect to temperature. This specific heat can be expressed as a function of temperature using the following equation:

$$c_p = -1.396\times10^{-7}\,T^2 + 1.377\times10^{-4}\,T + 0.288 \quad (3)$$

where cp represents the specific heat and T represents the temperature.

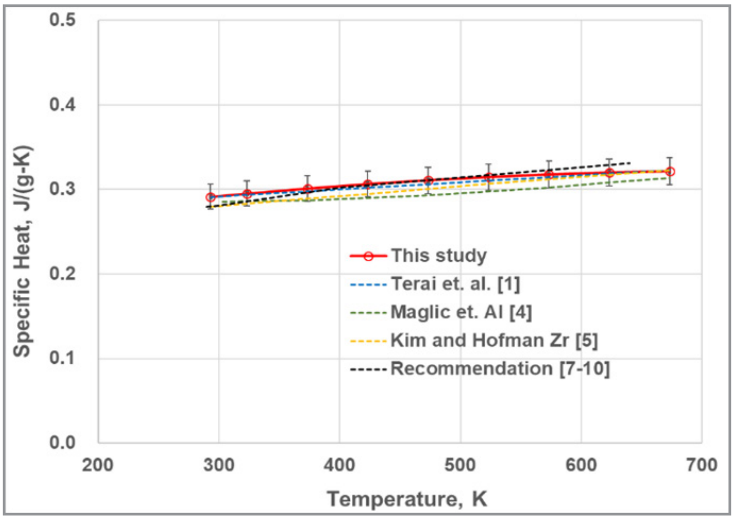


Figure 2: Specific Heat of Zircaloy-4 in the Temperature Range of 293–663 K with 5 % Error Bar

The red line in Fig. 2 represents the experimental data, whereas the other lines represent other research data for comparison. All reference data are within 5% error bars. Our data align well with those reported by Terai et al.[1]. Although the data reported by Maglic et al. [4] showed the greatest difference compared to our data, this difference is still within an acceptable 5% margin of error.

The specific heat of Zircaloy-4 increased slightly with an increase in temperature. The specific heat of Zircaloy-4 is slightly higher than that of pure zirconium, as reported by Kim and Hofman [5]. This is believed to be due to the high specific heat of Fe and Cr, which are added as alloying elements.

Several computer codes use specific heat data for zirconium-based alloys [8-12]. The correlations in FRAPCON-3.4 and FRAPTRAN 1.4, which are computer codes used to model steady-state and transient fuel behavior, respectively, in the regulatory analysis, are applicable to Zircaloy-2, Zircaloy-4, ZIRLO, and M5 alloys. These data points used in computer codes were based on data reported by Brooks and Stansbury [2]. At low temperatures, the measured specific heat of Zircaloy-4 was slightly higher than the data in the computer codes; however, at high temperatures, it was slightly lower.

Uncertainty of the Specific Heat Data of Zircaloy-4

To accurately quantify the uncertainties in repeated measurements and calibration corrections, it is crucial to distinguish and address both random (type-A) and systematic (type-B) uncertainties. The uncertainty of the measured specific heat of the Zircaloy-4 data was evaluated according to the measurement uncertainty expression guidelines (GUM) [13].

The Uncertainty was Evaluated in Four Stages:

1. Mathematical expression of the relationship between the measurand and input quantities
2. Evaluation of the standard uncertainty of each input estimate
3. Determination of the combined standard uncertainty
4. Calculation of the expanded uncertainty.

Mathematically Expression of Specific Heat

Specific heat can be mathematically expressed using the following equation:

$$c_p = \frac{1}{m} C_p f_{cal} \tag{4}$$

where c_p is the specific heat (J/g-K), m is the mass of the specimen (g), c_p is the heat capacity (J/K), and f_{cal} is a correction factor according to the self-calibration of the DSC using the SRM.

Evaluation of the Standard Uncertainty of Each Input Estimate

The factors affecting the uncertainty of the specific heat were the specimen mass, heat capacity of the specimen measured using DSC with respect to the temperature at a constant heating rate, and the correction factor (f_{cal}) of the self-calibration of the DSC.

The mass value of the specimen must be calculated by adding a correction value of the analytical balance to the measured value using an analytical balance:

$$m = m_m + b \tag{5}$$

where m is the mass value of the specimen *(g)*, m_m is the mass value measured using an analytical balance, and b is a correction factor based on the calibration of the analytical balance.

Uncertainty of the mass value is the uncertainty of the mass measured using an analytical balance and the uncertainty of the correction value of the analytical balance. The uncertainty of the specimen mass measured using an analytical balance consists of random uncertainty due to repeated measurements and systematic uncertainty due to the resolution limit of the analytical balance. Therefore, the mass uncertainty is

$$u(m) = \sqrt{u^2(m_m) + u^2(b)} = \sqrt{(u^2(m_{m-r}) + (u^2(m_{m-R}) + u^2(b)} \tag{6}$$

where $u(m)$ is the standard uncertainty of the mass, $u(m_m)$ is the uncertainty due to mass measurement using the analytical balance, $u(m_{m-r})$ is the random uncertainty due to repeated measurements, $u(m_{m-R})$ is the systematic uncertainty due to the resolution limit of the analytical balance, and $u(b)$ is the uncertainty of the correction factor of the analytical balance. For the standard uncertainty of the heat capacity of the specimen measured using

DSC, the random uncertainty can be expressed as the as the experimental standard deviation of the mean:

$$u(C_p) = s(C_p)/\sqrt{n} \tag{7}$$

where $u(C_p)$ is the random uncertainty for heat capacity, $s(C_p)$ is the standard deviation od the heat capacity measurements, and n is the number of repeats.

The uncertainty associated with the DSC calibration factor, f_{cal}, was obtained from the self-calibration certificate.

$$u(f_{cal}) = U_{fcal}/k \tag{8}$$

where $u(f_{cal})$ is the standard uncertainty of the DSC correction factor, Uf_{cal} is the expanded uncertainty, and k is the coverage factor.

Determination of the Combined Standard Uncertainty

The combined standard uncertainty was calculated using the previously evaluated standard uncertainties. Specifically, it was determined based on the standard uncertainties of the specimen mass, heat capacity, and correction factor as follows:

$$u_c = \sqrt{a_m^2\, u^2(m) + a_{C_p}^2\, u^2(C) + a_{fcal}^2\, u^2(f_{cal})} \tag{9}$$

where ax are sensitivity coefficients,

$$a_m = \frac{\partial}{\partial m}\left(\frac{1}{m} C_p f_{cal}\right) = -\frac{1}{m^2} C_p f_{cal} \tag{10}$$

$$a_{C_p} = \frac{\partial}{\partial C_p}\left(\frac{1}{m} C_p f_{cal}\right) = \frac{1}{m} f_{cal} \tag{11}$$

$$a_{fcal} = \frac{\partial}{\partial f_{cal}}\left(\frac{1}{m} C_p f_{cal}\right) = \frac{1}{m} C_p \tag{12}$$

Calculation of the Expanded Uncertainty

The expanded uncertainty (U) is obtained by multiplying the combined standard uncertainty $(u_c)$with the coverage factor (k):

$$U = k \times u_c \tag{13}$$

If the effective degrees of freedom (v_{eff}) are significantly high, it is appropriate to adopt $k = 2$ and assume that $U = 2u_c$, which defines an interval with a confidence level of approximately 95%. Uncertainty of the specific heat data measured using DSC in this study is listed in TABLE 2. It varied from 6.86×10^{-3}-1.16×10^{-2} J/g-K in the temperature range of 293-663 K, with an uncertainty of 2.14-3.93%.

Table 2: Uncertainty of the specific heat data of Zircaloy-4

Temp. K	Specific heat, cp J/g-K	Correction factor, f_{cal}	Standard uncertainty, u(x)			Combined uncertainty u_c, J/g-K	Coverage factor k	Expanded uncertainty, U	
			Mass, u(m), g	Heat capacity, $u(C_p)$, J/K	Correction factor, $u(f_{cal})$			J/g-K	%
323	5.62E-02	0.99	1.72E-05	7.93E-04	9.08E-03	4.91E-03	2.36	1.16E-02	3.93
373	5.73E-02	0.99	1.72E-05	7.42E-04	8.41E-03	4.63E-03	2.36	1.09E-02	3.63
423	5.83E-02	1.00	1.72E-05	7.23E-04	7.68E-03	4.47E-03	2.36	1.06E-02	3.44
473	5.91E-02	1.00	1.72E-05	6.93E-04	6.16E-03	4.10E-03	2.36	9.70E-03	3.13
523	5.98E-02	1.00	1.72E-05	5.73E-04	5.51E-03	3.47E-03	2.36	8.21E-03	2.61
573	6.05E-02	1.00	1.72E-05	4.91E-04	5.30E-03	3.08E-03	2.36	7.28E-03	2.29
623	6.09E-02	1.00	1.72E-05	4.08E-04	6.08E-03	2.90E-03	2.36	6.86E-03	2.14
663	6.11E-02	1.01	1.72E-05	3.51E-04	6.95E-03	2.90E-03	2.36	6.86E-03	2.14

Conclusions

In this study, the specific heat of Zircaloy-4 was determined using DSC. Additionally, self-calibration of the calorimeter and evaluation of the uncertainty associated with the specific heat data of Zircaloy-4 were performed.

The Results of this Study can be Summarized as Follows:

1. The correction value of 1% for the specific heat data of the SRM and an uncertainty of up to 2.39% confirmed that the device operated successfully.
2. The specific heat of Zircaloy-4 increased with increasing temperature. From 293 K to 663 K, the specific heat increased from 0.291 J/g-K to 0.321 J/g-K, with an uncertainty of 6.86×10^{-3} to 1.16×10^{-2} J/g-K, equivalent to 2.14–3.93%.
3. The specific heat of Zircaloy-4 was slightly higher than that of Zr, with all other research data falling within a 5% error margin.

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